

Li-ion battery cathode manufacture in Australia

A SCENE SETTING PROJECT



Contents

		Disclaimer	3
List of tables, figures and appendices	4	Executive summary	6
Introduction	11	Project Objectives	14
Cathode Active Materials	15	Precursor Chemicals	20
Engineering Development	54	Value-Add Estimates	80
Recommendations Summary	83	Acknowledgments	86
About the Future Battery Industries Cooperative Research Centre	87	About the Queensland University of Technology Institute for Future Environments	87
Endnotes	88	Appendix	90

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List of tables, figures and appendices

Figure 1	Demand of various cathode materials in the EV market	11
Figure 2	Flow diagram for Scene Setting Report objectives	13
Figure 3	Current processing methods for nickel sulphate	23
Figure 4	IGO's flowsheet to convert sulphide nickel concentrate to nickel sulphate	24
Figure 5	Outlook for supply and demand of nickel sulphate, 2012-2028 (kt Ni)	25
Figure 6	Estimated market balance for nickel, 2017-2028	26
Figure 7	Estimated supply and demand for nickel, 2017-2028	26
Figure 8	Estimated capacity and production for nickel sulphate, 2016-2022	27
Figure 9	Schematic of HPMSM production from HPEMM or carbonate ores	29
Figure 10	Simplified flowsheet of HPMSM production from oxide ore	30
Figure 11	MMC's flowsheet for HPEMM production from oxides ores in Nelspruit, South Africa	31
Figure 12	Estimated market demand for HPMSM and HPEMM in the battery industry, 2015-2040	32
Figure 13	HPEMM entry into HPMSM supply chain	33
Figure 14	Sample cobalt sulphate battery grade specifications	35
Figure 15	Simplified flowsheet for cobalt sulphate production	36
Figure 16	First Cobalt's conceptual flowsheet for cobalt sulphate	37
Figure 17	Mckinsey cobalt market outlook, 2017-2025	40
Figure 18	Cobalt supply and demand outlook, 2018-2028	41
Figure 19	Cobalt demand and price forecast, 2018-2028 (Roskill, 2019)	41
Figure 20	Simplified flowsheet of LiOH production by spodumene processing	46
Figure 21	Production of mined lithium by country	47
Figure 22	Lithium hydroxide production by source, 2000-2018 (t LCE)	48
Figure 23	Major producers of battery grade lithium hydroxide, 2014-2018	49
Figure 24	Total refined lithium capacity by integration, 2000-2018 (t/a LCE)	50
Figure 25	Total lithium supply and demand, 2017 vs 2025	52
Figure 26	Global forecast consumption of lithium by product, 2018-2028 (kt LCE)	53
Figure 27	NCM CAM synthesis process block flow diagram	54
Figure 28	Breakdown of Chemical Mixing	56
Figure 29	Breakdown of Co-Precipitation	57
Figure 30	Breakdown of Dry Solids Recovery	58
Figure 31	Breakdown of Lithiation and Doping	59
Figure 32	Breakdown of Calcination	60
Figure 33	Breakdown of Coating	61
Figure 34	Chemical Mixing and Co-precipitation reactor PFD	69
Figure 35	Dry Solids Recovery dewatering filter PFD	69
Figure 36	DrM FUNDABAC filter with 0.13m ² filter area	72
Figure 37	Horizontal plate and frame filter	72
Figure 38	Outotec Larox PF 0.4 filter	73
Figure 39	Dry Solids Recovery drying oven	74
Figure 40	Lithiation V-mixer	75
Figure 41	AWE V-mixer	75
Figure 42	Example of an RHK furnace	76
Figure 43	Batch rotating calcine kiln	77



Table 1	Summary of different NCM chemistries	17
Table 2	Sample nickel sulphate battery grade specifications	22
Table 3	Sample manganese sulphate battery grade specifications	28
Table 4	Largest producers of refined cobalt, 2013-2017 (tonnes)	30
Table 5	Sample lithium hydroxide battery grade specifications	45
Table 6	Key process design criteria	62
Table 7	Summary of electrical power draw	78
Table 8	Feed pricing	81
Table 9	Utility pricing	81
Table 10	Current market prices for P-CAM, CAM and precursor materials	81
Appendix A	Process flow diagram sketches	92-93
Appendix B	Process design criteria	94-102
Appendix C	Equipment list	103
Appendix D	Pilot plant capacity estimate	104-106
Appendix E	Filtration equipment	107-133
	E.1 IFS Consultant	
	E.2 Outotec	
	E.3 GBL Process	
Appendix F	Calcine kiln equipment	134-142

1. Executive Summary

Purpose

This research was commissioned to establish the economic feasibility and supply chain requirements for precursor manufacture in Western Australia at industrial scale. This report is one of a series of scene setting projects commissioned by the FBICRC to set direction and inform its initial research portfolio. It was undertaken by a team from the Queensland University of Technology with support from Hatch Engineering and contributions from Curtin University. The research details recommendations for cost effective, sustainable production of superior quality nickel-rich NCM (nickel cobalt manganese) battery cathode active material (CAM) and the supply of relevant precursor chemicals within Australia, while minimizing wastes. It will inform the FBICRC industry and research participants who have invested significant time and money in developing a proposal for a Cathode Precursor Production Pilot Plant project in Western Australia which is currently being scoped and costed.

Growing an Australian battery industry

The global automotive lithium-ion battery market is projected to reach USD 95.3 Billion by 2030 growing at an annualised rate of ~11%¹. In Europe alone, 500 GWh/annum of battery factories has been announced to be commissioned by 2025. The battery value chain supporting this growth offers opportunities to Western Australia given its endowment of key mineral resources and emerging technical grade battery chemical industry². The step to battery cathode active material and cathode precursor production is a key focus within the FBICRC's initial research portfolio.

Australia currently produces nine of the 10 mineral elements required to produce most lithium-ion battery anodes and cathodes, and has commercial reserves of graphite – the remaining element. Accelerating global demand presents Australia with a once-in-a-generation opportunity to transition into a major processing, manufacturing and trading hub if an adequate business case can be built. Critical components in advanced battery production – precursor, anode, cathode, electrolyte – can be manufactured in Australia. Battery manufacturing technology central to downstream lithium processing therefore stands as the critical gap in the Australian supply chain.

¹ Bloomberg, 21 November 2019.

² J.Wilson, 2020. The governance of battery value chains: Security, Sustainability and Australian Policy Options, FBICRC Scene Setting Report.

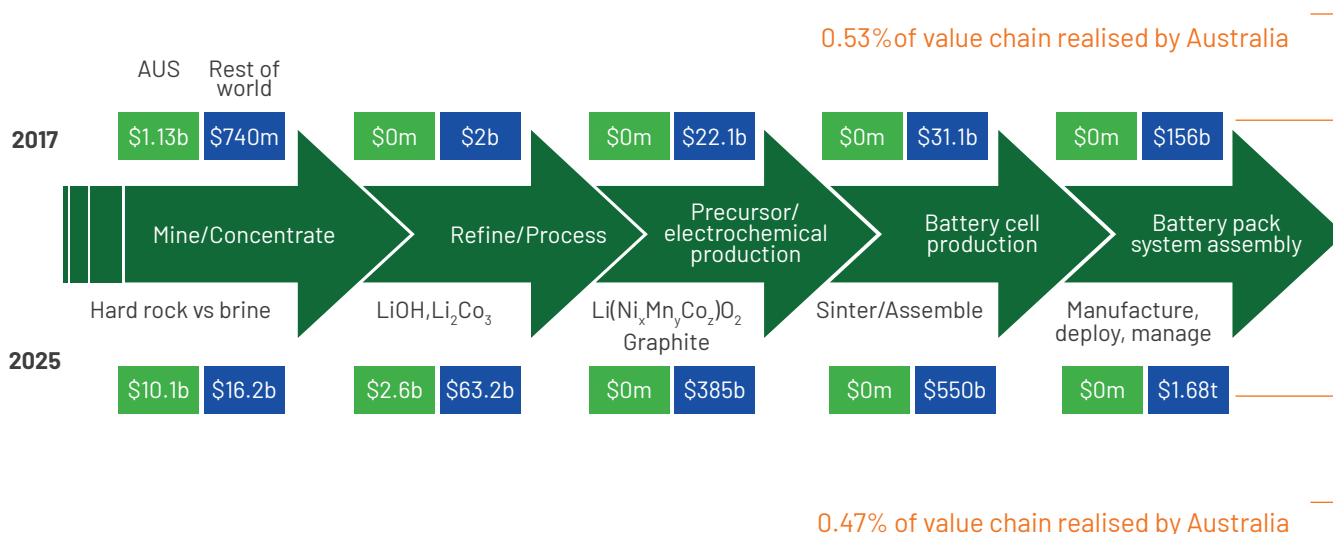
A number of mining groups are already advancing from raw battery minerals to the production of purified metal salts and their refinement to battery grade quality within Australia. These products are expected to supply the rapidly expanding global demand for vehicle electrification. NCM CAM is of particularly high relevance to the electric vehicle (EV) market, serving 53% of the market demand in 2019 and predicted to grow.

Due to the increasing market demand for NCM materials, and the Western Australian endowment and processing capability in lithium, nickel, manganese, cobalt chemicals, it has been chosen as the potential candidate for Australian CAM manufacture, and as such is the main focus of this Report. At an entry level to the value chain above the supply of concentrated minerals are the purified metal salts; above that is the mixed metal hydroxide precursor (P-CAM) product followed by the lithiated final CAM product.

Both P-CAM and CAM products are subject to strict quality specifications in terms of their impurities, particle size distribution and particle shape, and electrochemical attributes. Key to successful and sustainable production of these battery materials is the consistent achievement of quality requirements whilst ensuring that costs and environmental impacts are minimised.

Currently Australia exports the main commodities (Li, Ni, Mn, Co, C) used in the Lithium battery production in the form of mineral concentrates and very little of the value incorporated into manufacturing of lithium ion battery materials is retained in Australia. This is illustrated in the lithium value chain shown below where the production of concentrate yielded a revenue of \$1.13 billion for Australia in 2017, with no revenue obtained from the major value-adding steps, including precursor production that was worth \$22.1 billion². Future estimates expect precursor production to increase to around 17-fold by 2025. Battery precursor production is a crucial step to add value to the Australian battery industry and currently there are no facilities for this in Australia. With increasing global demand for lithium ion batteries Australia has been presented with a unique opportunity to transition into a major processing, manufacturing and trading hub to increase its share of market value.

Li-ion batteries - Lithium Value Chain 2017 - 2025



Future Smart Strategies 2018

Key Findings

The report provides a state-of-the-art technical assessment on the process to establish precursor manufacture in WA. It confirms its technical and commercial feasibility as the foundation for laboratory and pilot scale testing. The findings provide a firm foundation and direction for the next phase of the CRC's development work.

1.
The report identifies an international standard technique for WA manufacture

The 'co-precipitation' technique is identified by the report as the most suitable candidate for CAM manufacture. The main drivers for this being the maturity and low cost of the technology, which is the current industry standard for NCM/NCA CAM manufacture. The raw chemical supply required by this technique is also in line with current Australian production.

2.
A gradual ramp up of the pilot plant is proposed

Key recommendations of the report include a manufacturing schedule for differing NCM CAM types based on complexity of processing and current market demand. This gradual ramp up in processing capability will allow for development of the technical expertise required to produce high-quality CAM materials. This will reduce risk in the initial commissioning phase while also ensuring the material produced by the Pilot Plant is at the same level or better than what is currently accepted by cell manufacturers.

3.
A lab scale/mini plant phase before pilot scale production

A further recommendation is a lab-scale/mini plant phase to be implemented before pilot-scale production. This will provide validation and reasonable starting values to commence pilot-scale operation. As a much

² Future Smart Strategies 2018 .



smaller quantity of material is required at lab-scale, this will also reduce the operating costs and increase the speed of initial testing while minimising risk. The high level processing steps in the production of NCM CAM is the mixing and reaction of nickel, cobalt and manganese sulphate solutions with caustic soda and aqueous ammonia under inert conditions to produce a mixed hydroxide precipitate, which is washed, dried, lithiated with lithium hydroxide, calcined, ground, coated and re-calcined, before packaging in an inert and dry environment. Various additives may be added to give the CAM specific required properties. Aspects such as processing temperatures, reagent ratios, impurities, materials of construction, mixing control and retention time are key control parameters that have to be optimised in the process.

Next steps

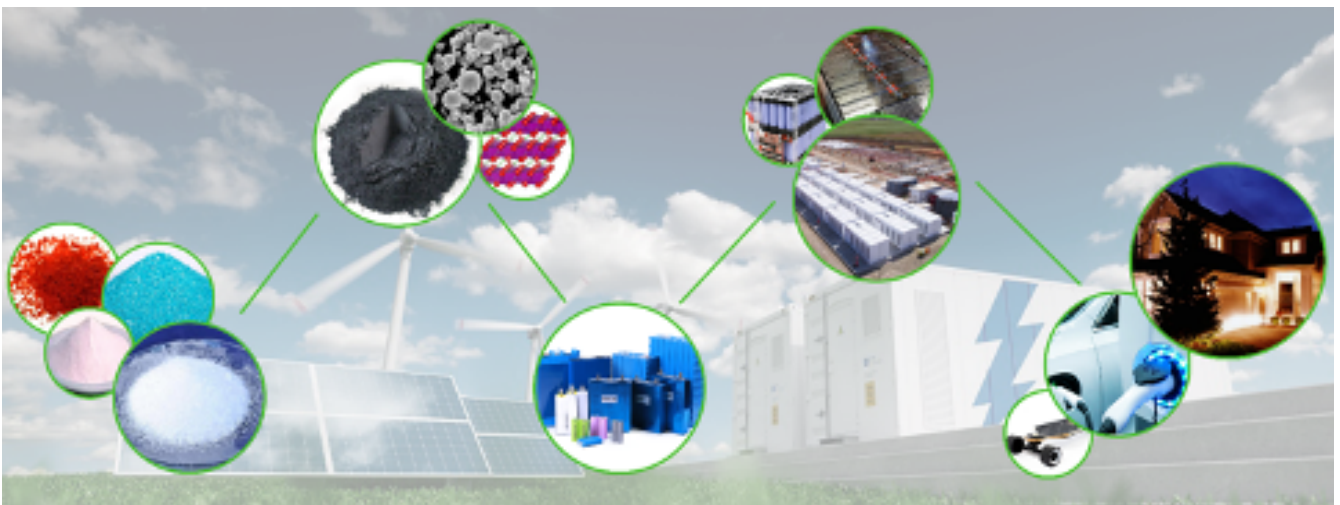
The aim of the 15 industry and four research participants in FBICRC supporting the cathode precursor pilot plant project is to demonstrate the technical and commercial viability of this further local processing step in the value chain. Process modelling and scale-up verification of final process design will be completed along with capital and operating costs for commercialisation at scoping study level. Environmental, safety and business benefits/risks will be analysed. Final project delivery will seek to prove the viability of the production of ultra-stable NCM/NCA cathode materials with a clear path to commercialisation.

Battery manufacturers are required to provide statistically representative numbers of good performing cells, which could be in the tens of thousands

or more, to validate their material is of required quality. This necessitates a basic capability to produce several hundreds of kgs of material with consistent performance characteristics. Thus, the FBICRC Pilot Plant to be established at CSIRO's Waterford facilities will be designed to reach outputs of this size or greater to demonstrate appropriate material performance within a reasonable timeframe. It will be based on a repurposing of BHP Nickel West's existing nickel sulphate pilot plant at these facilities which led to the current construction of an industrial scale nickel sulphate plant at the Kwinana nickel refinery, south of Perth. While some cobalt sulphate is available from the nickel refinery, additional cobalt sulphate is made available by Cobalt Blue, a producer from NSW. Manganese sulphate is to be provided by Pilbara Metals Group (PMG), or equivalent. Lithium hydroxide for lithiation of the P-CAM is sourced from Tianqi Lithium Australia (TLA). PMG and TLA are both based in the greater Kwinana-Rockingham industrial precinct in Western Australia.

While providing the required levels of outputs for validation in cells, the Pilot Plant also allows for the establishment of concept processes and the development of new intellectual property that is amenable to scale-up. The developed and demonstrated concept processes then have the potential to become the basis for commercial-scale CAM production plants within Australia. This scene setting project has provided the basic technical and supply chain information required for establishing the Pilot Plant.

FBICRC has already committed \$300,000 for the scoping phase of this Pilot Plant project, for verification of technical and processing capability. The final cost of the project will be scoped in this stage but based on industry support to date, it is expected to involve more than \$10M cash and in kind over four years.

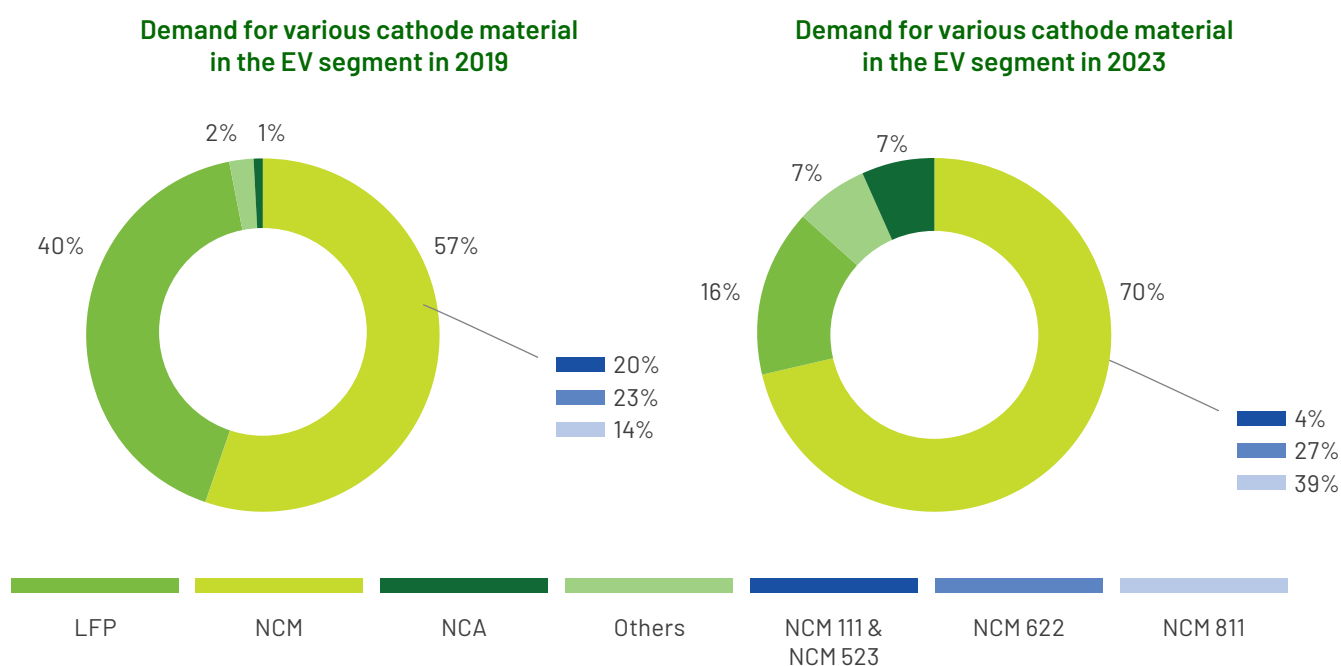


2. Introduction

Australia is rich in mineral resources that are currently required by the lithium-ion (Li-ion) battery industry. In particular, lithium, nickel and manganese minerals are abundant, and a number of mining groups are already advancing in the production of pure salts and their refinement to battery grade quality within Australia. These products are expected to supply the rapidly expanding global demand for vehicle electrification. NCM is of particularly high relevance to the electric vehicle (EV) market, serving 57% of the market demand in 2019 which is predicted to rise even further (see Figure 1). Due to the increasing market demand for NCM materials, it has been chosen as the most potential candidate for Australian CAM manufacture, and as such is the main focus of this Report. The rich domestic abundance of the key elements required for NCM CAM manufacture gives Australia a unique opportunity to decide at what level of the value-add chain it would like to participate, provided that an adequate business case can be built. At a very entry level above the supply of concentrated minerals are the purified metal salts, and above that is the mixed metal hydroxide precursor (P-CAM) product followed by the lithiated final CAM product. Given the strategic importance and economic impact that the battery industry appears it will have, the Australian Government and mining industries have joined efforts to establish the FBI CRC in order to explore, create and expand domestic battery manufacturing capability.

One of the approved Flagship FBI CRC Projects involves the establishment of a CAM Pilot Plant. This Pilot Plant will provide the basis for raw material validation in commercially applicable Li-ion battery cells. Battery manufacturers are required to provide statistically representative numbers

Figure 1 Demand of various cathode materials in the EV market¹

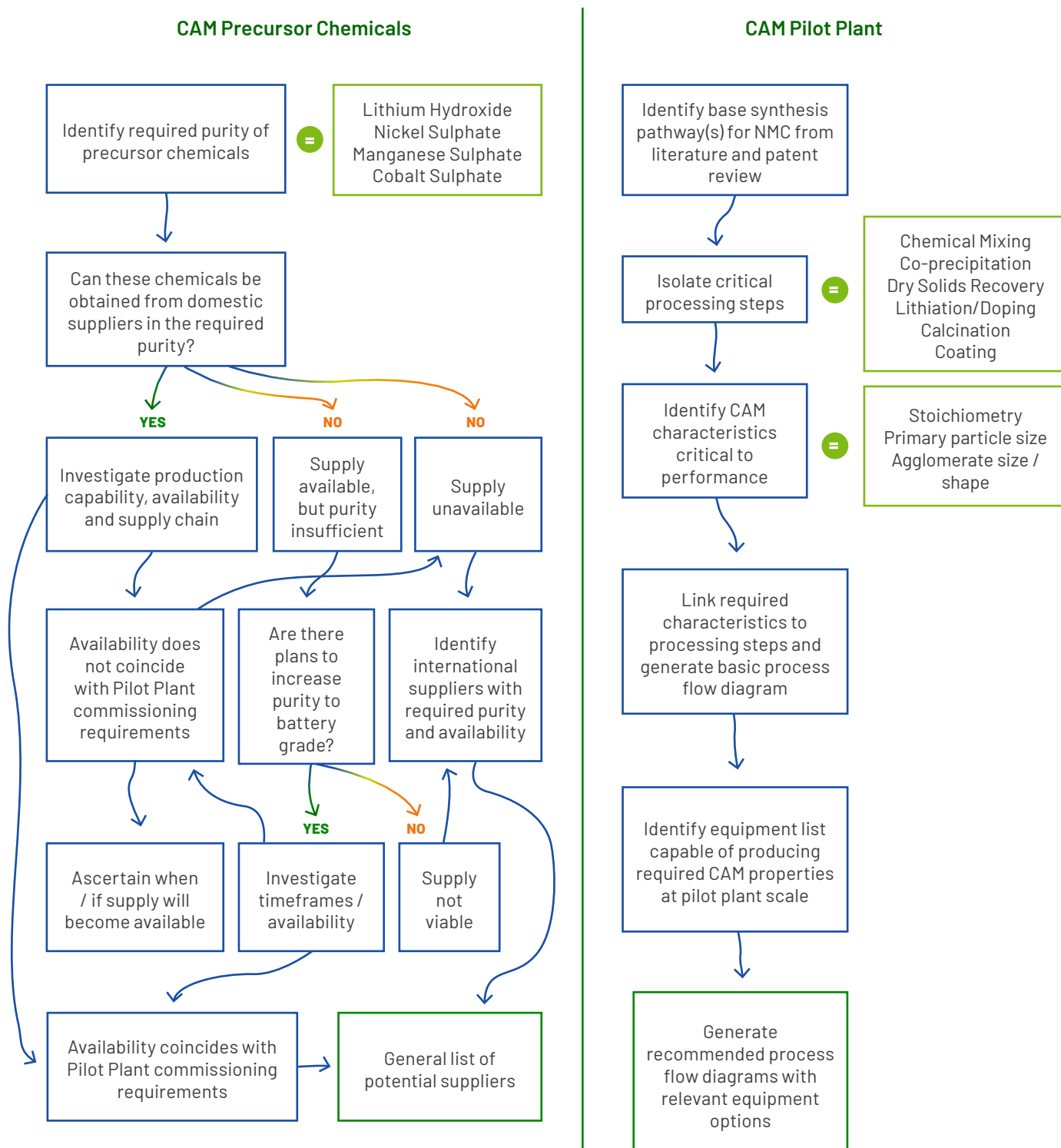


of good performing cells, which could be in the tens of thousands or more, to validate their material is of required quality. This necessitates a basic capability to produce several hundreds of kgs of material with consistent performance characteristics. Thus, the Pilot Plant will be designed to reach outputs of this size or greater to demonstrate appropriate material performance within a reasonable timeframe.

While also providing the required levels of outputs for validation in cells, the Pilot Plant also allows for the establishment of concept processes and the development of new intellectual property that is amenable to scale-up. The developed and demonstrated concept processes then have the potential to become the basis for commercial-scale CAM production plants within Australia. This Project focuses on the basic information required for establishing the Pilot Plant. Figure 2 presents a flow diagram of the main aspects covered by this Report.



Figure 2 Flow diagram for Scene Setting Report objectives



2.1 Project objectives

With regards to raw chemical supply:

- Identify what purity grade of precursor that is required for NCM CAM powder generation (99.9%, 99.99% etc) and what, if any, residual impurities may be critical to avoid.
- Identify the production capabilities of relevant chemical suppliers within Australia.
- If Australian supply is not likely to be feasible by the start date of operation for the larger CAM Pilot Plant, then recommend relevant international suppliers from which the appropriate supply can be obtained in the interim for initial Pilot Plant development.
- Ascertain the likelihood of potential Australian suppliers to produce the required precursor chemicals in the appropriate purity and estimate when supply may become available in each case.

With regards to the Pilot Plant recommendations:

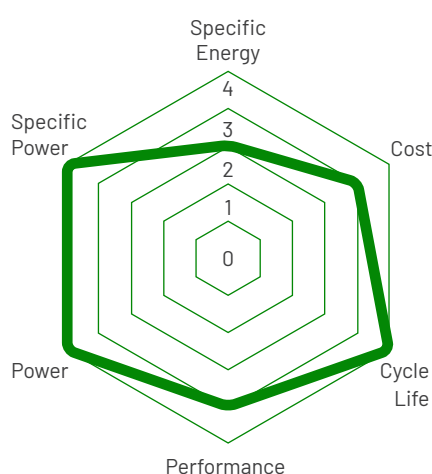
- Identify the base processing pathway for synthesising NCM from public domain.
- Identify what properties of NCM CAM are critical to performance (primary particle size, agglomerate size and morphology, stoichiometry, etc.).
- Identify what processes within the required synthesis steps are key to controlling these factors (i.e. pH control/stirrer speed in controlling agglomerates at the co-precipitation stage).
- Based on this, generate recommendations for suitable processing pathways and the necessary equipment capable of facilitating the control parameters required at pilot plant scale.

The information described above will be complemented by market information to provide context and reference for a value chain analysis, which will target identification of potential margins.

3. Cathode Active Materials

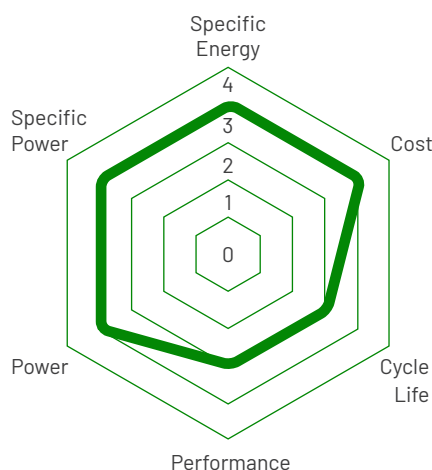
Many different CAM materials exist in the current Li-ion battery market, each with their own unique characteristics arising from the different combinations of elements used to make up their crystal structure. Li-ion cells incorporating the appropriate CAM are chosen to suit the application. For example, materials with high cyclability traded off against reduced energy density are suited to static energy storage applications, while high energy densities are preferred for applications where power-to-weight is critical (for instance EVs). Safety is also becoming an increasingly important factor driving CAM selection. Below is a summary of the most popular CAM materials currently on the market.

3.1 Lithium iron phosphate



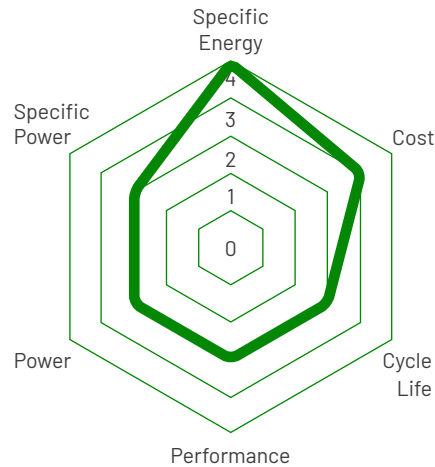
Lithium iron phosphate (LFP) is one of the most stable CAM materials available with great safety and low toxicity. It is also relatively low-cost due to its elemental composition and has good high temperature stability and rate capability. The downside is the material has low energy density compared against other common CAM materials. LFP currently has a large market share of the EV sector (~40%) which is predicted to drop in favour of higher capacity Ni-rich CAM.

3.2 Lithium Manganese Oxide



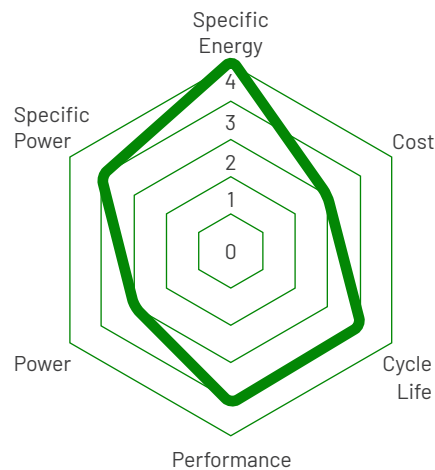
Lithium manganese oxide (LMO) is a spinel-type material and has been used commercially in Li-ion cells since the mid-90s. It has the benefit of low cost and high safety, however, suffers from poor high-temperature performance which is critical for modern cell implementation. As such it has limited use in its pure form but is seeing a resurgence in use as a stability additive in other CAM types. It is also a key component in a new class of 'over-lithiated oxide' (OLO) CAM which although promising, is still not market ready.

3.3 Lithium cobalt oxide



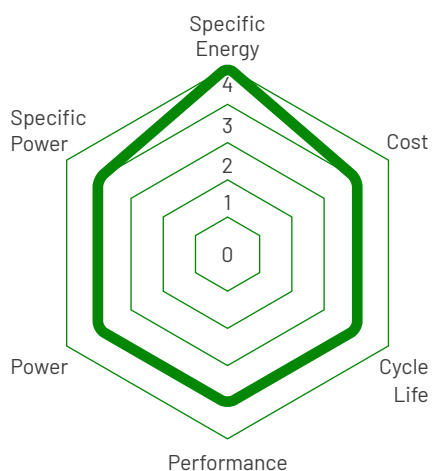
Lithium cobalt oxide (LCO) has a high specific energy and as such sees use mainly in consumer electronics. It has a very poor cycle life and low thermal stability which limits its discharge capability. Consequently, discharging LCO cells beyond their current rating can lead to overheating with risk of combustion and they are considered to have poor safety. LCO is also expensive due to the high cost of cobalt. This combination of properties makes LCO unsuitable for EV applications.

3.4 Lithium nickel cobalt aluminum oxide



Lithium nickel cobalt aluminum oxide (NCA) has one of the highest capacities among commercially implemented CAM materials. This combined with its high operating voltage gives NCA an exceptional energy density (220 Wh/kg). Because of these properties, NCA is the CAM of choice for Tesla's current EVs. NCA does however suffer from high cost due to complicated processing and cobalt content. Cell safety can also be an issue if the appropriate battery management is not implemented.

3.5 Lithium nickel cobalt manganese oxide



Lithium nickel cobalt manganese oxide (NCM) is similar to NCA in structure, with manganese replacing aluminum as the structural stability component. With a high capacity (which is still rising as research on the material continues) and operating voltage combined with great all-round performance characteristics, it has the highest uptake in the EV market. Its desirability stems from the ability to vary the elemental composition of the material and control its properties which can be tailored to suit requirement. This material is expanded on further below.

NCM battery types are generally identified by their varying nickel, manganese and cobalt composition. Nickel is responsible for energy density and specific capacity, while cobalt and manganese are present for structural stability, increasing cycle life and safety. Increased nickel content therefore has the benefit of increasing energy density and capacity while lowering costs through cobalt replacement. However, this is at the cost of reduced structural stability and cycle performance. This reduced stability is rectified by doping and/or surface coating of the material. Table 1 summarises the main characteristics of NCM batteries.

Table 1 Summary of different NCM chemistries¹

Item	Actual Capacity mAh/g	Advantages	Disadvantages
NCM 111	150	<ul style="list-style-type: none"> Relatively balanced energy density, cycle performance, and safety performance 	<ul style="list-style-type: none"> Relatively low first charge/discharge efficiency Has cation mixing Relatively low discharge potential
NCM 523	160	<ul style="list-style-type: none"> Relatively high specific capacity Good thermal stability 	<ul style="list-style-type: none"> Poor balance between cycle performance, discharge performance under different currents, self-discharge, etc.
NCM 622	170	<ul style="list-style-type: none"> Good processability High energy 	<ul style="list-style-type: none"> Cycle performance is relatively poor
NCM 811	200	<ul style="list-style-type: none"> High capacity Low cost 	<ul style="list-style-type: none"> Requires strict production conditions Product easily absorbs moisture
NCA	200	<ul style="list-style-type: none"> High capacity 	<ul style="list-style-type: none"> Requires strict production conditions Product easily absorbs moisture Not stable

3.5.1 Stoichiometry

The most common NCM CAM compositions available are as follows:

- NCM 111: made up of one third of each of nickel, cobalt and manganese. Historically chosen for their stability and durability. The NCM 111 battery suffers from relatively poor energy density due to the low nickel content. This chemistry is also comparatively expensive due to the high cobalt content which is currently driving the development of improved battery types with lower cobalt. The NCM 111 battery is considered the simplest NCM battery type to manufacture with a long-term history of development. Current production of NCM 111 is very limited due to lack of market demand.
- NCM 523: the next commercial iteration after NCM 111. Similarly, has high thermal stability but poor energy density due to lack of nickel and increased cost due to high cobalt content.
- NCM 622: made up of 60 mol% nickel, 20 mol% manganese and 20 mol% cobalt. Currently a widely used battery type with long term stability when doped and coated appropriately. A 65 mol% nickel analogue is also present on the market due to lowered cost from reduction in cobalt content.
- NCM 811: made up of 80 mol% nickel, 10 mol% manganese and 10 mol% cobalt. Currently becoming a widely used battery type with the higher nickel content improving various battery properties such as capacity, tap density and volumetric energy density. The low cobalt content is a driving consideration due to the high cost and lack of availability of cobalt. The NCM 811 battery chemistry is more sensitive to environmental conditions than the NCM 111, NCM 523 or NCM 622 types, and there are difficulties in the production, handling and storage of this material. As with NCM 622, doping and coating of the material is required for it to be feasible in Li-ion cells. There is also a relatively new NCM 811 CAM product with further nickel increase up to 83 mol%, with current drivers pushing nickel content even higher.
- NCA: technically not an NCM analogue as it does not contain manganese. However, as nickel contents continue to trend upwards in NCM formulation, the difference between NCA and NCM begins to become negligible, with the processing conditions for each being nearly identical. Nickel, cobalt and aluminium make up the main composition of the cathode typically in an 84/12/4 ratio respectively, though nickel contents are being pushed up to increase capacity and reduce costs through cobalt minimisation.

3.5.2 Morphology

NCM CAM also exists in two distinct morphology analogues, these being traditional polycrystalline materials and single crystalline materials.

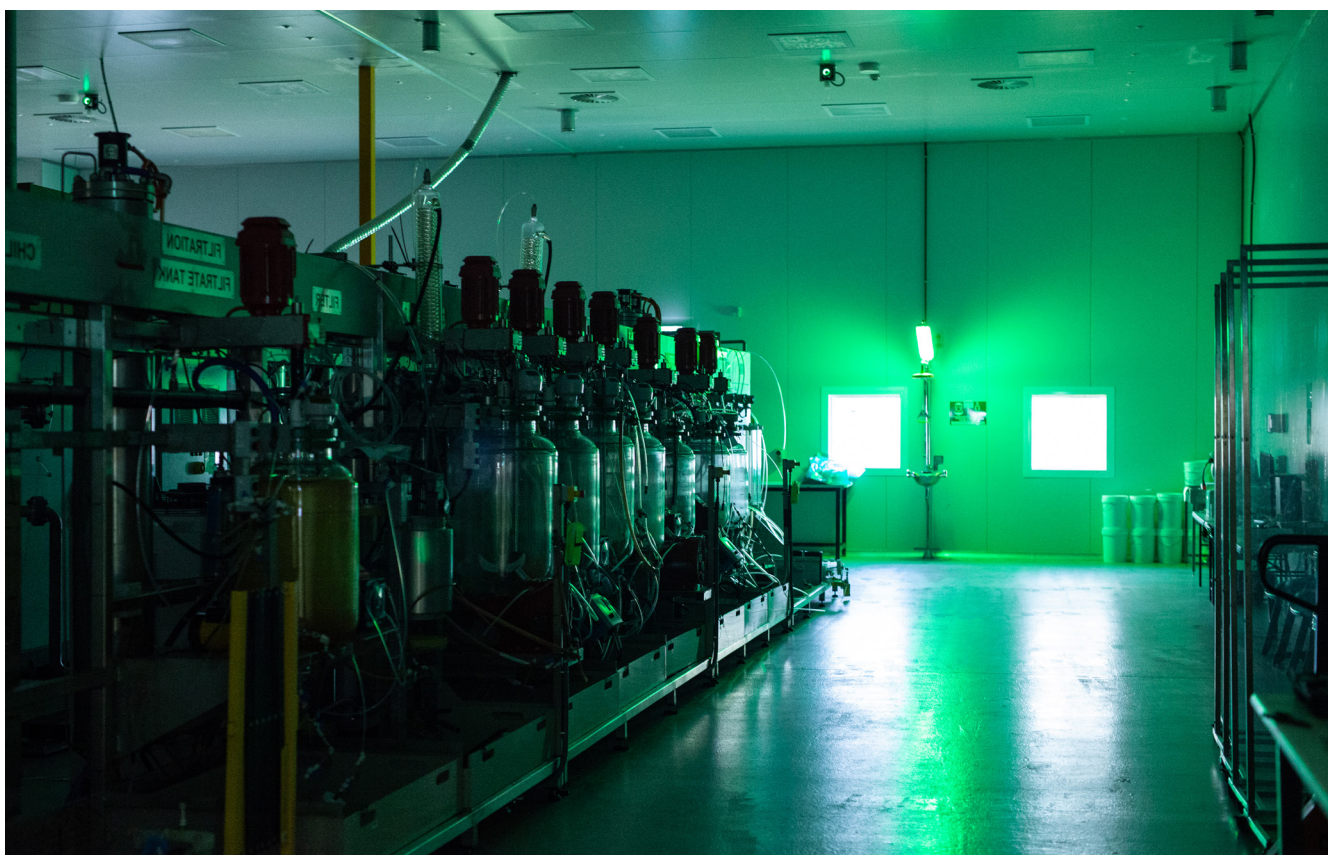
3.5.2.1 Polycrystalline

Polycrystalline materials consist of agglomerates (or secondary particles) of smaller primary particles. These types of materials commonly consist of secondary particle agglomerates of ~10 µm made up of nano-sized

primary particles (~400 nm). The size of the secondary agglomerates varies depending on end-user requirements. For example, a common current market requirement is a bi-modal secondary particle distribution consisting of a mixture of 4 and 14 μm secondary particles.

3.5.2.2 Single crystalline

Single crystal NCM is produced with a larger primary particle of around 3–4 μm with minimal and ideally no agglomeration, producing large grain size single particles. Similarly, 3–4 μm agglomerates can be formed which are then subjected to a longer calcination period to facilitate grain boundary elimination between primary particles. A large grain size enhances structural stability during cycling (especially at high temperature) and increases cycle life, with reduced gas formation during operation. However, this is at the trade-off of reduced energy density and increased price. As such single crystalline CAM is desirable in applications where safety and cyclability are important, and NCM 523 in single crystalline form is still commercially produced due to demand for the product. Single crystalline CAM is particularly useful in cell formats with poor structural integrity such as pouch and prismatic cells due to a reduction in bloating. It is likely that due to the requirement for increased safety and cyclability in EV applications utilising prismatic format cells, that single crystalline materials will become increasingly popular on the market in the future.



Li-ion battery cathode manufacture in Australia

4. Precursor Chemicals

The focus of this section is to determine:

- What chemicals are required for NCM cathode synthesis?
- What raw material specifications are required for each of these precursor chemicals?
- How are they produced?
- What is the current state of domestic production for each of these chemicals?

4.1 Processing and relevant chemicals

Many different synthetic pathways exist for the formation of NCM CAM, of which the most commonly employed include but are not limited to solid state mixing, sol-gel, co-precipitation and hydrothermal techniques. In general, solid state mixing techniques are not amenable to precision stoichiometric control as is required by high performance NCM compounds, and thus this technique will not be explored here. Hydrothermal synthesis is also excluded due to complications in process scaling. Both sol-gel and co-precipitation synthesis techniques provide the greatest degree of process control for the critical performance characteristics of NCM. Both these processes also possess ease of scalability and thus are the best options for CAM Pilot Plant. This Report will focus solely on the co-precipitation method as it is currently an industry standard with a proven track record.

As such, Australian mineral suppliers have focused their efforts on producing the relevant metal sulphates commonly used as the precursors to the co-precipitation technique. It is therefore the recommendation of this Report that the Pilot Plant implement this technique for NCM CAM powder generation to ensure compatibility with domestic chemical suppliers and end-user requirements.

The raw chemicals required to form CAM include the following:

- Nickel sulphate
- Manganese sulphate
- Cobalt sulphate
- Lithium hydroxide.

Other metal salt precursors can be used such as carbonates or oxalates. However, due to the maturity of the sulphate process combined with current domestic chemical refining trends, the metal sulphates will be the focus of this Report. The quality of these raw materials is critical for the manufacture of quality NCM cathode materials. The raw materials should be sourced as high-quality battery grade precursor feed materials, which require no pre-treatment prior to use.

The current supply chain identified for these raw materials indicates that none are currently produced in Australia for commercial sale at the required scale for the FBI CRC Pilot Plant. However, a number of Australian mining companies are currently bringing online the capability to produce battery

grade metal sulphates and lithium hydroxide on continent for potential use in domestic CAM production.

As mentioned previously, commercial production of NCM CAM has two distinct product tiers resultant from the processing stages required to make the material. These are the mixed metal hydroxide CAM precursor (P-CAM) which does not contain lithium, and the final CAM product which does contain lithium and is ready for incorporation into battery cells. It is not uncommon for manufactures to only produce P-CAM which is sold on to other manufacturers for lithiation. As will be discussed in the processing section of this Report, P-CAM production is primarily wet chemical processing while CAM production is mainly focused on calcination techniques. CAM production methodology varies substantially with changing stoichiometry and becomes increasingly complicated with rising nickel content, while P-CAM processing remains mostly the same regardless of the stoichiometry.

4.2 Nickel sulphate

Nickel sulphate is the primary precursor raw material in NCM batteries. Its demand in battery applications has risen significantly in recent years not only because of increased use of NCM and NCA batteries, but also due to the introduction of more nickel-rich cathode compositions (such as NCM 811 and NCM 622).

Nickel sulphate is produced from a variety of nickel intermediates, including nickel matte, MSP, MHP, or using high-purity nickel products (Class I nickel) in the form of powder or briquettes. Class I nickel is categorised by having a purity greater than 99%, and the output of such high-quality product must be accelerated in the next decade to meet the expected increase in demand driven largely by the battery industry.

4.2.1 Battery grade specifications

High-purity feedstocks (Class I nickel) or various nickel intermediates are necessary for the production of battery-grade nickel sulphate. Control over the concentration of impurities in precursor materials is essential in maintaining chemical stability and performance in batteries. Several mass compositions of nickel sulphate used in worldwide Li-ion battery industries are shown in Table 2. In addition to compositional requirements, the product must also comprise uniform and fine crystals with zero-level moisture and no dust contamination².

Table 2 Sample nickel sulphate battery grade specifications⁵

Element	IGO	Europe 1	Europe 2	China 1	China 2	China 3	WA 1	Others
Ni (%)	22.1-22.3	22.3	22.3	22.2	22	22.2	22.3	22.2
Co (ppm)	40-140	2	10	30	10	30	80-120	100-250
Cu (ppm)	1-9	1	2	1	10	1	1	5
Al (ppm)	1	1	-	10	-	10	1	-
As (ppm)	1-2	1	-	-	2	-	-	-
Ca (ppm)	1	1	-	7	5	7	2	5-10
Cd (ppm)	1	1	2	2	10	1	1	-
Cr (ppm)	1	1	-	3	-	3	1	5-10
Fe (ppm)	1-6	1	2	4	10	3	1	5
K (ppm)	1	1	-	-	-	-	1	5
Mg (ppm)	1-4	1	-	20	-	20	1	5-10
Mn (ppm)	1	1	-	-	-	-	1	5-10
Na (ppm)	1	5	-	30	-	30	5	-
P (ppm)	1	2	-	-	-	-	-	-
Pb (ppm)	1	1	2	6	10	5	1	5-10
Si (ppm)	1	5	-	10	-	10	2	-
Zn (ppm)	1-5	1	2	2	10	1	1	5

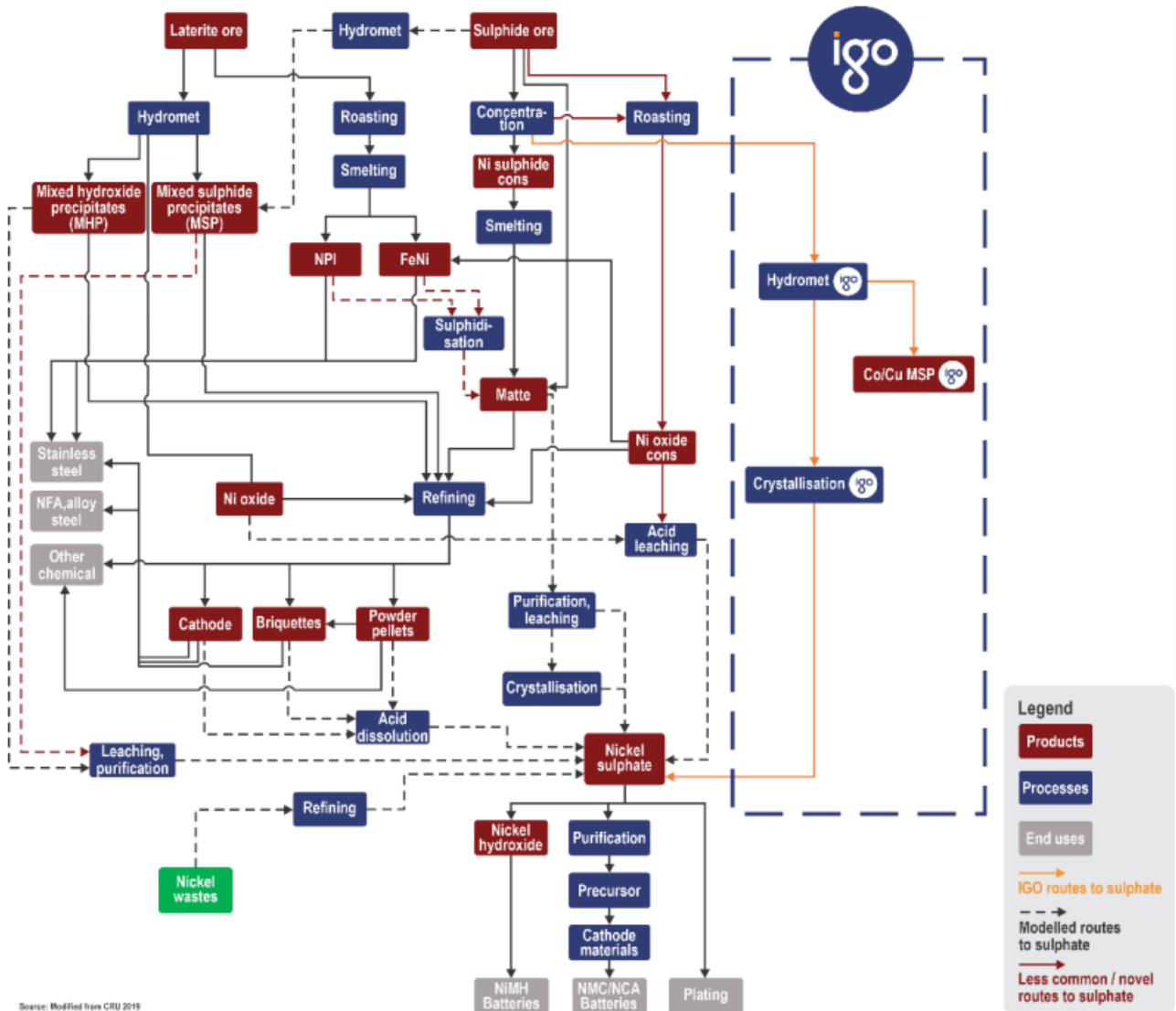
Modified from multiple source. The character "-" represents zero or unknown data

4.2.2 Process method

Several commercial options exist for the production of battery-grade nickel sulphate. Figure 51 outlines the current metallurgical processes for nickel sulphate and also shows the method proposed by IGO, which has recently completed the pre-feasibility stage of engineering.

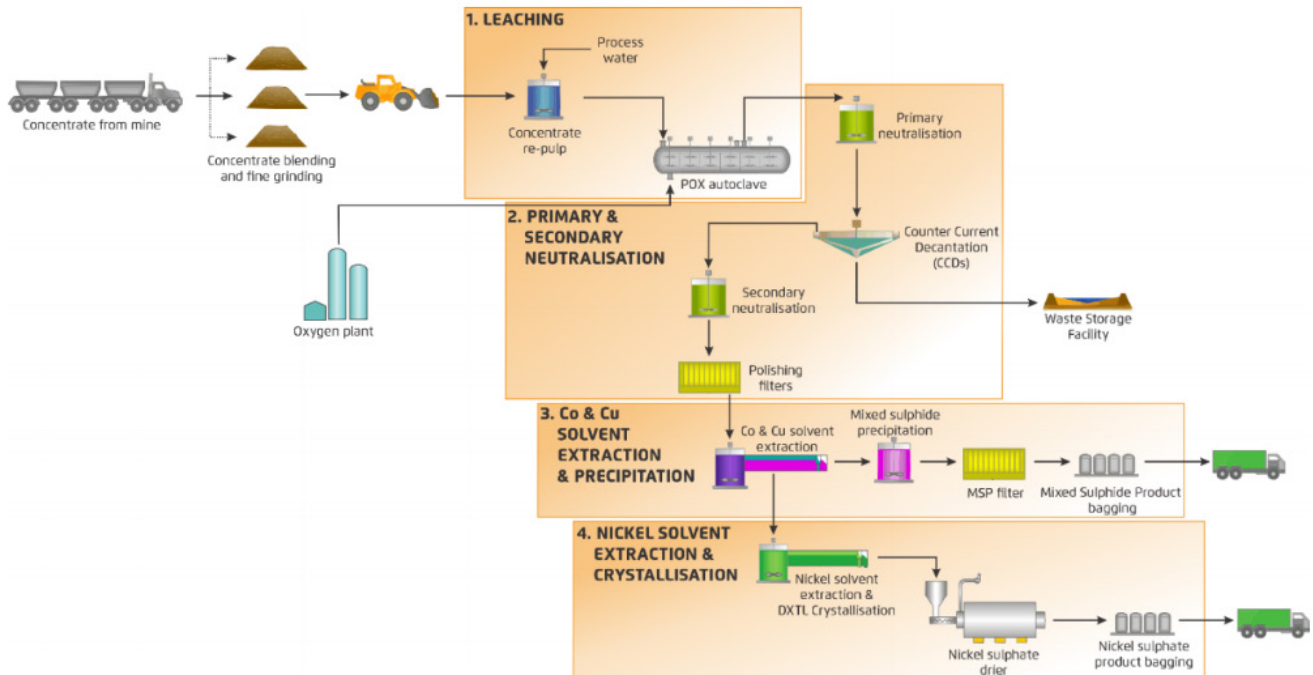


Figure 3 Current processing methods for nickel sulphate³



Due to the significant presence of sulphidic nickel resources in Australia, most nickel production is restricted to pyrometallurgical processes (i.e. smelting) and requires a very large capital investment, thus making it difficult for new players to enter the market. Therefore, the availability of nickel sulphide could be greatly increased by the development of new low-cost processing methods for sulphide ores. Hence, IGO's proposed hydrometallurgical route is of significant commercial interest. A more detailed flowsheet of IGO's downstream nickel sulphate process is shown in Figure 4.

Figure 4 IGO's flowsheet to convert sulphide nickel concentrate to nickel sulphate³



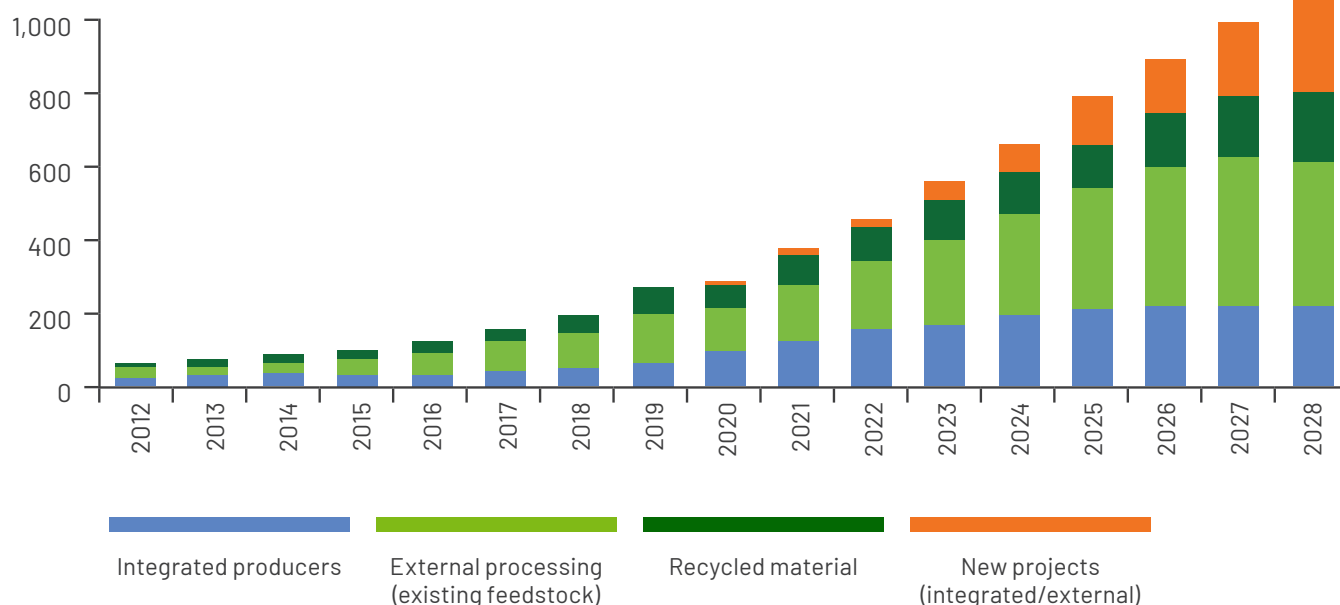
4.2.3 Supply overview

Most precursor producers are located in Japan, South Korea and China. The majority of nickel sulphate is produced in Japan and China, although additional supply is sourced from other producers including Nornickel's Harjavalta plant in Finland, Umicore's Olen plant in Belgium, and three smaller producers in Taiwan. Less significant producers are also active in India, Germany, and the United States⁴. The supply outlook until 2028 for nickel sulphate is shown in Figure 5, which illustrates the assumed boost in production from the commissioning of new greenfield projects. In addition, production from recycled material would also become important in meeting the future demand for nickel sulphate.

Australian supply of nickel sulphate is expected to rise in response to pressure from the battery market. BHP Nickel West is currently constructing a nickel sulphate plant at the Kwinana Nickel Refinery. Stage 1 is projected to produce up to 100 kt/a of battery-grade nickel sulphate⁵. The process involves the smelting of concentrates from Mt Keith, Leinster, and Kambalda to produce a nickel sulphide matte with roughly 47% Ni. The matte is then granulated and refined in Kwinana into premium-grade powder and briquettes containing 99.8% nickel, which is to constitute the Class I feedstock for the nickel sulphate plant.



Figure 5 Outlook for supply and demand of nickel sulphate, 2012-2028 (kt Ni)⁴



4.2.4 Industry players and refining capacity

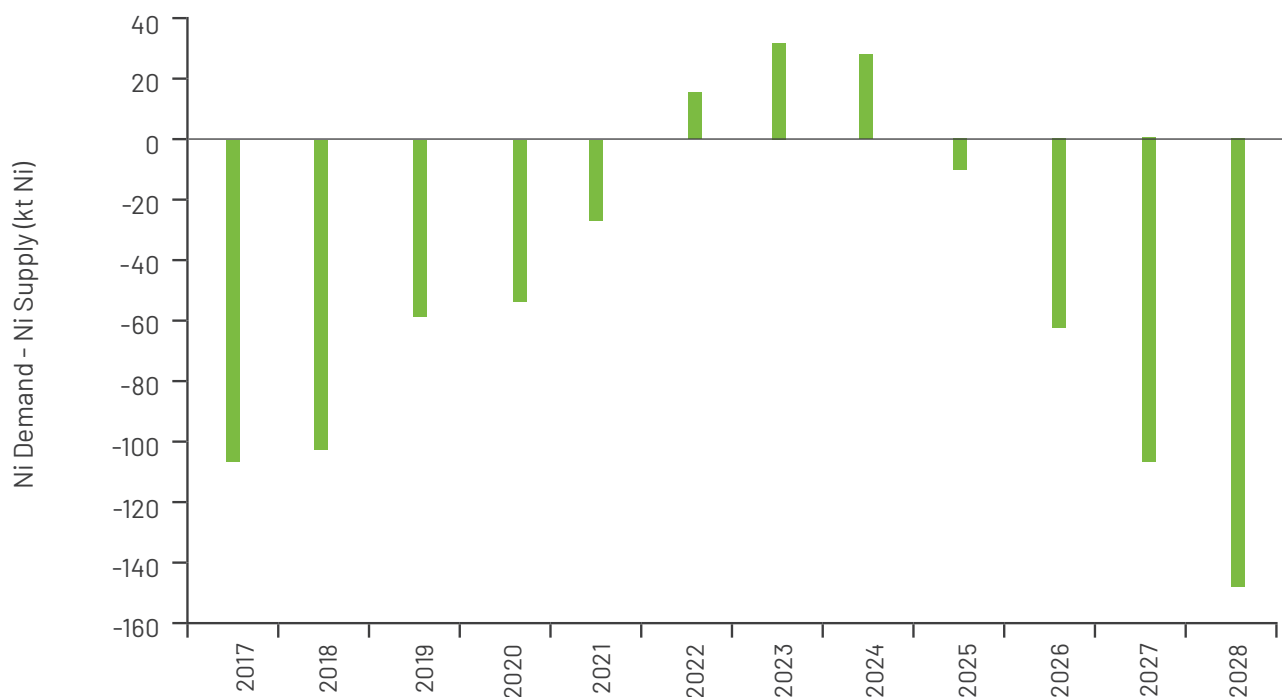
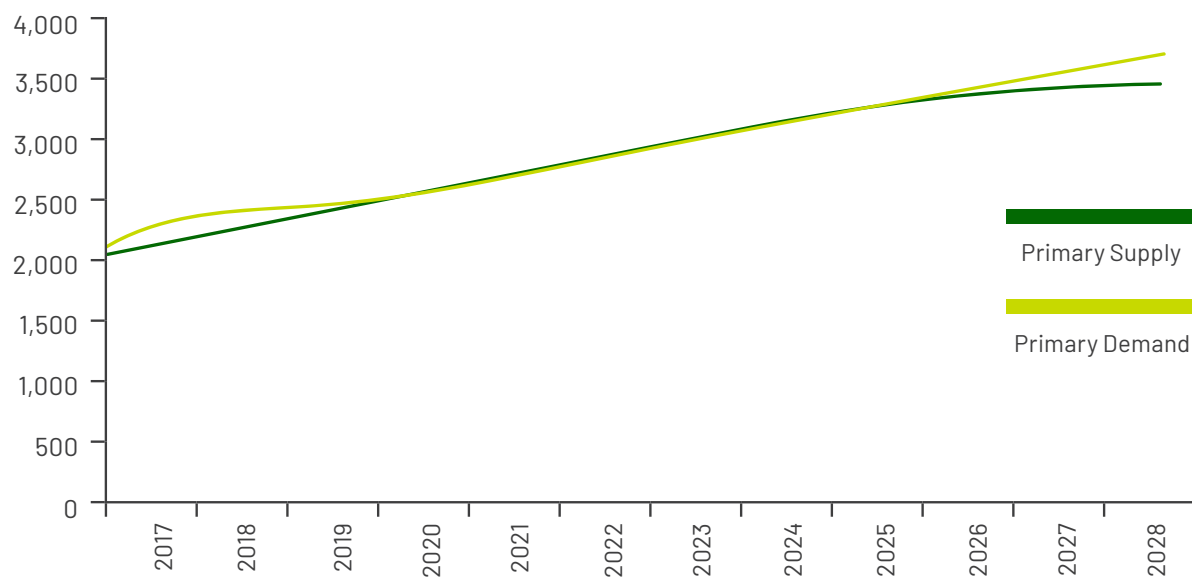
According to SMM (2020)⁶ global refining capacity for nickel sulphate was 1.39 Mt in 2019, with China comprising over 43%. Capacity is expected to increase substantially to a total of 2.5 Mt by 2022, amounting to a compound annual growth rate of 12%⁶. The following key industrial players are expected to contribute to much of the capacity growth:

- Jinchuan Group (China) – currently 80 kt/a nickel sulphate, reportedly adding 25 kt/a by early 2020⁷
- BHP Nickel West (Australia) – 100 kt/a nickel sulphate plant commissioning expected second half (H2) of 2020
- Terrafame (Finland) – currently 170 kt/a nickel sulphate⁸
- Various wet processing operations in Indonesia.

4.2.5 Market outlook

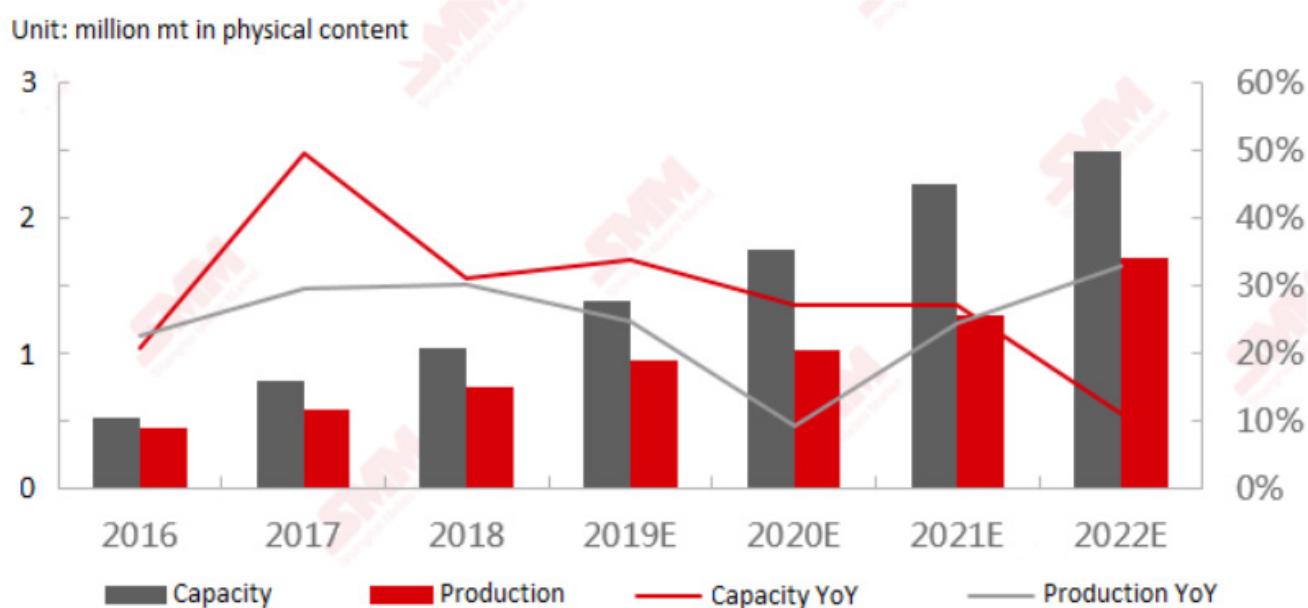
The rapid growth of the battery industry over the last decade led to a significant accumulation of nickel stocks and, in turn, a corresponding fall in nickel prices. This price fall has limited the economic incentive for projects to produce high-grade intermediates or Class I nickel, thereby contributing to the supply shortage of nickel observed in recent years.

According to Roskill (2019), the global nickel market recorded a deficit of 102 kt in 2018 which is predicted to narrow to just 27 kt by 2021⁴. Following this, however, Roskill predicts that the nickel shortage will reemerge due to rising consumption and lack of new investments. Figure 6 shows the estimated market balance (demand - supply) for nickel from 2017-2028. As a whole, however, the nickel market is expected to steadily increase over the next decade with demand rising from 2503 kt to 3538 kt from 2020 to 2028, respectively (see Figure 7).

Figure 6 Estimated market balance for nickel, 2017-2028⁴**Figure 7** Estimated supply and demand for nickel, 2017-2028⁴

Predicted trends in the nickel market as a whole are expected to be similarly reflected in the nickel sulphate market. Moreover, the increased requirements for high-grade feedstocks in precursor production may emerge as a bottleneck in the supply of nickel sulphate. Despite possible shortages, global production of nickel sulphate is projected to increase by over 80% from 0.94 Mt in 2019 to 1.7 Mt in 2022⁶. Figure 8 shows the projected increase in capacity and production for nickel sulphate as well as Year-Over-Year measurements for each metric.

Figure 8 Estimated capacity and production for nickel sulphate, 2016-2022⁶



4.2.6 Australian supply and availability

BHP Nickel West – nickel sulphate refinery expected operational by Q2 2021. BHP Nickel West has expressed interest in supplying the FBI CRC CAM Pilot Plant with nickel sulphate, and have some stock remaining from piloting available for lab-scale trials.

IGO – announced in November 2019 that the downstream nickel sulphate facility will not proceed to detailed engineering stage due to renewed offtake agreements⁹.

4.2.7 International supply and availability

In the event that supply from BHP Nickel West is delayed then nickel sulphate can be sourced from Jinchuan (China), Jilin Jien Nickel (China), or Umicore (Belgium).

4.3 Manganese Sulphate

In 2018, consumption of manganese sulphate and oxide for battery applications was 41.1 kt, comprising only 0.2% of the overall manganese market. Manganese sulphate and oxide are distinctly used in the synthesis of NCM and LMO batteries, respectively. Therefore, the focus of this report will be on manganese sulphate as a precursor material.

The large scale of the manganese market (18.6 Mt in 2018), availability as a feedstock, high processing capacity, and low-cost compared to other precursor materials make it a relatively low risk component in the supply chain for Li-ion batteries. Manganese sulphate is therefore used to replace part of the cobalt content in NCM batteries to reduce manufacturing costs.

4.3.1 Battery grade specifications

For NCM CAM, high-purity manganese sulphate monohydrate (HPMSM) is used as a feedstock. Note that HPMSM differs from other lower product grades which are commonly used for agricultural applications (see Table 3 for sample specifications). Manganese sulphate for battery applications must have a minimum Mn concentration of 31.8% by weight¹⁰. Impurities such as Ca, Mg and Na have negative impacts on high-temperature morphology and cycling performance of Li-ion batteries.

Table 3 Sample manganese sulphate battery grade specifications¹⁰

Items	Units	Specifications
Mn	%	31.8% min
Pb	ppm	10 max
As	ppm	10 max
Cd	ppm	10 max
Ca	ppm	50 max
Mg	ppm	50 max
Na	ppm	50 max
Fe	ppm	10 max
Zn	ppm	10 max
Cu	ppm	10 max

Another key element in manganese precursor production is selenium (Se), as it can dictate the viability of different feedstocks. Selenium is added in the production of electrolytic manganese metal (EMM – 99.7% Mn) to suppress hydrogen evolution and improve current efficiency during electrolysis. Unfortunately, selenium reports to the cathode and contaminates the EMM product. Due to safety concerns about the high toxicity of Se-compounds (namely hydrogen selenide), direct HPMSM production from EMM is not considered a suitable processing option. Furthermore, exposure to the toxic effects of selenium can be extremely hazardous for product handlers of downstream battery manufacturers¹¹.

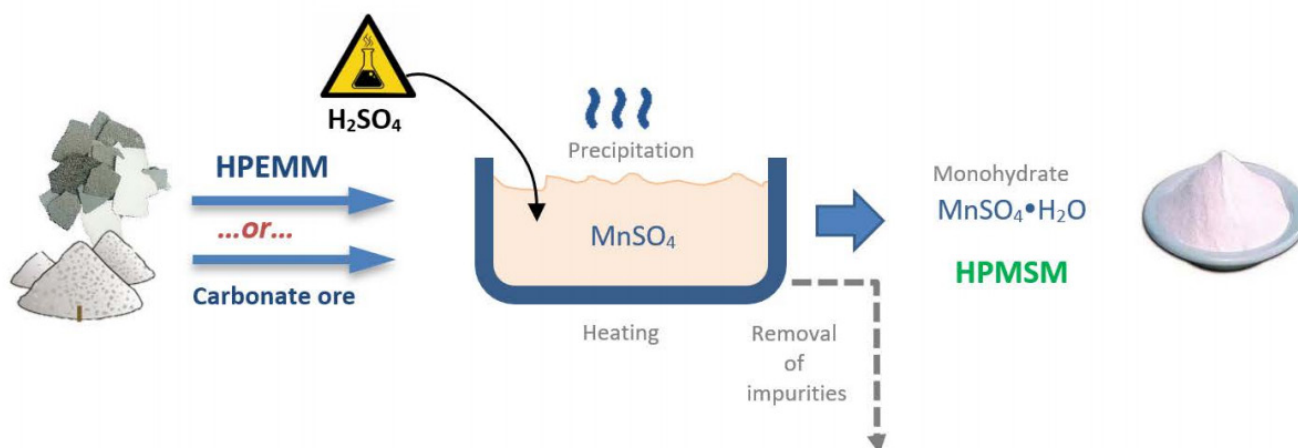
Therefore, EMM must first be converted into 'Se-free' 99.9% EMM (HPEMM) before being suitable as feedstock for HPMSM production.

The high demand for HPMSM has led to the recent development of effective processing technologies for using HPEMM as a feed in many Chinese refineries⁴. A key requirement for battery makers using HPEMM is to limit the concentration of selenium to 3 ppm (conventional EMM typically contains 1800 ppm Se).

4.3.2 Process method

HPMSM is produced by either direct leaching of carbonate ores with sulphuric acid or by conversion from HPEMM. A schematic of the production process is shown in Figure 9. Oxide ores can also be used as a feedstock although they require additional reductive processing to lower manganese into a soluble oxidation state (Mn^{2+})¹². Solid waste generation is, however, reduced significantly (up to 80%) with the use of oxides compared to carbonates due to the absence of calcium and magnesium carbonates which constitute much of the leach residue⁴.

Figure 9 Schematic of HPMSM production from HPEMM or carbonate ores¹²



The production of HPEMM involves the electrolysis of manganese sulphate solutions containing ammonium sulphate, followed by the addition of sulphur dioxide to achieve the high purity. Production of HPMSM via HPEMM is considered an effective processing strategy as it prevents the carryover of impurities from electrolytic solutions to the final product. Moreover, direct ore processing relies heavily on the consistency of feedstock quality and may require additional process steps to control impurities and meet product specifications. At least three Chinese suppliers use HPEMM in the process shown in Figure 9.

Despite the disadvantages, the scarcity of high-quality carbonate ores and recent push to reduce solid waste generation has led to an increase in the direct processing of oxide ores, particularly throughout China. As

mentioned, reductive hydrometallurgical (leaching) or pyrometallurgical (calcination) processes must be employed for oxides to allow manganese to dissolve in the acid solution. The proposed flowsheet for oxide ore processing for the Butcherbird Manganese Project in Western Australia is shown in Figure 10¹³. Note that the process is to be designed to allow for flexible alternation between HPMSM and EMM as final products, depending on market demands. Manganese Metal Company's (MMC) flowsheet for converting oxide ores into HPEMM is also shown in Figure 11. Their refinery in South Africa exports over 30 kt/a to various NCM cathode producers.

Figure 10 Simplified flowsheet of HPMSM production from oxide ore for the proposed Butcherbird Project in Western Australia¹³

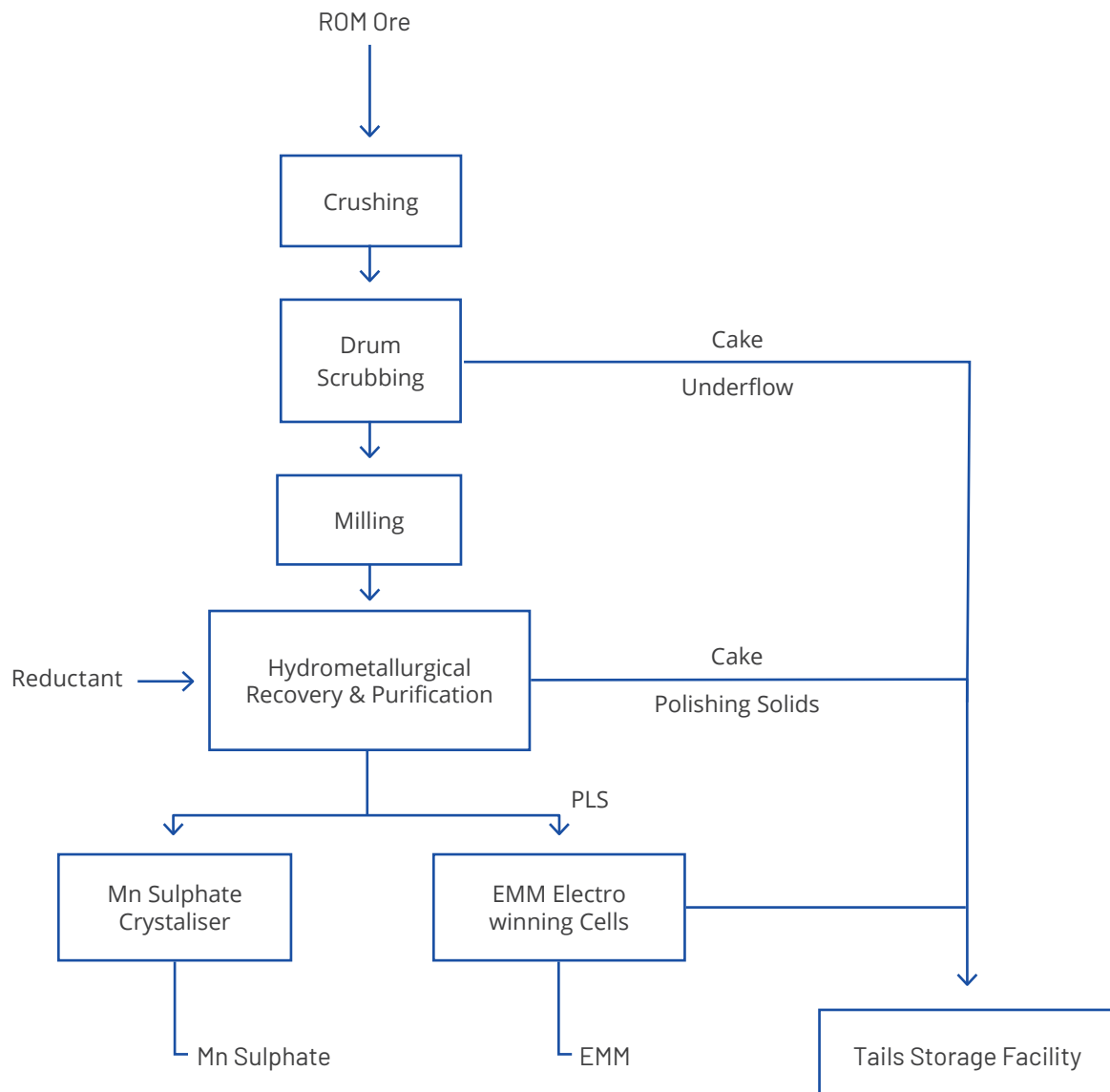
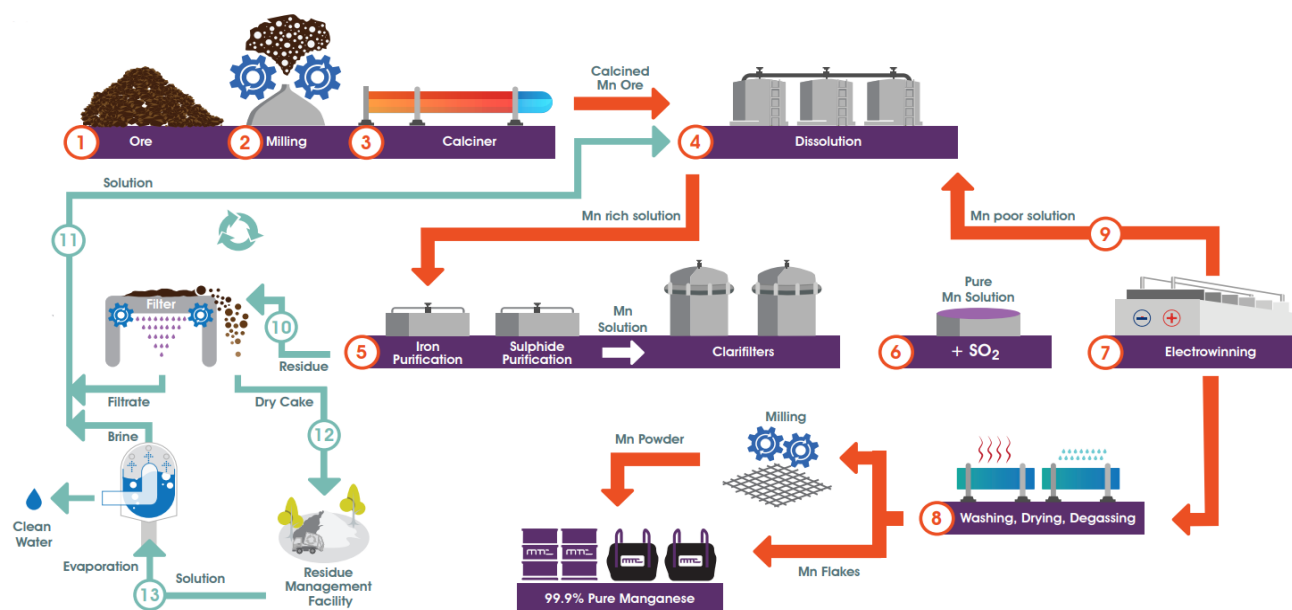


Figure 11 MMC's flowsheet for HPEMM production from oxides ores in Nelspruit, South Africa¹¹



4.3.3 Supply overview

Global production of HPMSM was estimated at 28 kt in 2017, comprising only 17% of the total MSM output¹². Over 87% of HPMSM is produced by Chinese suppliers, with seven plants in operation and several currently under construction. There are also singular precursor production facilities in the USA, Mexico, Japan and Belgium. Domestic supply is projected to become available to world markets in 2020 with the anticipated completion of Pilbara Metals Group's 40 kt/a plant in East Rockingham, Western Australia¹⁴. Finalisation of project funding could likely influence PMG's planned starting date of production.

4.3.4 Industry players and refining capacity

Major producers in China include CITIC Dameng Mining, Guizhou Redstar, Haolin Chemical and several others. CITIC's refining capacity is estimated at approximately 15 kt/a¹⁵. Outside China, the only significant producer of HPMSM is Prince Erachem, with production facilities in Mexico and Belgium. Nippon Denko in Japan is also producing very small amounts of HPMSM from imported EMM. MMC's 30kt/a HPEMM refinery in Nelspruit, South Africa exports 95% of production to worldwide industries, much of which is re-dissolved to generate HPMSM for Li-ion batteries.

Production capacity is expected to increase considerably in the next five years, primarily as a result of China's plan to expand capacity of HPMSM from 100 kt/a to 940 kt/a over the next 3-4 years¹². Refining capacity is also projected to grow from the output of several potential new players entering the industry, including:

- Euro Manganese Inc (EMN) claiming to begin production of both HPMSM and HPEMM in the Czech Republic by 2022 at 50 kt/a Mn. The project

plans to reprocess fine-grained tailings from historical pyrite mining operations¹⁵.

- Pilbara Metals Group (PMG) proposing to produce 40 kt/a of battery grade HPMSM by 2020 from oxide ores in Western Australia¹⁴.

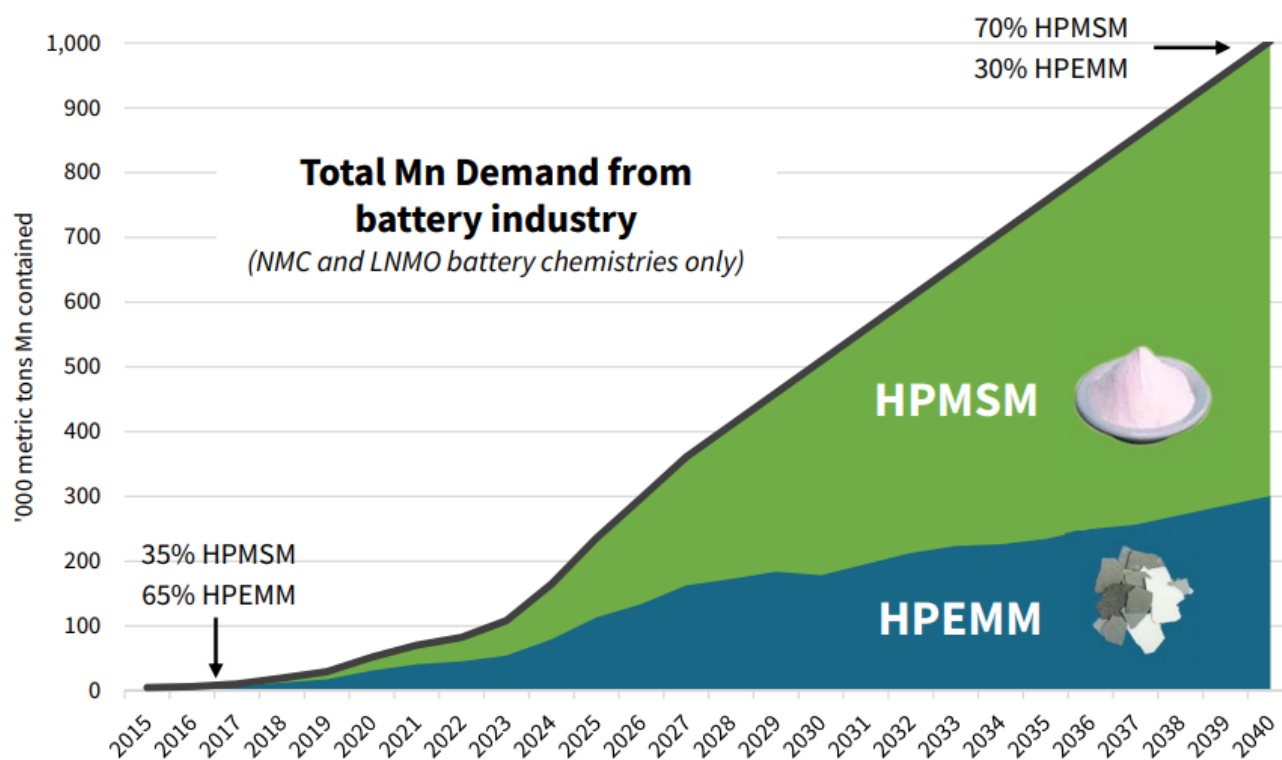
Element 25 is currently in the pre-feasibility stage for the Butcherbird Project in Western Australia. Starting date and production rate not released, although the oxide ore reserve is the largest onshore manganese resource in Australia and estimated to be >260 Mt¹³.

4.3.5 Market outlook

4.3.5.1 HPMSM demand

The demand for manganese from the battery industry, according to CPM (2019), is predicted to increase 80-fold between 2017 and 2037¹⁶. Figure 12 illustrates this economic forecast, showing the relative contributions from HPMSM and HPEMM to the overall demand.

Figure 12 Estimated market demand for HPMSM and HPEMM in the battery industry, 2015-2040¹⁶



While the trend toward more nickel-rich NCM CAM chemistries is expected to impact the market demand for both cobalt and manganese, the outlook for manganese remains promising for several reasons, including:

- The shift toward high-nickel NCM CAM may not be as rapid as expected, with NCM 622 and 523 holding large portions of the market until 2025⁴.

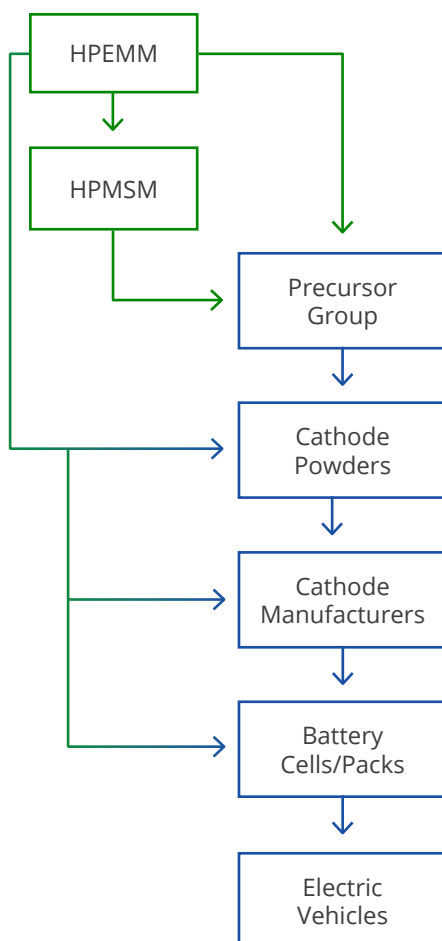
- BASF is reported to be developing a cathode chemistry with 70% manganese, 20% nickel and less than 5% cobalt, expected to be in production by 2021.
- The recent development of lithium-sulphur batteries with manganese dioxide nano-sheets in a 25wt% manganese/sulphur cathode. Such chemistries have the potential for greater energy storage at a reduced cost¹⁷.

4.3.5.2 HPMSM supply

Supply of HPMSM is expected to meet the growing demand with several greenfield projects under construction coupled with the upgrading of many production facilities to accept different feedstocks.

With the relatively low capital intensity of converting HPEMM to HPMSM and forecast pricing premia (\$6850 AUD/t HPEMM vs \$12 350 AUD/t HPMSM), it is likely that industry players will internalise conversion processes and allow for HPEMM as primary feedstock¹⁵. This emergence of HPEMM into the HPMSM supply chain is illustrated in Figure 13.

Figure 13 HPEMM entry into HPMSM supply chain¹⁵



4.3.6 Australian supply chain and availability

Pilbara Metals Group (PMG) plan to begin production of HPMSM at a rate of 40 kt/a by 2020. The refinery is planned to be located at East Rockingham, Western Australia.

Preliminary discussions with PMG indicate that they will be able to provide 10-20 kg of battery grade manganese sulphate to the FBI CRC Pilot Plant Project for lab-scale CAM synthesis trials by May 2020. More material may be available for pilot-scale CAM production as PMG ramp up their production capabilities.

4.3.7 International supply and availability

Outside China, Prince Erachem is the only current producer of HPMSM with refineries in Mexico and Belgium. Several companies (i.e. EMN) are claiming to commence production of HPMSM in the next 3-4 years, although such projections are tentative and subject to changes in funding and raw material supply.

4.4. Cobalt Sulphate

Li-ion batteries are the primary end-use for cobalt and accounted for 57% of cobalt demand in 2018. As EV uptake continues to increase, the contribution of batteries to the metal's demand is also anticipated to rise.

Although there is a trend to move towards thrifting of cobalt, particularly in NCM CAM, the outlook for the next 10 years indicates that no complete substitute for the element will be found and its rising demand will likely materialise. These concerns surrounding cobalt generally stem from its high cost, long-term supply availability and the underlying human rights problems that have been seen during its production (especially in the DRC).

However, as mentioned above, cobalt remains a critical element for commercial battery chemistries and its consumption in both oxide and sulphate form should continue in the short-medium term future⁴.

4.4.1 Battery grade specifications

Cobalt oxide and sulphate are extensively demanded in the manufacture of Li-ion battery cathodes. NCM CAM relies on cobalt sulphate, therefore only sulphates are the focus of this review. Chemical specifications for battery grade cobalt sulphate heptahydrate ($\text{CoSO}_4 \cdot 7\text{H}_2\text{O}$) are shown in Figure 14. The product is typically sold as a crystalline powder in propylene bags, palletised to approximately 1 tonne.

Figure 14 Sample cobalt sulphate battery grade specifications¹⁸

Product : Cobalt Sulfate – Standard Grade

CAS No : 10026-24-1

Appearance

Pink – Red odorless crystalline powder

Chemical Analysis

	Spec	Typical
Cobalt, as Co, wt %	>21.00	21.06
Manganese, as MN, ppm	<3	0.5
Iron, as Fe, ppm	<3	2.0
Nickel, as Ni, ppm	<80	22.0
Copper, as CU, ppm	<3	ND
Sodium, as Na, ppm	<10	1.5
Calcium, as Ca, ppm	<10	1.0
Magnesium, as Mg, ppm	<10	0.5
Zinc, as Zn, ppm	<3	2.0
Lead, as Pb, ppm	<3	0.3
Chromium, as Cr, ppm	<3	0.5
Cadmium, as Cd, ppm	<3	0.1
Aluminium, as Al, ppm	<3	0.5
Silicon, as Si, ppm	<10	1.0
Potassium, as K, ppm	<3	0.5
Chloride, as CL, ppm	<10	<10
Insoluble, ppm	<50	5.0

4.4.2 Process method

Over 13 different feedstocks can be used to produce cobalt intermediates, the most common being copper (55%) and nickel (35%) concentrates. The supply of cobalt is therefore largely dependent on the demand for such base metals. A variety of processing options exist for cobalt sulphate, as illustrated by Outotec's flowsheet in Figure 15. First Cobalt's concept flowsheet for cobalt sulphate production is also shown in Figure 16.

Figure 15 Simplified flowsheet for cobalt sulphate production¹⁹

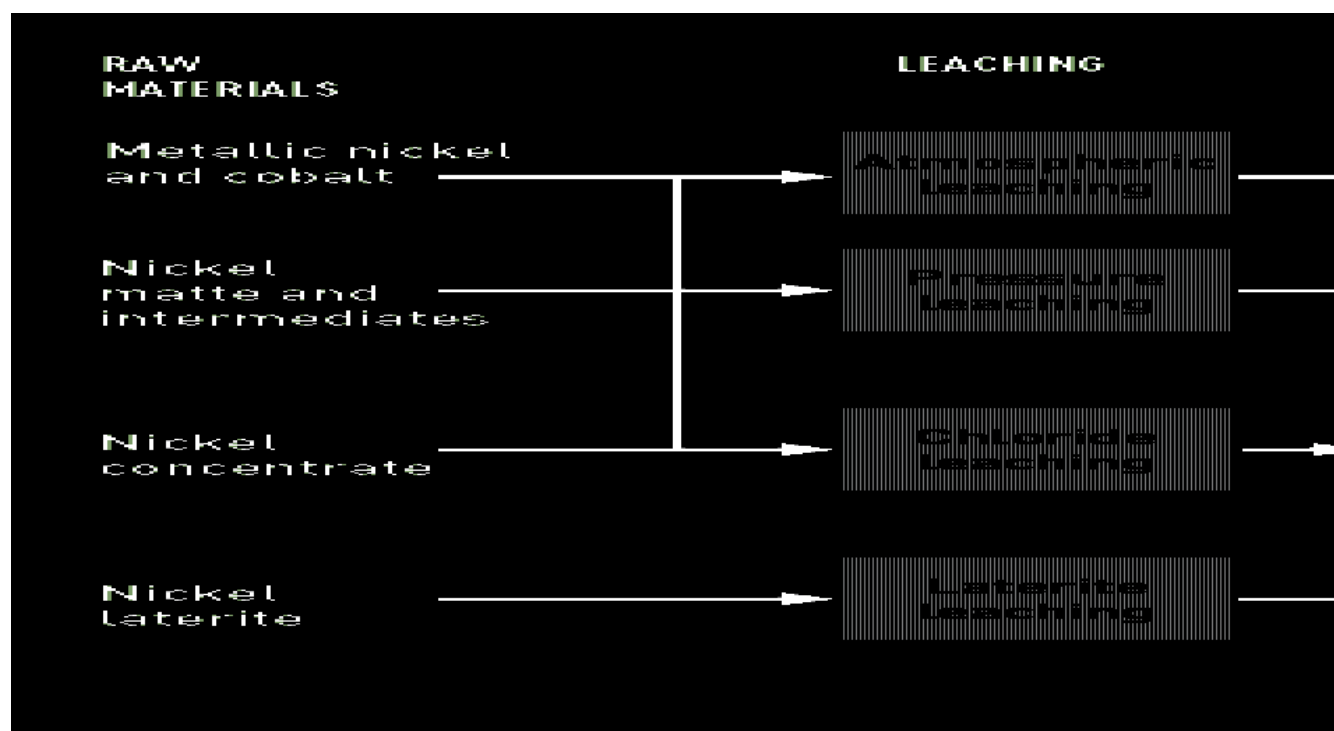
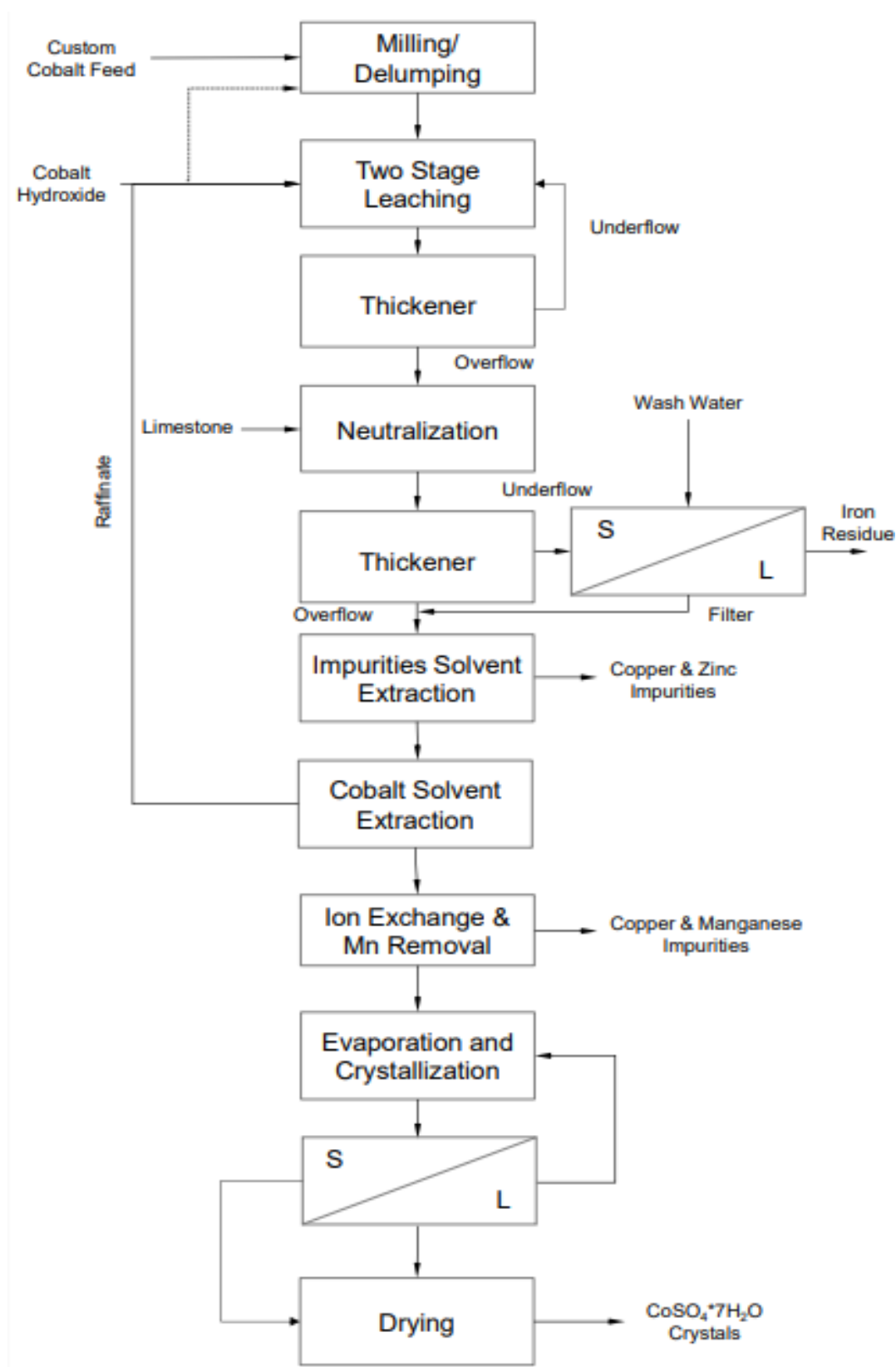


Figure 16 First Cobalt's conceptual flowsheet for cobalt sulphate²⁰



4.4.3 Supply overview

The total global cobalt supply is between ~124–140 kt. This is split between:

- Mined cobalt of 115–125 kt
- A recycling contribution of 12–15 kt.

Generally, it is estimated that 63% of the cobalt consumed in Li-ion batteries is in oxide form, with the remaining 37% in sulphate form⁴. As ~70% of cobalt is mined in the Democratic Republic of Congo (DRC) the distribution of cobalt suppliers globally is relatively poor and it resembles a monopolistic structure. Outside of the DRC, the other major players are Russia, Cuba and Australia who combine for just 13% of global supply²¹.

4.4.4 Industry players and refining capacity

The top three players account for ~40% of global mined cobalt²¹:

- Glencore (22%)
- Gecamines (9%)
- China Molybdenum (7%).

The largest producers of refined cobalt are listed in Table 4. Note that the figures displayed are relevant to 2018 and only select companies produce cobalt sulphate. Of those listed (apart from Chinese producers), key suppliers of cobalt sulphate are Glencore, Umicore, Freeport Cobalt and Sumitomo.

Table 4 Largest producers of refined cobalt, 2013–2017 (tonnes)²²

Company	Country	2014	2015	2016	2017	2018
Ambatovy	Madagascar	2,915	3,464	3,273	3,053	2,852
CTT	Morocco	1,391	1,722	1,568	1,428	1,619
Gecamines	DRC	500	400	400	400	400
Glencore	DRC	2,8	2,9	0	0	0
	Norway	3,6	3,1	3,5	3,5	4,2
	Australia	2,9	3,3	3,2	3	3,2
NPMC	Canada	3,21	3,733	3,693	3,601	3,234
Freeport Cobalt	Finland	11,452	8,582	11,187	12,221	12,874
Sumitomo	Japan	3,654	4,259	4,305	4,159	3,669
Umicore	Belgium	5,85	6,306	6,329	6,987	6,36
Vale	Canada	2,051	1,858	1,851	2,906	2,918
Chambishi	Zambia	4,317	2,997	4,725	2,52	1,613
Norilsk	Russia	2,302	2,04	3,092	2,077	1,8
†	China	39,292	48,719	45,046	69,6	78,36
Others		5,52	4,733	1,72	1,485	1,245
TOTAL		91,754	98,113	93,889	116,937	124,344

† Company-specific production is not readily available

As shown in Table 4, the refining is also monopolistic with at least 60% of global refining capacity concentrated in China. Refining capacity for cobalt sulphate is projected to increase with several new projects underway, including:

- Terrafame's battery chemicals plant in Sotkimo, Finland, under construction and projected to produce 7.4 kt/a cobalt sulphate²³.
- First Cobalt's refinery in Ontario, Canada, is also said to be recommissioned in mid-2020 at a production rate of 2 kt/a cobalt sulphate.
- BHP Nickel West aiming to include cobalt sulphate as a product as part of the stage two expansion of the nickel sulphate plant at the Kwinana refinery.



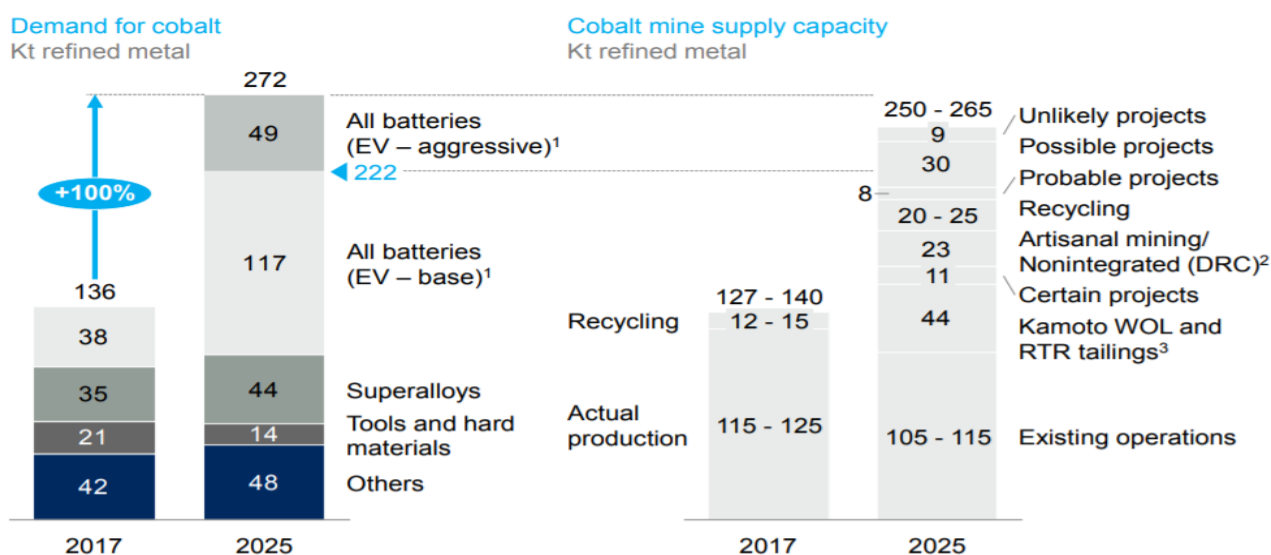
4.4.5 Market outlook

Quantitative metrics predicted by market outlooks can vary widely depending on the assumptions used by each organisation developing the reports. This study identified Roskill and McKinsey as two reliable providers of such information.

According to McKinsey, industry could add between 110-120 kt by 2025 and bring the total potential supply to 225-235 kt. An additional 20-25 kt of recycled cobalt supply would bring the total supply to 245-255 kt by 2025. Supply from copper projects expected to increase to 75% while nickel projects will represent ~20% of supply. A summary of McKinsey's forecasts is shown by Figure 17 below.

Figure 17 McKinsey cobalt market outlook, 2017-2025²¹

Cobalt supply and demand 2017 vs. 2025



1 Battery includes automotive (HEV, PHEV, BEV), trucks and buses (light, medium, heavy), 2- and 3-wheelers, machinery (forklifts and others), grid storage, consumer electronics

2 Includes nonintegrated capacity which is reliant on purchased ore and/or preconcentrate from smaller and/or artisanal operations. This capacity is not tracked on a mine-by-mine basis, but tracked on a processing plant level, assumed to be fed by mines not tracked individually in the other buckets

3 Large increase explained largely by ramp-up of whole ore leach operations by Glencore, already commissioned, and tailings recovery (RTR project) by ERG, expected to be commissioned by the end of 2018. Together accounting for ~41 kt

SOURCE: McKinsey

Roskill's equivalent forecasts are shown in Figures 18 and 19 below, and although they appear to be slightly conservative compared to McKinsey's, they highlight a similar upward trend for the cobalt market. It is interesting to note however that Roskill predicts that the price of cobalt will remain relatively constant until 2028, even though the demand is anticipated to rise.

This is likely due to comparable increases in supply that match the demand and prevent any substantial price increase. Roskill also predicts that cobalt sulphate consumption in Li-ion batteries will increase 4.5-fold from 24 kt Co to 108 kt Co between 2018 and 2028, respectively⁴.

Figure 18 Cobalt supply and demand outlook, 2018-2028⁴

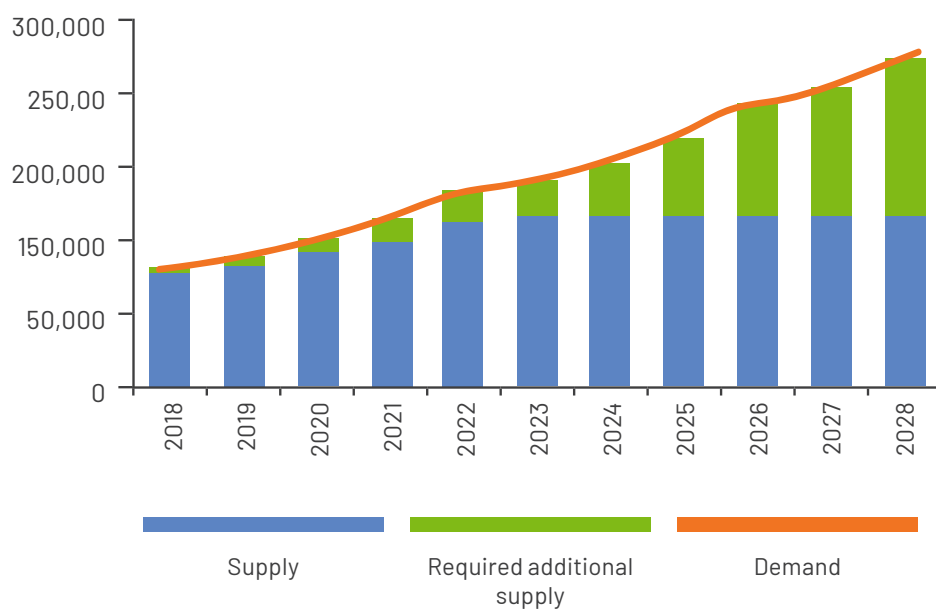
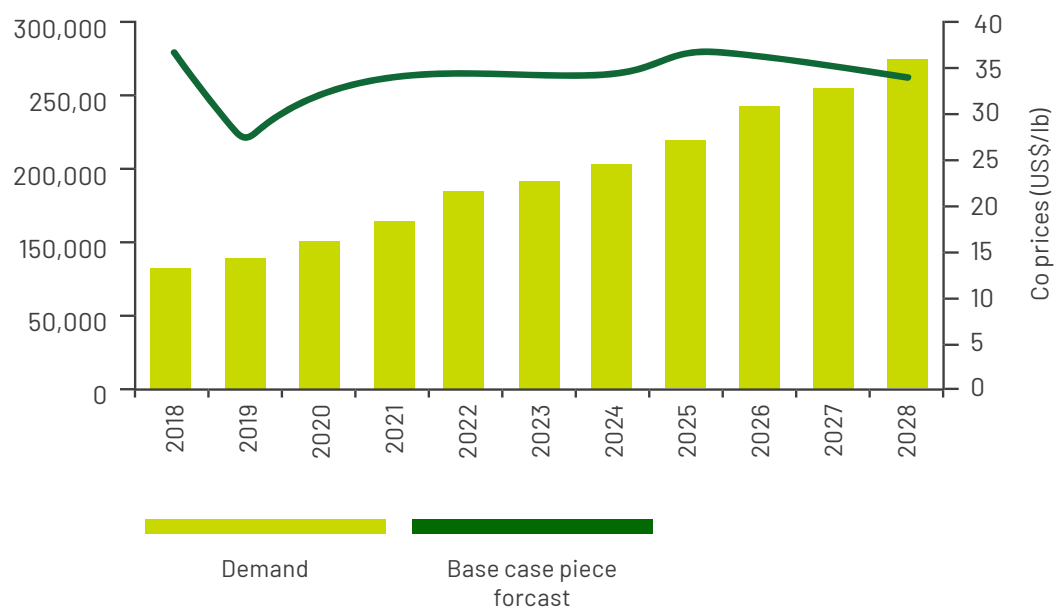


Figure 19 Cobalt demand and price forecast, 2018-2028⁴



4.4.5.1 Developments in DRC

Approximately 40–45 kt of the cobalt mine capacity additions by 2025 are expected from two projects in the DRC.

- 30 kt – Expansion of Glencore’s Kamoto Copper Company (JV between Glencore’s Katanga Mining (75%) and the state miner Gecamines (25%))
- 15 kt – Metalkol Roan Tailings Reclamation (RTR) project from Eurasian Resources Group (ERG).

Through this and other initiatives it is expected that the DRC’s market share will rise to ~75% of cobalt mine supply globally²¹.

4.4.5.2 Developments outside DRC

While DRC is anticipated to retain its market leading position as a cobalt producer, there are significant socio-political risks associated with its availability. As such, for ensuring stable long-term global supply it may become attractive for further developments to be made outside DRC.

However, such developments do not appear to be in abundance. The largest planned expansion outside the DRC is the extension of the Mopani Mine in Zambia, contributing an additional 2.5–3 kt of cobalt annually. No major mine capacity expansion is expected outside of Africa by 2025, although several early stage projects in other regions e.g. Sunrise in Australia may progress further and contribute production capacity²¹.

4.4.5.3 Refining developments outside of China

If midstream producers and OEMs in other regions are to use the increased cobalt mined supply, additional refining investments outside of China will be needed, e.g. for increased recovery cobalt in existing nickel and copper operations. To do this OEMs, smelting and refining companies will need to assess the technical and economic viability of such investments. However, lack of transparency and capacity outside of China may present additional obstacles along the value chain²¹.

4.4.5.4 Future sources of cobalt

As mentioned earlier, the forecasted price of cobalt is anticipated to be relatively constant with new suppliers emerging into the market to take advantage of the rising demand. Roskill predicts that, should prices remain at a significant premium to the US\$13/lb observed from 2012–2016, then it is possible that a greater variety of cobalt suppliers may emerge due to:

- Increase in capacity utilisation and targeting of cobalt rich areas which become more economically viable when cobalt prices rise.

- Increase in artisanal suppliers of cobalt who can only supply during periods of high price.
- Increase in output from tailings dumps such as copper and cobalt rich dumps of which there are many in Central Africa.
- Return of closed nickel operations from increase in nickel prices will result in greater by-production of cobalt.
- Additional feedstock from other nickel producers. e.g. operations in Philippines and New Caledonia which produce nickel materials for nickel pig iron production in China.

4.4.6 Australian supply and availability

There are currently no suppliers of cobalt sulphate in Australia. Note that BHP Nickel West produce a mixed cobalt/nickel sulphide precipitate as a by-product from the Kwinana nickel refinery.

Cobalt Blue is currently in the early stages of commissioning a pilot-scale cobalt sulphate refinery, with an initial production of 200–300 kg expected by Q4 2020. Cobalt Blue has tentatively committed 30 kg of this production to the FBI CRC Pilot Plant Project for initial lab-scale CAM synthesis trials.

4.4.7 International supply and availability

Freeport Cobalt, Glencore, Terrafame and Umicore are potential international suppliers.

4.4.8 Future concerns

4.4.8.1 Ethical Issues at DRC

The supply chain for cobalt is very monopolistic and as such any future concerns about its primary suppliers are worth noting. In this case the DRC has historically not been short of controversy as a cobalt producer.

In the past, there have been many supply disruptions in the DRC and it is currently undertaking substantial changes to its mining law and is in the midst of several major ownership disputes²¹.

When coupled with the notorious child labour and CSR related problems of mines operating in the region, there is growing concern about the ability of DRC to provide a stable supply of cobalt. This problem is further compounded by the fact that the traceability of the material's supply chain has become an important ethical parameter amongst consumers.

A recent example of the repercussions this may bring was the lawsuit filed by human rights firm International Rights Advocates against tech giants Apple, Google, Dell, Microsoft and Tesla. The lawsuit accused the companies of aiding and abetting in the death and serious injury of Congolese children claimed to be working in cobalt mines in these companies' supply chains²⁴.

If the lawsuit, and related societal pressures, actively reduce the battery consumers' appetite for using cobalt sourced from the DRC there is a likelihood that short term prices may rise and demand for non-DRC cobalt to reach temporary highs as cobalt suppliers, while the DRC resolves the issues related to its supply chain.

It should be noted that substantial efforts are currently in progress by the industry to combat these issues.

4.4.8.2 Adoption of low cobalt battery chemistries

Besides the supply chain issues of the DRC, there is also the inherent risk that global shifts in the demand for certain battery chemistries, particularly those with reduced quantities of cobalt (e.g. NCM 811) will prevent demand from rising as significantly as anticipated. This may have negative knock on effects on the price and economic viability of multiple cobalt manufacturers (especially those outside the DRC).

4.4.8.3 Role of China

While China is the major player in the cobalt refining space, it has no significant domestic resources of cobalt and 90% of China's cobalt comes from DRC. Thus, there is an imperative from China to increase its focus on investing in the region. Should China develop their investments in the DRC it is possible that it will have greater ownership and control of the entire cobalt supply chain and thus make it very difficult for any other countries around the world to become a major cobalt player.

4.5 Lithium Hydroxide

Over the next five years lithium hydroxide is expected to surpass lithium carbonate as the primary lithium precursor material in battery cathodes. This transition is largely attributable to the emergence of CAM chemistries with high nickel contents (e.g. NCM 622 and NCM 811), which require the use of lithium hydroxide for effective synthesis at lower temperatures²⁵.

Historically, lithium hydroxide has been mostly sourced from the conversion of lithium carbonate after brine processing, although the capacity for production from mineral concentrates is projected to increase significantly⁴. This study will focus on lithium hydroxide produced by the processing of lithium-based mineral resources (i.e. spodumene, lepidolite, etc.).

4.5.1 Battery grade specifications

The required specifications of precursor $\text{LiOH} \cdot \text{H}_2\text{O}$ for battery applications are shown in Table 5. It is important to control the level of impurity elements such as sodium, iron, and potassium to maintain performance, longevity, and safety of Li-ion batteries⁴. In addition to their chemical composition, the crystals must be fine sized (0.11 mm) with a very low solubility in water and HCl. Higher grade specifications with more stringent limitations on impurities (<0.3 wt%) are also emerging for specialised applications²⁶.

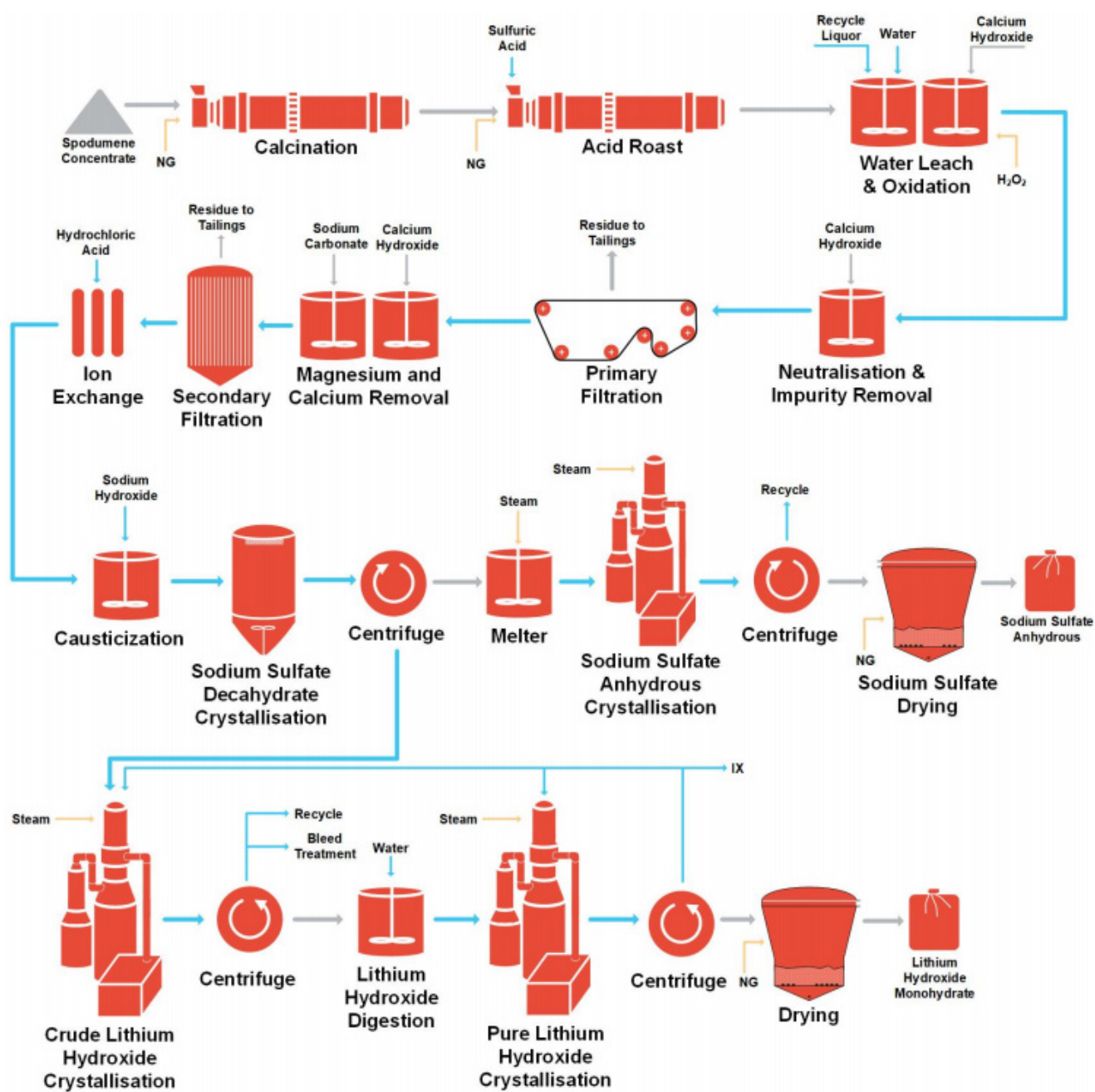
Table 5 Sample lithium hydroxide battery grade specifications²⁷

Item	Standard (wt%) ≤	Typical (wt%)
LiOH	≥56.5	56.5
CO ₂	≤0.500	0.300
Na	≤0.002	0.0015
K	≤0.001	0.0002
Mg	≤0.001	0.0010
Ca	≤0.0015	0.0002
Al	≤0.001	0.0003
Fe	≤0.0005	0.0003
Ni	≤0.001	0.0003
Cu	≤0.0005	0.0002
Pb	≤0.0005	0.0002
SO ₄ ²⁻	≤0.010	0.0020
Cl	≤0.002	0.0012
Mn	≤0.0005	0.0003
Si	≤0.003	0.0015
Zn	≤0.001	0.0002
Insoluble in Acid	≤0.010	0.005

4.5.2 Process method

Lithium hydroxide is produced from spodumene ore by first concentrating the mineral to approximately 5-6%, followed by multi-stage refining to generate the precursor product. Conventional methods are used in the concentrator with comminution, dense-medium separation and flotation employed in the process. The refinery requires a combination of pyrometallurgical and hydrometallurgical unit operations to produce the final product. A simplified flowsheet of the refining of spodumene concentrates is shown in Figure 20. Sodium sulphate is also produced as a by-product of the process, which is often sold separately to cement manufacturers.

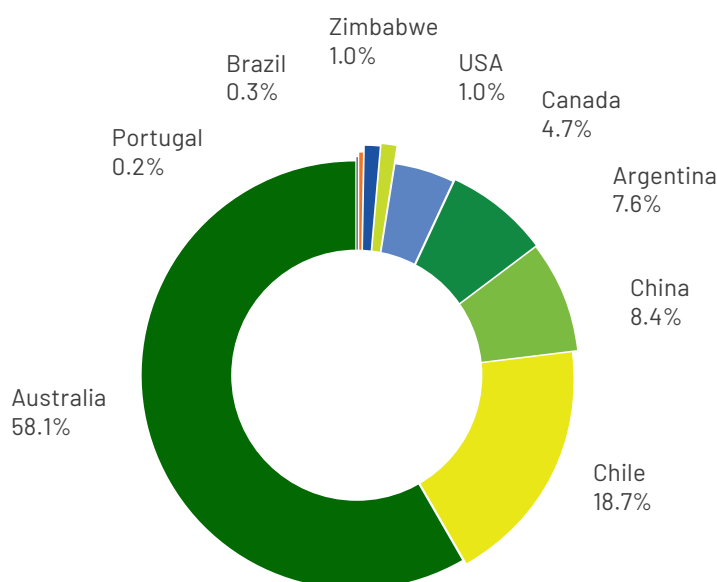
Figure 20 Simplified flowsheet of LiOH production by spodumene processing²⁸



4.5.3 Supply overview

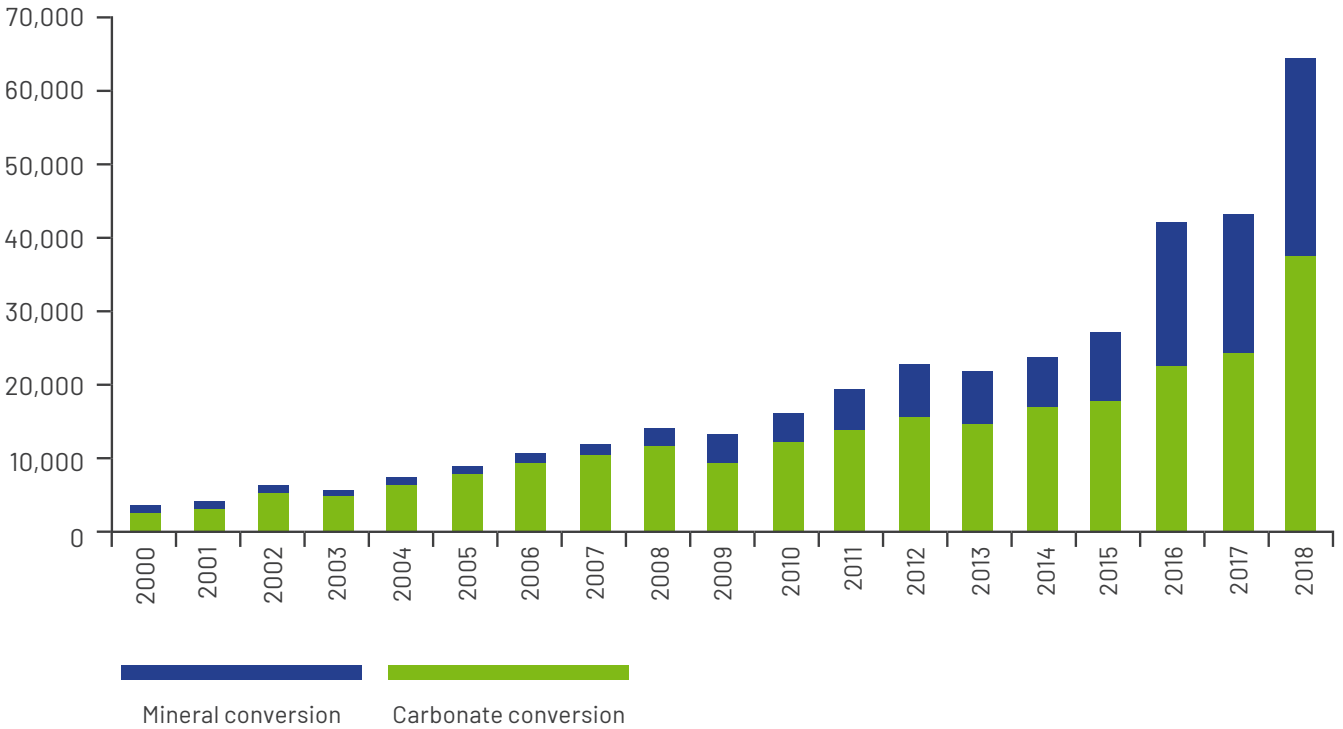
Lithium mineral operations accounted for over 60% of production in 2018 and increased 27.7% from the previous year to an approximate supply of 252 kt LCE. The rapid increase in production was predominately a result of the ramp-up of lithium mineral operations in Western Australia. In the same year, operations were commissioned by Altura Mining, Pilbara Minerals, and Alliance Mineral Assets. Australia is currently the largest producer of mined lithium, accounting for over 58% of worldwide supply⁴. Australia's abundance of lithium-rich orebodies and development of refining operations (i.e. Covalent Lithium) provides a likely prospect for reliable domestic precursor supply. Other key countries in the supply chain include Chile, China, and Argentina, as illustrated in Figure 21.

Figure 21 Production of mined lithium by country⁴



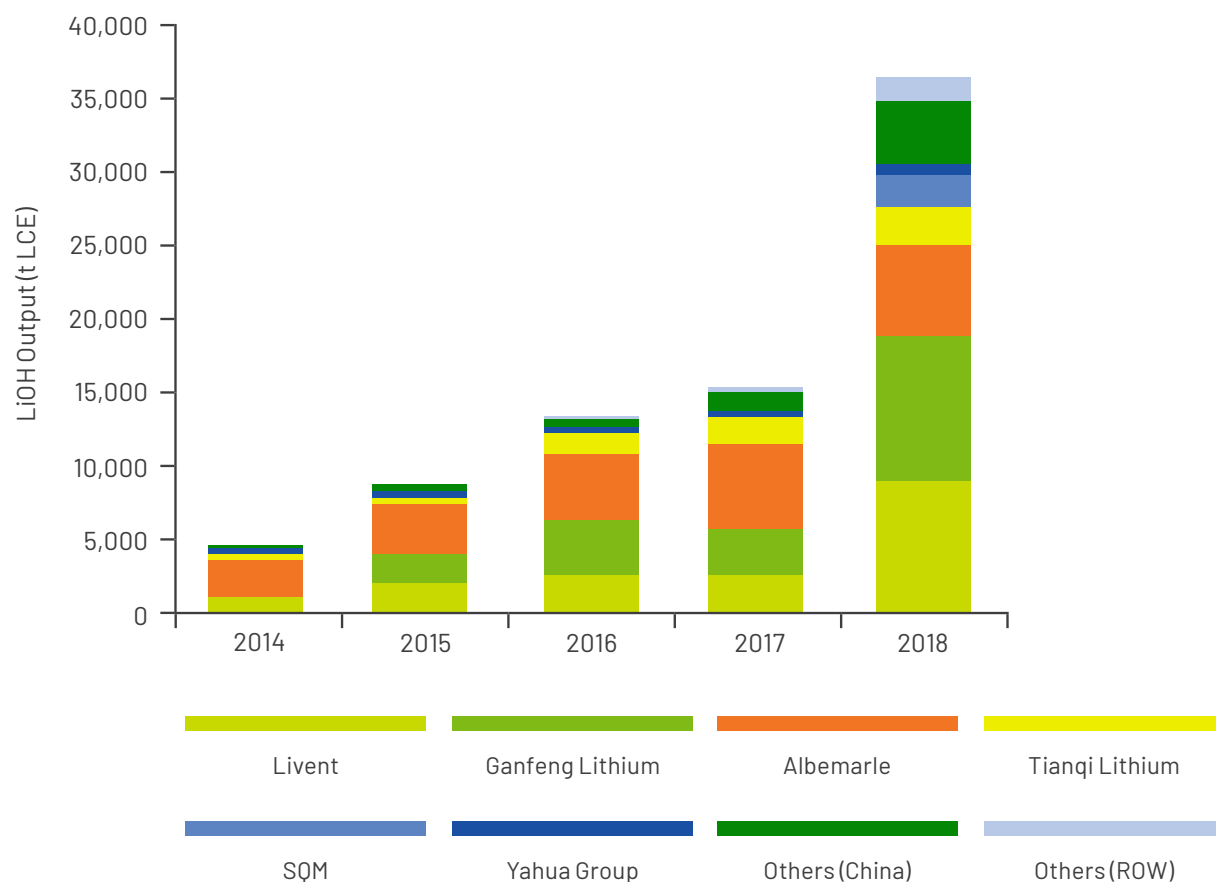
In 2018, total lithium hydroxide supply was approximately 65 kt LCE. As mentioned, the rapid ramp-up of several mineral conversion operations prompted an average annual growth rate of 50% between 2016 and 2018. This dramatic shift is reflected in Figure 22, which also shows the overall combined output from carbonate conversion after brine processing.

Figure 22 Lithium hydroxide production by source, 2000-2018 (t LCE)⁴



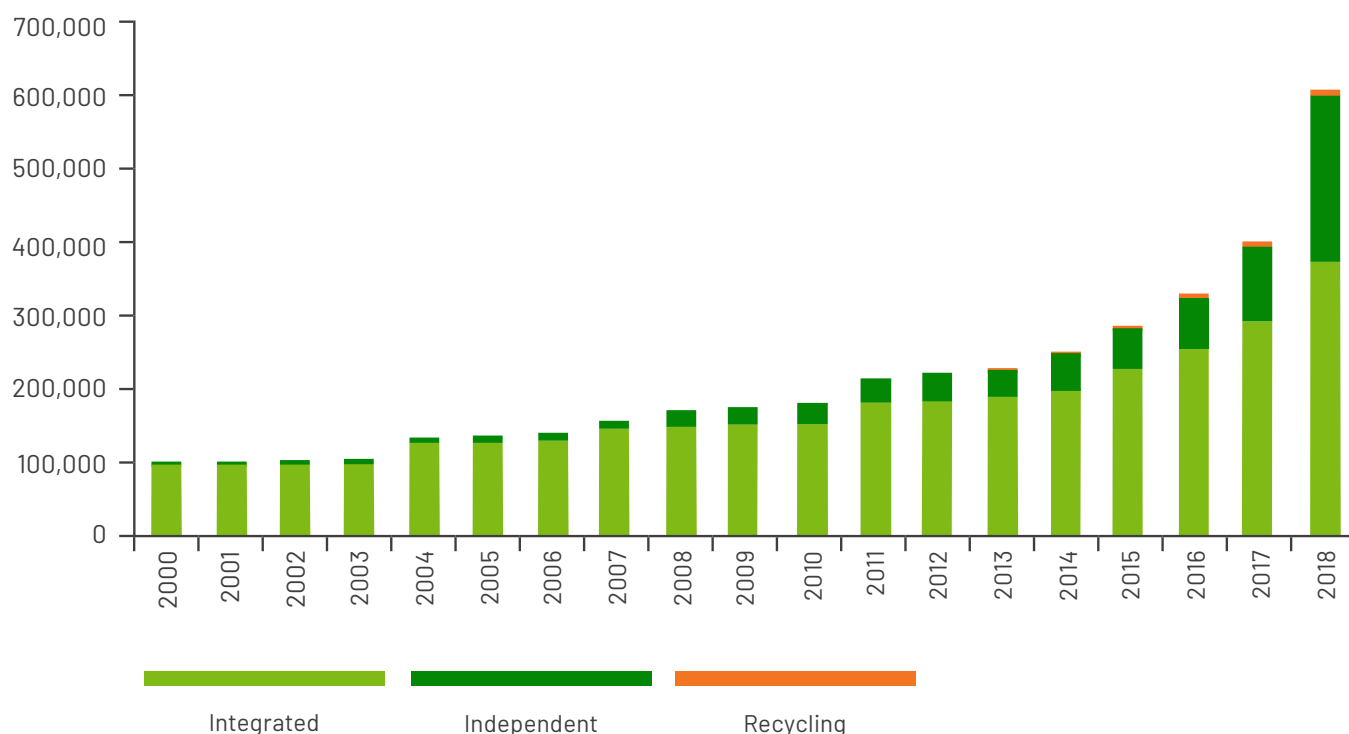
4.5.4 Industry players

There are several key industry players in the lithium hydroxide market, most notably Albemarle, Livent, and Ganfeng Lithium. In 2018, these companies comprised 61.6% of the market share and each produced over 10 kt LCE of battery and technical grade lithium hydroxide⁴. Figure 23 shows the market share of the major producers of lithium hydroxide (battery grade only).

Figure 23 Major producers of battery grade lithium hydroxide, 2014–2018 (adapted from⁴)

4.5.5 Refining capacity

Refined lithium hydroxide capacity has grown exponentially since 2015, a trend that is expected to continue steadily with the introduction of many independent production facilities. In 2018, global refined lithium capacity totalled 540 kt LCE, with 306 kt LCE from mineral conversion operations⁴. While lithium carbonate still constitutes a greater proportion of refining capacity (roughly 70%), the capacity for lithium hydroxide is projected to increase significantly to meet the growing demand of nickel-rich cathode chemistries. Figure 24 shows the refined capacity of total lithium by both integrated and independent processing facilities since 2000. Since 2015, integrated capacities increased by 64.1% while independent operations more than quadrupled in production capability⁴.

Figure 24 Total refined lithium capacity by integration, 2000–2018 (t/a LCE)⁴

Currently, over 80% of Australia's spodumene is exported to Chinese converters, although domestic LiOH refining capacity is expected to increase with greenfield projects such as Covalent Lithium and Tianqi's refineries at Kwinana, as well as Albemarle's facility in Kemerton²¹. Several key factors contributing to the forecasted growth in LiOH refining capacity include:

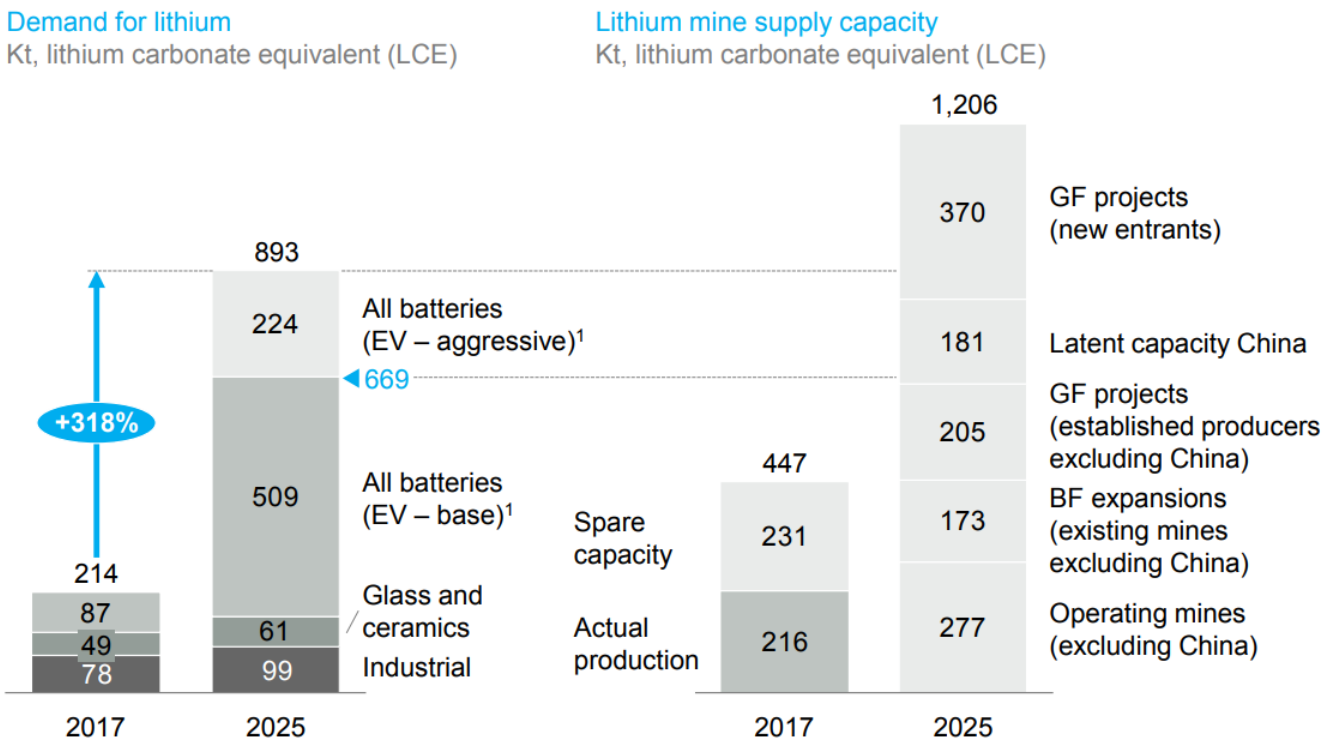
- Albemarle proposal to triple capacity at the Xinyu II plant to 30 ktpa LCE, with the addition of a 20 ktpa LCE lithium hydroxide circuit scheduled to begin operation early 2020.
- Albemarle's agreement with Mineral Resources to construct a further 50 ktpa LCE hydroxide plant by 2028.
- SQM's partnership with Wesfarmers (Covalent Lithium) to construct a hydroxide refinery at Kwinana said to come online by late 2022, projected to output 40 ktpa LCE, with additional land space for 200% capacity expansion.
- General Lithium's apparent 54 ktpa LCE hydroxide plant in the Hubei province, with Stage 1 expected to be online by 2021.



4.5.6 Market outlook

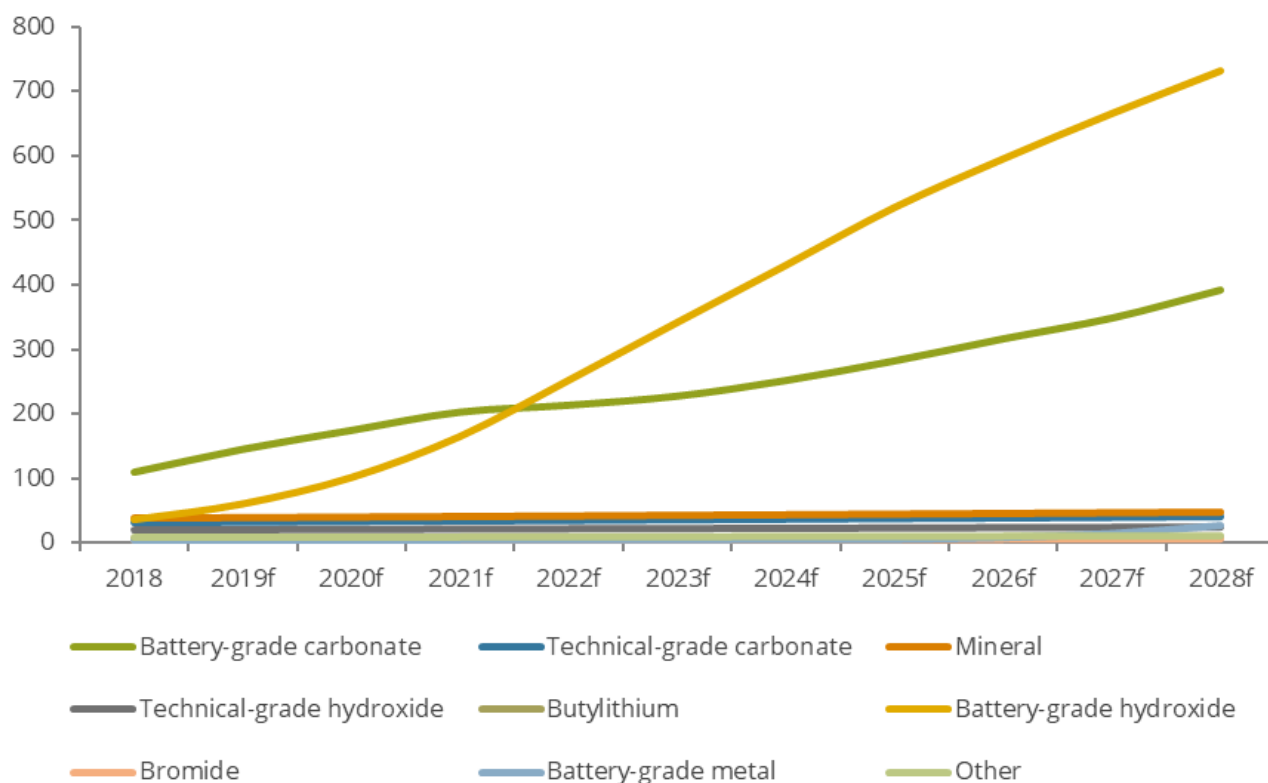
Regulatory trends in vehicle electrification indicate that the lithium hydroxide market will continue to grow significantly. As discussed, the current transition toward nickel-rich cathodes is likely to drive the growing demand for lithium hydroxide as a battery precursor. Roskill (2019) predicts a compound annual growth rate for lithium hydroxide demand in rechargeable batteries of 35.3% from 2018 to 2028 by base-case estimations⁴. Likewise, Mckinsey (2017) has predicted a 318% increase in total lithium demand from 2017 to 2025, with supply expected to reflect a similar upsurge²¹ (see Figure 25). The figure also shows supply and demand projections broken down into end-use categories for current and future operations.

Figure 25 Total lithium supply and demand, 2017 vs 2025²¹



The consumption of battery-grade lithium hydroxide is expected to surpass lithium carbonate precursors by 2022. In addition, Roskill (2019) predicts that it will comprise over 65% of the battery-grade lithium market by 2028⁴ (see Figure 26). The preference for lithium hydroxide as a precursor in new cathode chemistries is expected to bring about a supply deficit, particularly beyond 2025. Although total refined capacity is projected to exceed demand (see Figure 25 above), historical capacity utilisation has rarely surpassed 75%.

Figure 26 Global forecast consumption of lithium by product, 2018-2028 (kt LCE)⁴



4.5.7 Australian supply chain and availability

Tianqi Lithium - plant commissioning is currently in progress with first product expected H2 of 2020. Tianqi has expressed interest in supplying the FBI CRC Pilot Plant Project with battery grade LiOH as it becomes available.

Albermarle - Kemerton refinery projected to come online late 2021 with 60-75 kt/a LiOH.

Covalent Lithium - recently announced a delay in detailed engineering, seeking to re-evaluate project options to meet initial budget projections.

4.5.8 International supply and availability

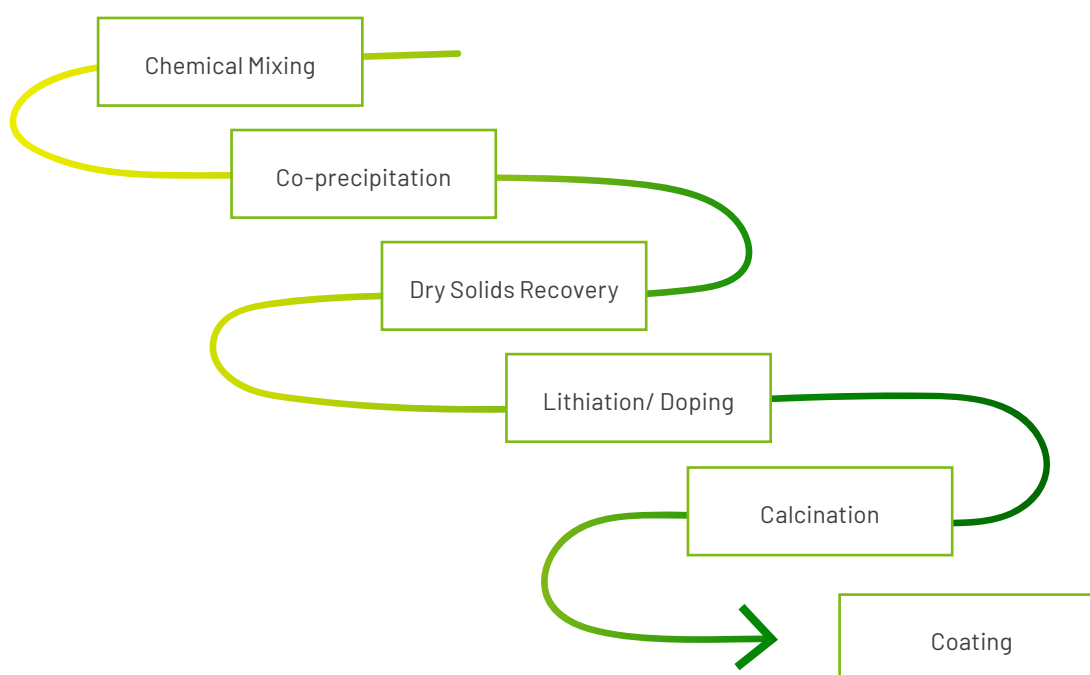
Albermarle and Livent are potential international suppliers of LiOH. Further delays in domestic supply may require prolonged acquisition from international producers.

5. Engineering Development

5.1 Process Block Flow Diagram

A comprehensive literature review was conducted to identify recent synthesis improvements and determine potential best-practice process flow sheets for the preparation of NCM CAM. This was then compared against available information regarding current commercial NCM CAM manufacture. Process recommendations for the FBI CRC Pilot Plant were then generated based on this combined data. NCM 622 is recommended as the starting compound for initial trials as further increase in nickel content drastically complicates processing. NCM 622 will provide an ideal starting point for initial process design and piloting while ensuring equipment specifications are forwards compatible for higher nickel content compounds. The below process recommendations are therefore specific to NCM 622 (65/15/20). 65 mol% nickel NCM 622 is chosen due to its reduced cost from lowered cobalt content. Any reference to NCM 622 henceforth is assumed to be 65/15/20 stoichiometry. Where known, requirements that differ for NCM 811 will be mentioned, especially when they pertain to equipment requirements. The block flow diagram for the process consists of six key process stages. This is illustrated in Figure 27 below.

Figure 27 NCM CAM synthesis process block flow diagram



The overview process description of the process block flow diagram is as follows:

- Chemical Mixing – Full dissolution of precursor materials in an agitated reactor vessel
- Co-Precipitation – Addition of NaOH & NH_4OH to form a $\text{Ni}_x\text{Mn}_y\text{Co}_z(\text{OH})$ precipitate slurry.
- Dry Solids Recovery – Dewatering of the precipitate slurry via filtration followed by washing with deionised water to remove any sulphate impurities from the precipitate and drying. P-CAM product is then obtained.
- Lithiation and Doping – Dry mixing of the P-CAM with LiOH and desired dopants while ensuring particle morphology is maintained.
- Calcination – A 2-stage calcination process in an oxygen rich atmosphere. Mild grinding is then required before coating can take place.
- Coating – Addition of Al_2O_3 powder or other desired coating compounds through gentle dry mixing and calcination which provides adhesion of the coating materials to the surface of the final CAM product.

5.2 Process Flow Diagrams

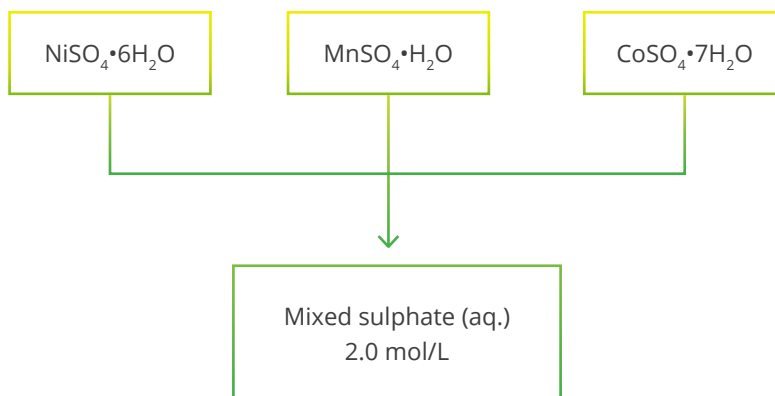
The development of the PFDs has been based on the concept of the use of the BHP Nickel Sulphate pilot plant facility at the CSIRO Waterford facility in Western Australia. The PFDs developed and the overall description of the process is dictated by the existing BHP/CSIRO pilot plant facility and the equipment from this pilot plant. The pilot plant is a small-scale batch operation facility. A number of the key equipment components and process steps for the preparation of NCM CAM material in the BHP/CSIRO pilot plant can be utilised, but this will need to be supplemented by new specific equipment items in order to facilitate the full flowsheet operation.

Using the block flow diagram as a basis, a set of PFDs have been developed, which incorporate the BHP/CSIRO pilot plant equipment and new equipment items as necessary. This will be further described in later sections, see Appendix A. It should be noted that CAM Pilot Plant operation as dictated by the decision to use the existing BHP/CSIRO pilot plant facilities requires a number of manual operations for transfer and movement of solids either as feed or as products from the various stages in the flowsheet. The mass of material processed as part of the batch operation nature of the Pilot Plant and the capacity of the existing reactor vessels is not large. Batch treatment rates of approximately 10kg have been estimated. As such, manual transfer of materials is part of the operation and this will form part of the process description.

5.2.1 Chemical mixing

The block flow diagram for the chemical mixing and dissolution process is generally depicted by the following block flow diagram (Figure 28):

Figure 28 Breakdown of Chemical Mixing

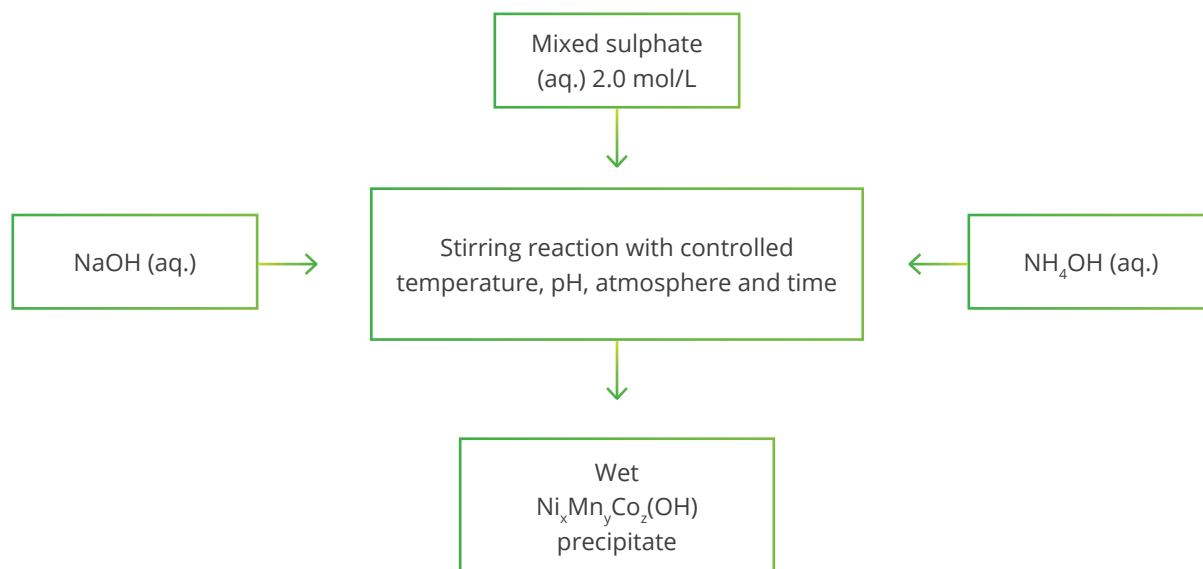


- Nickel, cobalt and manganese sulphate precursor materials are pre-weighed to suit the batch size operation and manually transferred to the reactor vessel.
- The pre-weighed nickel, cobalt and manganese sulphate precursor materials are added to a stirred vessel containing de-ionised water and dissolved. The precursor materials weights added per batch are defined by stoichiometric requirements. Required dopants may be added at this stage, though it may lead to inconsistencies between batches. It is therefore recommended that doping be carried out at the calcination stage.

5.2.2 Co-precipitation

The block flow diagram for the co-precipitation process is generally depicted by the following block flow diagram (Figure 29):

Figure 29 Breakdown of Co-Precipitation

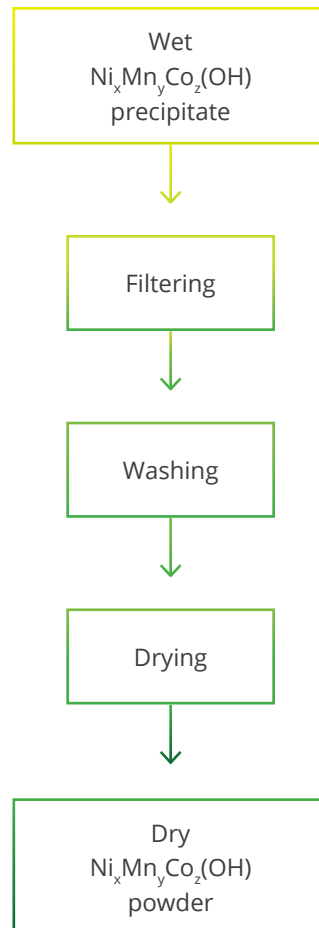


- The mixed metal sulphate solution is added to a reactor vessel with NaOH solution and NH₄OH solution at controlled rates so as to maintain a consistent solution pH. Solution pH control is critical as alkali conditions will cause the formation of impurity phases and loss of spherical particle morphology.
- NaOH is used to assist pH control.
- NH₄OH is used as the complexing/co-precipitating agent. Ammonia concentration has a large effect on particle size and shape and will need to be tailored based on requirement. Increased ammonia concentration facilitates a smaller particle size. If concentration is too high some material may remain in solution leading to incomplete reaction.
- Constant stirring is required. Stirring speed affects particle size and morphology and will need to be tailored based on requirement. Higher mixing speed generates a smaller particle size.

5.2.3 Dry solids recovery

The block flow diagram for the dry solids recovery process is generally depicted by the following block flow diagram (Figure 30):

Figure 30 Breakdown of Dry Solids Recovery

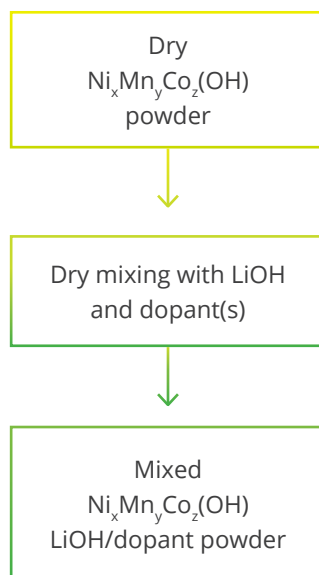


- Wet precipitate slurry is pumped to a dewatering filter. Filtrate from the filter is transferred to an IBC for disposal. Washing of the filter cake is conducted to remove any residual sulphate and is achieved using de-ionised water which is pumped through the filter. At completion the filter is opened and the solids either dropped or manually removed.
- The collected $\text{Ni}_x\text{Mn}_y\text{Co}_z(\text{OH})$ precipitate is then dried via heating and P-CAM is obtained.

5.2.4 Lithiation and doping

The block flow diagram for the lithiation process is generally depicted by the following block flow diagram (Figure 31):

Figure 31 Breakdown of Lithiation and Doping

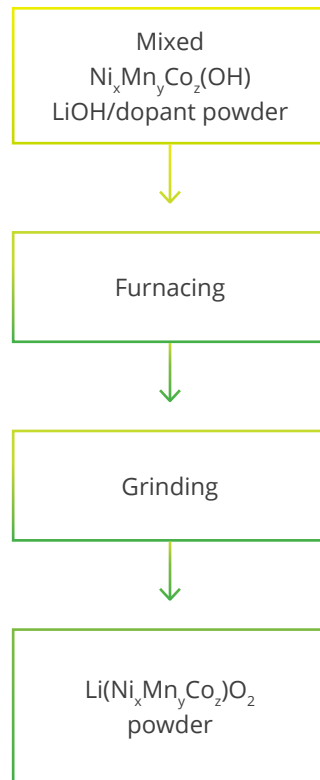


- P-CAM powder is manually transferred to a powder mixer. Lithium Hydroxide powder is manually added to the mixer to achieve a 2% stoichiometric excess of lithium hydroxide. A gentle dry mix is applied preventing any changes to particle morphology.
- LiOH particle size must be fine to facilitate increased dispersion.
- Any required dopants are to be introduced via mixing at this stage also. Common dopants include but are not limited to TiO₂, ZrO₂, Al₂O₃ and MgO. Nano-powders are preferred to enhance dispersion. Specific doping regimes are not divulged by industry, as such trials will have to be performed to ascertain which compounds (if any) are required and in what amounts.

5.2.5 Calcination

The block flow diagram for the calcination process is generally depicted by the following block flow diagram (Figure 32):

Figure 32 Breakdown of Calcination

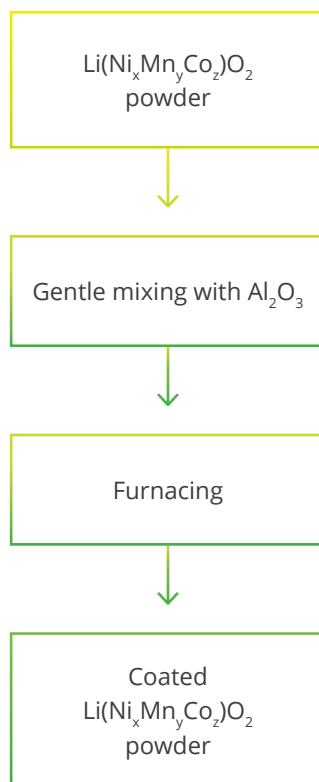


- Mixed powder is calcined by a 2-stage procedure; a low temperature stage followed by a high temperature stage. Temperature profiles are dictated by target stoichiometry, with increasing accuracy in temperature targets required at higher nickel contents.
- Calcining atmosphere is dictated by stoichiometry; <65% nickel requires oxygen enriched air while >65% nickel requires a pure oxygen environment. This in turn dictates the appropriate furnace equipment.

5.2.6 Coating

The block flow diagram for the coating process is generally depicted by the following block flow diagram (Figure 33):

Figure 33 Breakdown of Coating



- CAM is then dry mixed with required coating agents(s) so as to not alter particle morphology.
- Powder mixture is then calcined to facilitate adhesion of the coating material to the CAM surface.

5.3 Process Design Criteria

Utilising the information generated through literature review, process design criteria has been developed. Main control factors include pH levels, reactant concentrations and temperatures along with other criteria which have been used to define the overall flowsheet, process description and the fundamental details of the operation. A Process Design Criteria (PDC) has been developed to define the key operating, mass balance and equipment sizing criteria for the pilot plant. The overall engineering PDC can be found in Appendix B.

As previously mentioned, NCM 622 is the recommended stoichiometry for initial synthesis trials. Increase in nickel content past ~65 mol% stoichiometries begins to become increasingly complicated particularly at the lithiation/calcination/coating stages. As high-Ni CAM compositions are only relatively new they are yet to reach wide implementation in commercial Li-ion cells (though this is currently being rapidly developed). As such, detailed information regarding processing of these materials is well protected and hard to obtain. Where relevant, based on literature review of public domain material, mention of processing variations required for high-Ni content CAM manufacture will be included to ensure equipment recommendations are conducive to synthesis of these materials in future iterations.

5.3.1 Key Process Design Criteria

The key design criteria deemed critical in the production of NCM 622 CAM are noted in the following Table 6.

Table 6 Key process design criteria for NCM 622

Parameter	Stage	Value	Affects	Consequences
Co-precipitation temperature	Co-precipitation	55-60 °C	P-CAM morphology and stoichiometry	Heat is required to ensure dissolution and sufficient activation energy. Failure will lead to inconsistencies in $\text{Ni}_x\text{Mn}_y\text{Co}_z(\text{OH})$ precipitate.
Precipitation pH target	Co-precipitation	12.0	P-CAM morphology; sphericity of secondary particle agglomerates	Excess alkali conditions will lead to a loss in spherical secondary particle morphology and formation of impurity phases.
NH_4OH concentration	Co-precipitation	1-2 mol/L	P-CAM morphology; primary particle and secondary agglomerate size and sphericity.	High concentration can retain metals in solution preventing homogenous co-precipitation

Parameter	Stage	Value	Affects	Consequences
Stirring speed	Co-precipitation	200-1000 rpm	P-CAM morphology; primary and secondary agglomerate particle size	Lack of control in desired morphology
Atmosphere	Co-precipitation	Nitrogen	Purity of P-CAM	Formation of unwanted oxide phases
Drying Temperature	Dry solids recovery	80 °C	Purity of CAM	Low temperatures will leave moisture in particles which is detrimental to calcination.
Mixing intensity	Lithiation	Gentle	P-CAM morphology	If mixing is too vigorous it will damage the particle morphology, damaging CAM properties.
Furnacing Temperature	Calcination	Stage 1 450-480 °C Stage 2 700-750 °C	Purity of CAM, CAM morphology	Low temperatures will cause particles to lack calcination; high temperatures will result in the formation of impurity phases and grain growth.
Grinding	Calcination	TBD	CAM morphology, effectiveness of coating	Damage to CAM morphology
Coating Temperature	Coating	700 °C	CAM electrochemical performance, CAM morphology	Coating does not adhere to CAM surface

A description of the process design criteria for each of the main process steps as defined by the overall block flow diagrams is provided for NCM 622 in the following sections. Mention of process design criteria for NCM 811 is also included where known.

5.3.2 Chemical mixing

- Ratio of $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$, $\text{MnSO}_4 \cdot \text{H}_2\text{O}$ and $\text{CoSO}_4 \cdot 7\text{H}_2\text{O}$ determined by desired stoichiometry. For NCM 622 require a Ni:Co:Mn ratio of 65:15:20 mol%.
- Target mixed sulphate (aq) solution concentration of 2.0 mol/L.
- Stirring required, speed can be variable, only condition is full dissolution of precursor chemicals.
- Ambient temperature and pressure satisfactory, heating may be used to speed up dissolution provided solution concentration is maintained.
- Battery grade precursor chemicals required.
- Deionised water required as solvent.

5.3.3 Co-precipitation

- pH of reaction vessel must be precisely controlled. Excessively alkali conditions will cause the formation of impurity phases and loss of spherical secondary particle morphology. pH 12.0 required for NCM 622. *NCM 811: pH 11.5–11.6.*
- Constant temperature of reaction vessel required (55–60 °C).
- Constant stirring required; increased speed facilitates narrow secondary particle size distribution with increased density. 200–1000 rpm recommended. Stainless steel propeller blades. The propeller amount (per vessel), size and shape can vary depending on the vessel size/shape and required material properties. Blade position in the vertical direction can be changed to tailor properties. A multilayer propeller shaft can be used.
- Concentration of NaOH (aq.) may vary between 2.0 and 4.0 mol/L.
- Concentration of NH₄OH (aq.) may vary; increased concentration promotes uniform spherical secondary particles. 1–2 mol/L is required for NCM 622. *NCM 811: As high as 17 mol/L can be used for NCM 811.*
- Residence time to reach completion may vary between 12–32 hours. *NCM 811: residence times are typically much longer, in the order of 10 days.*
- Inert atmosphere required to mitigate formation of impurity oxides. Nitrogen to be used.

5.3.4 Dry solids recovery

- Washing with DI water to remove any residual sulphate impurity. Typically, 3 washes are sufficient.
- Drying to occur at 80–120 °C for 12–24 hrs.

5.3.5 Lithiation and doping

- Thorough mixing of dry P-CAM with LiOH achieved by gentle dry mixing so as to not destroy particle morphology. High intensity milling not an option.
- LiOH must be either purchased in fine particle form or if stock is coarse it must be ground down. Fine particulate LiOH is highly hygroscopic so considerations need to be taken into account for storage and handling. Water content should be minimised to maximise throughput.
- 2% excess LiOH used to mitigate calcination losses.
- Doping agent(s) used needs to be nano powdered to enhance dispersibility.
- Type and amount of doping agent used industrially is considered sensitive intellectual property and is not disclosed and varies between manufactures. Doping is often tailored to end-user's requirements.
- Common dopants include but are not limited to: Al₂O₃, ZrO₂, TiO₂ and MgO.
- Total dopant content is typically less than 1%.
- Dopant type can affect multiple properties but generally:
 - Al, Zr and Mg increase structural stability.
 - Ti and Mg decrease material resistivity.

5.3.6 Calcination

- Calcination is a 2-stage process; 450–480°C for 3–5 hrs under flowing oxygen enriched air followed by 750°C for 12–15 hrs.
NCM 811: temperature will vary for NCM 811, with more precise control required for hitting temperature targets.
- Atmospheric oxygen content of 50–60%.
NCM 811: pure oxygen environment required with strict retention of atmosphere i.e. a well-sealed furnacing cavity.
- Due to the maximum temperature required (750°C), stainless steel furnaces cannot be used, and a refractory ceramic interior is needed. Stainless steel will leach Cr⁺⁶ ions at this temperature which is highly toxic to humans.
- Final CAM powder is susceptible to oxidation and must be stored under inert conditions.
NCM 811: this is especially relevant at higher nickel content.

5.3.7 Coating

- Dry coating implemented for cost reduction. Wet coating creates more homogenous and effective coatings but requires many extra processing steps.
- Thorough mixing of CAM powder with coating material achieved by gentle dry mixing so as to not destroy particle morphology. High intensity milling not an option.
- Al₂O₃ commonly used. ZrO₂ and boron may also be added to assist with structure retention and conductivity respectively.
NCM 811: Al₂O₃ not suitable for NCM 811.
- Coating material must be nano powdered to assist with dispersion.
- Typically less than 1% total coating material used.
- Calcination required to facilitate adhesion of the coating material to the CAM surface.
- Calcination temperature ~700°C.

5.3.8 De-ironing

- Cell manufacturers require very low magnetic content in CAM material. To minimize Fe contamination through the entirety of processing de-ironing should be conducted at every processing stage.
- If the material is in slurry form, permanent magnets can be used along pipes runs.
- If the material is in powdered form, more powerful electromagnets should be used.

5.4 Mechanical Design

The development of the mechanical design of the FBI CRC CAM Pilot Plant is based on Process Flow Diagrams, Process Design Criteria and projected treatment rate expected for the production of the NCM 622 CAM material product.

5.4.1 BHP/CSIRO pilot plant

As noted in section 5.1 the FBI CRC CAM Pilot Plant will be based on the use of the BHP Nickel Sulphate pilot plant equipment located at the CSIRO facilities at Waterford in Western Australia. The BHP/CSIRO pilot plant is a purpose-built facility designed to operate on a batch treatment basis.

The pilot plant is supported by:

- Service requirements such as clean water and compressed air.
- Laboratory analytical facilities for sample analysis, process control and product quality testing.
- Operating personnel provided by CSIRO.
- Workshop facilities for minor maintenance and Pilot Plant modifications.
- A separate area to receive and handle bulk quantities of precursor feed materials, which can also serve as a handling area to pre-weigh and prepare quantities of feed materials for the treatment process.

The facility is enclosed in an air-tight, dust free and clean building with floor clean up facilities.

5.4.2 Compatible equipment

5.4.2.1 Reactor vessel

The CSIRO pilot plant has a number of existing 100 L reactor vessels. These vessels can be potentially used for both the Chemical Mixing and Co-precipitation stages. These vessels are made of glass and are double skinned to allow for either heating or cooling of the reactor vessel contents. Heating is achieved through circulation of heating oil through an electric heating circuit. The reactor vessels are agitated by mechanical agitators with pneumatic drives which provides the ability to vary the speed and energy intensity of the reactor vessel agitation. These reactor vessels are operated as batch unit operations. As such these vessels dictate the quantity of material which can be treated through the pilot plant as defined by the process criteria developed for the complete process.

An important consideration for the use of glass reactor vessels is possible contamination by leaching of silicon from the reactor wall due to the high pH of the stirred solutions. It is not clear if this will be an issue and testing will need to be carried out early to ascertain whether they are appropriate for use. 316 stainless steel is commonly used as the reactor vessel material, and new equipment that matches this specification will need be purchased in the event the existing BHP/CSIRO pilot plant reactor vessels are not appropriate.

5.4.2.2 Pumps

Various chemically resistant pumps of different types are available for use in pumping and dosing of reactant materials and transfer of precipitate slurry in both the Chemical Mixing and Co-precipitation stages.

5.4.2.3 Drying ovens

The BHP/CSIRO pilot plant has 2 large electrically heated drying ovens which can be utilised for drying the filtered co-precipitate. The drying ovens can each accommodate a number of trays of material. Drying temperatures up to 120 °C may be used.

5.4.3 BHP/CSIRO pilot plant capacity analysis

The capacity of the precursor battery cathode preparation plant is dictated by the constraints of the existing pilot plant equipment sizes and capacities. The following capacity analysis is based on the flowsheet and process design criteria for the preparation of NCM 622 CAM powder.

The first stage in the FBI CRC CAM Pilot Plant flowsheet is the Chemical Mixing stage in which the various precursor feed solids are mixed and dissolved to form a mixed sulphate solution concentration of 2.0 mol/L. The second stage in the flowsheet is the Co-precipitation stage where $\text{Ni}_x\text{Mn}_y\text{Co}_z(\text{OH})$ is precipitated through the addition of NaOH solution to a concentration (2 to 4 mol/L) and NH_4OH solution at 1-2 mol/L. A precisely controlled pH of 12.0 must be maintained. The NaOH solution concentration added to the Co-precipitation is assumed to be 50% w/w. The NH_4OH solution concentration added to the Co-precipitation is assumed to be 30% w/w.

For both these stages of the CAM preparation flowsheet it has been assumed that the 100 L capacity, glass, double skinned reactor vessels in the existing pilot can be utilised for this duty. There are 3 x similar reactor vessels which can be used for this batch duty. The 100 L reactor vessels have the following capability:

- Pneumatic drive which will allow for testing of agitation speed and energy input as a test variable.
- Double skin jacket to allow for either heating or cooling of the vessel for temperature control.
- Electric oil heater and circulation system to facilitate the vessel heating.
- Instrumentation nozzles to allow for insertion of pH control instrument.
- Nozzles at the top of the vessel to allow for the manual addition of the precursor.
- Discharge pump capability to pump the reactor contents to the Dry Solids Recovery stage.

An understanding of the batch capacity of the identified 100 L reactor vessels will determine the overall capacity of the pilot plant in processing capability to produce NCM 622 CAM. To determine the potential capacity of a reactor vessel a simple model of the mixing, dissolution and co-precipitation process has been developed. The model is based on ensuring that at the end of the Co-precipitation stage the level in the reactor vessel is constrained at 85% to prevent overfilling of the reactor. The precursor solids, water and reagent solutions are added as defined by the PDC to achieve this volume at the end of the co-precipitation. Details of the model input, assumption and output are contained in Appendix D.

Based on a number of assumptions the estimated mass of $\text{Ni}_x\text{Mn}_y\text{Co}_z(\text{OH})$ precipitated slurry capable of being produced from each batch operation of the 100 L reactor is 8.24 kg (material). The time to complete a batch of $\text{Ni}_x\text{Mn}_y\text{Co}_z(\text{OH})$ precipitated slurry is estimated as follows:

- Fill reactor vessel with quantity of deionised water: 0.25 hours
- Manually load each of the pre-weighed precursor materials: 0.75 hours
- Allow time for complete dissolution: 0.5 hours
- Heat the reactor to 55-60°C: 1 hour
- Add NaOH and NH_4OH solutions at a controlled rate as the mixed solution is added to the reactor vessel while maintain a pH of 12.0: 1.5 hours
- Precipitation reaction time (between 12 and 32 hours): use 24 hours
- Time to discharge and flush vessel to dewatering: 1 hour

From the above analysis the total estimated time for this batch operation is 29 hours. There are 3 (100 L each) reactors available for this operation which can be staggered and operated in parallel. It is assumed that the pilot plant will be operated on a 24 hour per day basis.

On the basis of the above analysis and utilising all 3 reactor vessels the estimated Pilot Plant capacity is of the order of 20.5 dry kg per day. Depending on the final reaction time adopted for the batch operation this capacity could vary from between 34.9 dry kg per day (12 hours precipitation reaction time) to 16 dry kg per day (32 hours precipitation reaction time).

The remaining equipment required for the subsequent stages of the flowsheet have been analysed and assessed to ensure that the above estimated daily production capacities can be maintained.

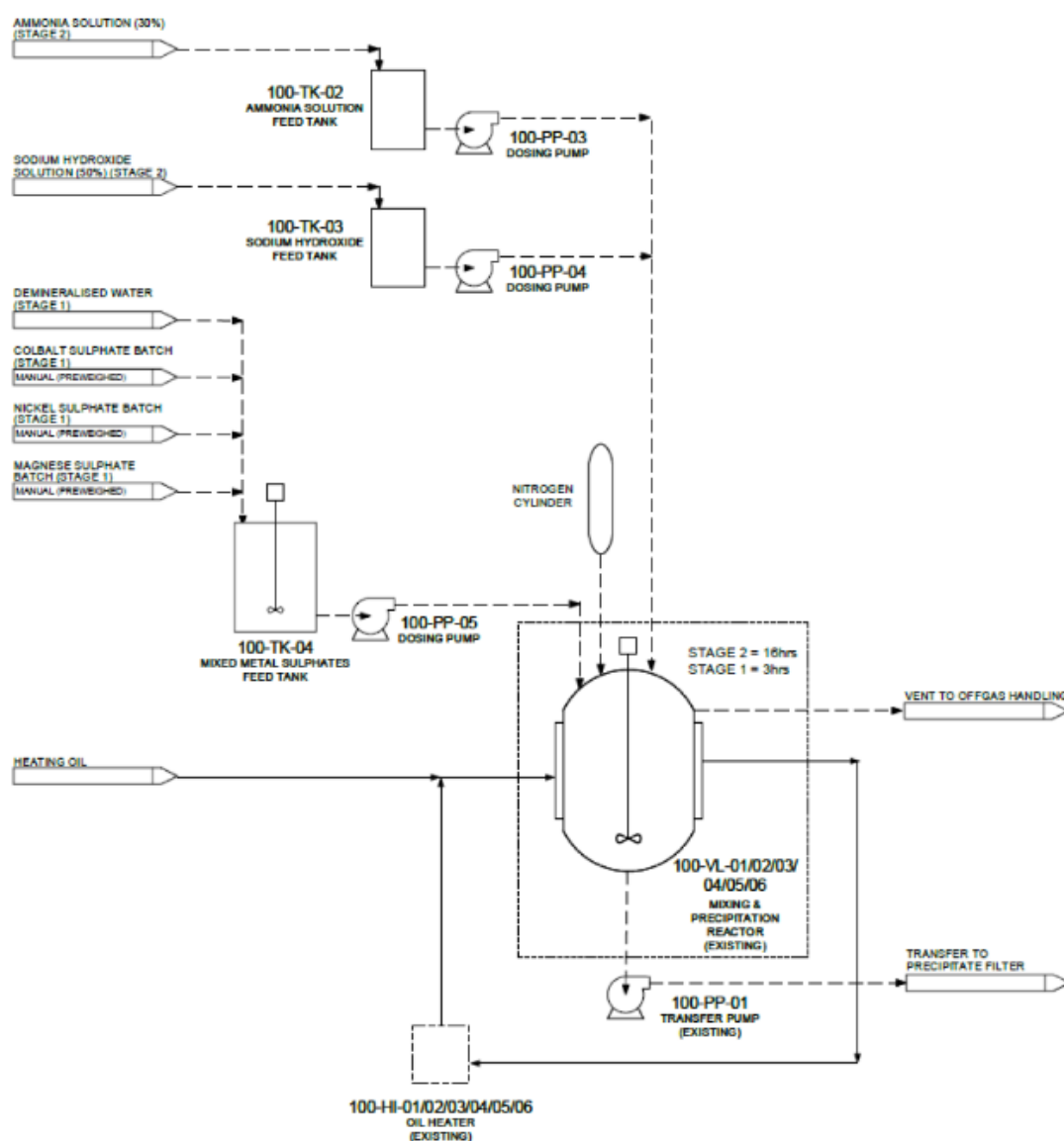
5.4.4 Major equipment descriptions

The major equipment items required for each stage of the process flowsheet are described in the following sections. See Appendix A for the Process Flow Diagram (PFD) sketches prepared which depicts the representation of the Pilot Plant operation and shows the equipment requirements for each step of the process flow sheet. Minor equipment items such as pumps are assumed available from the existing pilot plant installation.

5.4.4.1 Chemical Mixing and Co-Precipitation: reactor vessel

The Chemical Mixing and Co-precipitation stages of the flowsheet operation are assumed to be conducted in the existing pilot plant reactor vessels. The Chemical Mixing and Co-precipitation stages are depicted as follows in Figure 34:

Figure 34 Chemical Mixing and Co-precipitation reactor PFD



There are 6 x similar reactor vessels available. The reactor vessels are fully enclosed double skin glass vessels with elliptical top and bottom and the ability to circulate hot oil in the outer skin for vessel heating purposes. A dedicated electric hot oil heater and oil circulating pump are provided for each reactor vessel. The reactor vessels are equipped with a pneumatically driven agitator. Speed of the agitator and energy input can be varied by varying the air pressure to the pneumatic drive head.

The reactor vessel is equipped with nozzles to allow installation of instrumentation such as temperature and pressure for reactor monitoring purposes. Nozzles are also located at the vessel top are to allow for the introduction of pre-weighed cursor feeds for the mixing stage and sodium hydroxide and ammonium hydroxide solutions for the co-precipitation stage.

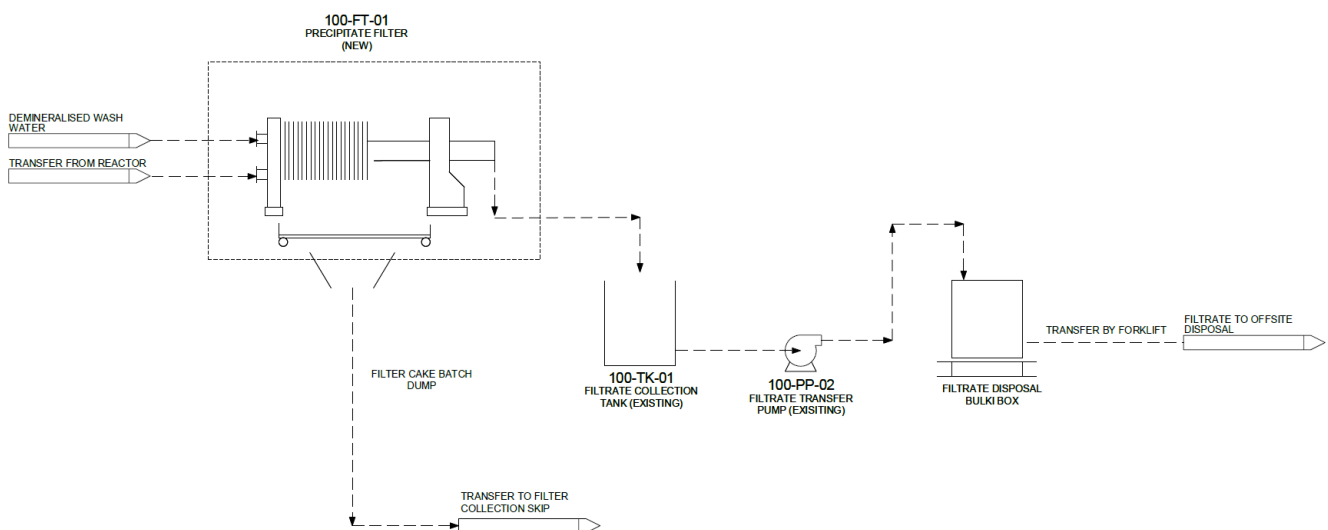
Discharge nozzles at the base of the reactor vessel are connected to peristaltic pumps for the vessel discharge to the co-precipitate dewatering equipment. Operated in batch mode it has been estimated that each batch operation for the Chemical Mixing and Co-precipitation stages will take approximately 29 hours. A total of 3 x reactor vessels is planned to be operated in the Chemical Mixing and Co-precipitation stages of the process to produce the co-precipitated slurry.

As previously mentioned, if Si contamination is found to occur from the tank walls, similar reactors will need to be purchased with 316 stainless steel tank reactors.

5.4.4.2 Dry Solids Recovery: dewatering filter

The dry solids dewatering filter is depicted as follows on the PFD (Figure 35):

Figure 35 Dry Solids Recovery dewatering filter PFD



It is estimated that the solids content of the final co-precipitated slurry will be of the order of 8 to 10% w/w. For an industrial application this is generally low for direct feed to a filtration operation and a dewatering step such as a thickener is used to minimise the filtration dewatering step. In order to simplify the Pilot Plant operation a single step filtration operation can be utilised first (although this may need to be revisited if additional filtrate washing is required). This can be oversized in terms of filtration area required to overcome the excess solution to be treated. The batch operation of the Chemical Mixing and Co-precipitation stages will also allow sufficient time to complete a batch filtration operation.

A suitable filter for this duty does not exist in the BHP/CSIRO pilot plant and a new filter will have to be procured. The potential equipment options available for the filtration duty are described and reviewed below. Due the availability of existing drying ovens at the BHP/CSIRO pilot plant facility, multiple stand-alone filtration systems have been considered for use in combination with these ovens. Other filtration options are also specified here which would do not incorporate the existing drying ovens for the drying step. It is important to note that filtration/drying processes are known to introduce impurities to dried P-CAM powder which then requires de-ironing for removal. This is with the exception of the GL Filtration F450 system which is designed to minimize impurity contamination (de-ironing still recommended). This type of self-contained system may be suitable for the production throughput capacity of the Pilot Plant based on initial estimates. However, these types of systems are not used industrially due to reduced throughput capacity and noncontinuous operation.

Potential equipment options for filtration or filtration/drying at the Dry Solids Recovery stage are as follows:

- Candle type filter.
- Horizontal plate and frame pressure filter.
- Vertical plate and frame pressure filter.
- GL Filtration FD540 system.

Candle type - the candle type filter is generally used for low solids filtration for fine solids separation. This is generally perceived as a solution polishing type of operation.

GBL Process can supply a skid mounted pilot scale test rig of the DrM Fundbac polishing type filters. See Appendix E for details. The test rig has the following general specification:

- Type of filter: Candle pressure filter.
- Number of candle elements = 1.
- Filtration area = 0.3 m².
- Feed pump type = Piston.
- Feed tank = 100 Litres.

The proposed candle filtration test rig is depicted in Figure 36.

Figure 36 DrM FUNDABAC filter with 0.13m² filter area



Horizontal plate and frame pressure filter - plate and frame filters are an industry standard for solid liquid separation dewatering applications. This equipment would be used in combination with existing drying ovens. IFS-Consultants are able to supply a simple skid mounted tailor-made small plate and frame filter which would be suitable. See Appendix E for details. The filter will be manually operated and require no hydraulic drives for opening and closing the filter chambers as generally used in industrial applications. This makes this design simple, robust low cost and easy to install. The filter has the following detailed specifications:

- Filtration plate dimensions 470 mm x 470 mm.
- Maximum number of plates for the filter frame = 10.
- Expected number of plates required for the duty = 3 to 6.
- Filtration area equivalent for nominally 4 plated required = 1.3 m²
- Cake discharge to trays available at the existing pilot plant.

The proposed horizontal plate and frame filter is depicted in Figure 37.

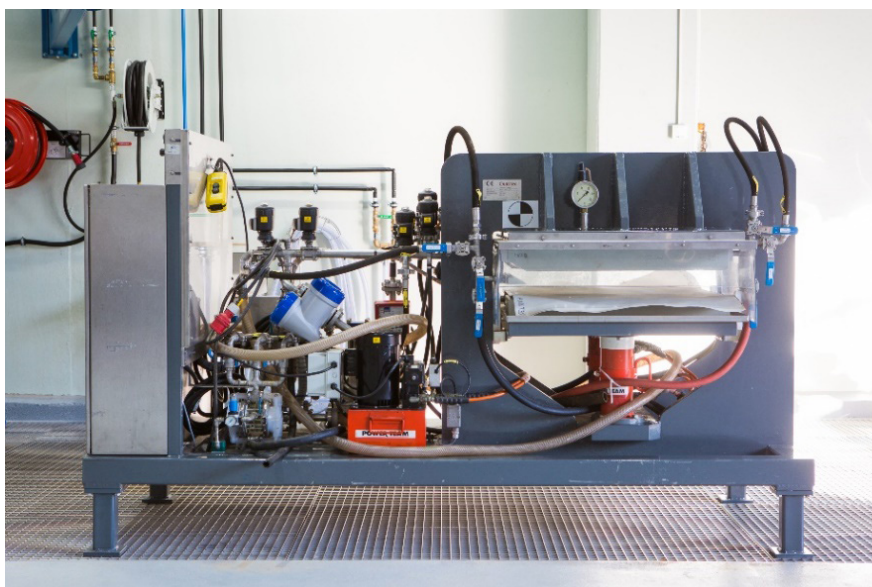
Figure 37 Horizontal plate and frame filter



Vertical plate and frame pressure filter - this filter type is operated on a similar principle as the normal plate and frame filter except that the industrial scale filter utilises a continuous filter cloth and the filter chambers are horizontally arranged such that the filter is oriented vertically. This equipment would be used in combination with existing drying ovens. Outotec supplies this type of filter as their Larox PF range of filters. See Appendix E for details. The smallest standard size filter offered by Outotec is a fully automated unit with a filtration area of 1.6m². This is a complex and expensive machine which requires considerable cost to install. It is not recommended that this option be looked at.

Alternatively, Outotec has a small pilot scale unit with a filtration area of 0.4m². This is skid mounted and relatively easy to install and set up for operation. The filtration area offered in this test unit may be undersized for the expected duty. However, the filtration can be undertaken on a number of batches to treat a full reactor volume with the dewatered filter cake from each batch separately discharged. The proposed vertical plate and frame filter is depicted in Figure 38.

Figure 38 Outotec Larox PF 0.4 filter



Of the options investigated the IFS-Consultants vertical plate pressure filter is recommended for the pilot plant duty. This represents what appears to be a cost effective, simple and robust filter type well suited to the pilot plant operation.

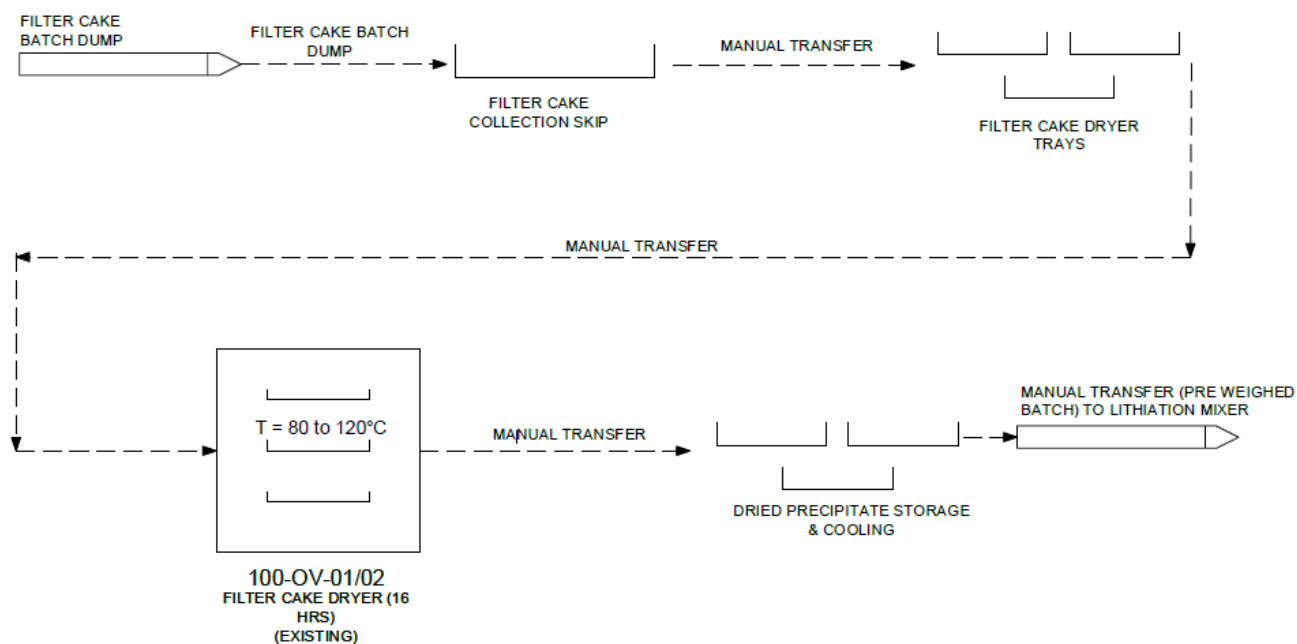
As an alternative Outotec has indicated that it is willing to consider an arrangement whereby the proposed small-scale pilot test filter can be rented or leased rather than purchased. This could be a cost-effective approach and may well suit the proposed campaign nature of operation of the Pilot Plant.

GL Filtration FD450 – this is a combination system for filtering and drying that is designed to minimise impurity contamination and contact with air. This option would not incorporate the existing drying oven for the drying step. Based on preliminary throughput estimates this equipment may be suitable for Pilot Plant use. However, this equipment is not used industrially due to throughput limitations and may not be suitable if production volumes are increased.

5.4.4.3 Dry Solids Recovery: drying oven

The Dry Solids Recovery stage drying oven is depicted as follows on the PFD (Figure 39):

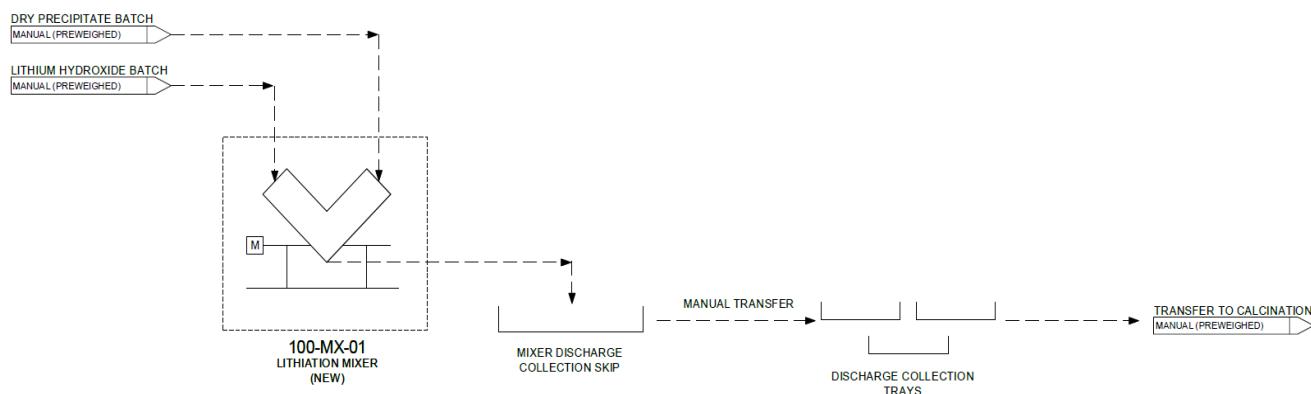
Figure 39 Dry Solids Recovery drying oven PFD



The existing BHP/CSIRO pilot plant has 2 large electrically heated drying ovens which can be utilised for drying the filtered co-precipitate. The drying ovens can each accommodate a number of trays of material. Drying temperatures up to 120 °C may be used.

5.4.4.4 Lithiation: mixer

The lithiation mixer is depicted as follows on the PFD (Figure 40):

Figure 40 Lithiation V-mixer PFD

A suitable mixer for this duty does not exist in the BHP/CSIRO pilot plant. A new mixer will have to be procured. The potential equipment options available for the powder mixing duty is described and reviewed.

There are a number of simple mixing devices for gently mixing dry powder products available on the market. A simple and effective unit is the V-mixer. The proposed V-mixer equipment is depicted in Figure 41.

Figure 41 AWE V-mixer

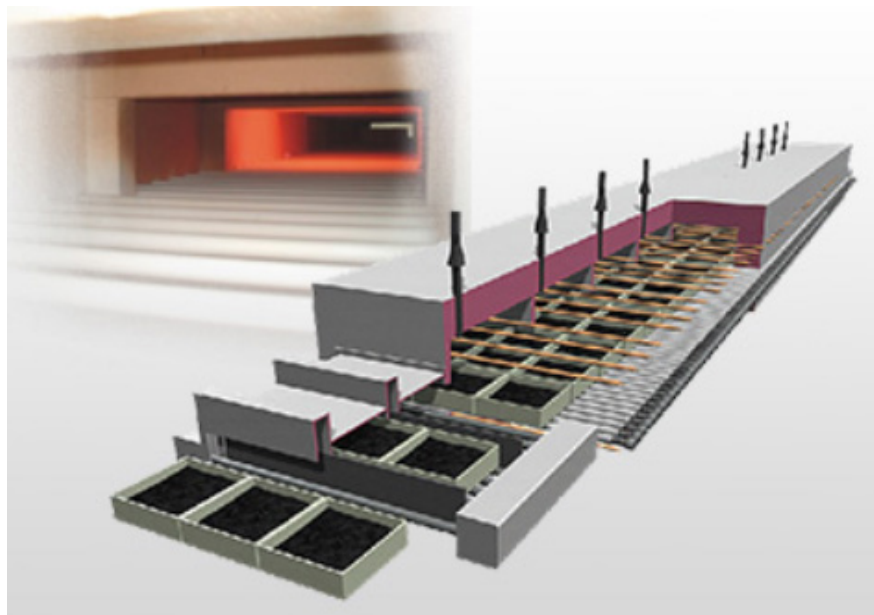
Standard industrial equipment sizes suitable for the Pilot Plant operation are available from a number of suppliers. The AWE Group can supply a V-mixer with the following general specification:

- Electrical rotating skid mounted V-mixer model SVP-0.05.
- Materials of construction – stainless steel.
- Overall/effective volume – 50 L/20 L.
- Electrical power requirements – 0.75 kW.

The V-mixer equipment can be fully skid mounted for ease of installation.

5.4.4.5 Calcination: kiln/furnace

Calcination of CAM material is industrially undertaken by use of roller hearth kilns (RHKs). This is due the highly oxidising conditions present in the furnace hot zone at high temperature (>700 °C) from enriched or complete oxygen atmospheric environments. RHKs are made from refractory ceramic components within the heating zone and are thus able to handle the required conditions without producing contaminants. Trays of mixed P-CAM/ LiOH/dopant are loaded into alumina saggars which are passed through the furnace hot zone on SiC rollers. The internal furnace refractory is made from alumina also. An example of an RHK is pictured below in Figure 42.

Figure 42 Example of an RHK furnace

However, these furnaces are not necessarily the ideal solution as they have low throughput capacity, high energy consumption, have large temperature differentials and uneven heating profiles and poor sealing. These types of furnaces are also expensive and not suited to the throughput volume of material that will be produced by the Pilot Plant. Enquiries to suppliers regarding RHKs at the expected Pilot Plant capacity could not be filled. It is therefore recommended that rotary kiln (RK) furnaces be investigated for the purpose of calcination instead.

Due to forward compatibility with NCM 811 production (requiring a well-sealed, complete oxygen environment) and concerns with stainless at temperature above 700 °C in enriched oxygen environments, stainless steel cannot be used as the RK tube material. The material must either be constructed from or coated internally by a refractory material. Alternatively, the furnace wall material must be made from a steel that does not leach Cr^{+6} and is resistant to scaling and oxidation at temperatures up to at least 1000 °C. Due to the fragility of alumina, RKs are not typically constructed from this material. Some possible RK solutions are mentioned below. However, they are not compatible with NCM 811 production and so are not considered suitable. Further sourcing of the appropriate RK type still needs to be completed. Due to the relatively low estimated throughput volume from the Pilot Plant, a possible solution may be a static furnace with alumina tube/walls, though an RK is preferred.

There were two options available for RKs:

- Batch rotating kin – electrically heated.
- Continuous rotating kiln – electrically heated.

Batch rotating kiln – a batch rotating kiln with a batch charge capacity of between 25 to 50 kg of material would suit the Pilot Plant throughput capacity. The unit is fully skid mounted and includes a tilting mechanism to discharge the calcined material at the end of the operation. A typical skid mounted tilting batch rotating calcining kiln is depicted in Figure 43.

Figure 43 Batch rotating calcine kiln



Cooling of the kiln discharge would be achieved by ambient cooling of the discharged material in the kiln discharge bin/container. This would be achieved over a 12 to 24 hour period with periodic stirring of the material. The kiln exhaust gas would be simply ducted via a hood over the kiln equipment with a fan drawing from the hood via a duct and discharging via a simple duct and exhaust stack outside the pilot plant building. Feed material is charged to the kiln manually. An example of the equipment is contained in Appendix F.

This furnace type is not suitable for future NCM 811 production as a fully sealed, complete oxygen atmosphere is required which cannot be achieved. Precise control of temperature and environment would also be difficult, and the unit would most likely not be suitable for NCM 622 either.

Continuous rotating kiln – a rotary kiln capable of accepting a continuous feed with a nominal capacity of 25 kg of material has been estimated to suit the requirements of the pilot plant. The kiln would be electrically heated and come complete with feed bin, feed screw conveyor, discharge mechanism, product cooling mechanism, product discharge bin and exhaust gas handling system. The intention would be to have the complete facility skid mounted. The feed material would be manually loaded into the kiln feed bin. A control panel, monitoring process control variables, would be provided as part of the package.

5.4.4.6 Coating: mixer

The equipment for the mixing of coating material and CAM would be the same as specified above for the Lithiation stage.

5.4.4.7 Coating: kiln/furnace

The equipment for the coating calcining process would be the same as specified above for the lithiated material calcining kiln. The duties for the lithiated material calcining and coated material calcining can be shared in the same kiln by processing batches of material as required. Ideally a second furnace would be required. Due to the lowered temperature range required for the calcination of coating material, an RK with a stainless steel hot zone may be feasible provided the temperature remained below 700 °C.

5.5 Electrical Power Requirements

The electrical power requirements for the operation of the pilot plant have been built up on the basis of the preliminary equipment list prepared. Power draw for existing pilot plant equipment items have been taken from the equipment list provided by BHP/CSIRO for the existing pilot plant facilities.

Power requirements for new equipment items for the pilot plant have been estimated from equipment vendor catalogues and data sheets or quoted equipment items from the equipment vendors. The electrical power requirements for the operation of the pilot plant based on the proposed flowsheet and equipment specifications is summarised in the following Table 7.

Table 7 Summary of electrical power draw

Item	Description	Installed Power (kW)	Operating Power (kW)
1	Circulating oil heater	7.5	6.0
2	Circulating oil heater pump	0.5	0.35
3	Dewatering feed pump	3.0	2.4
4	Dewatered precipitate drying ovens	5.0	4.0
5	Calcining kiln	40	35

As the pilot plant operation will be on a batch basis the total operating electrical load for the facility will vary depending on which equipment is operating.

The peak load expected would be in the case of 3 x reactor vessels, 2 x drying ovens and the calcining kiln are all operating at the same time. This equates to a total electrical operating load of 60.5 kW.

5.6 Buildings and Support Facilities

5.6.1 General pilot plant building facilities

The pilot plant will be located at CSIRO Waterford in the building currently housing the BHP/CSIRO Nickel Sulphate pilot plant. This building provides all the necessary facilities required for the operation of the Pilot Plant. A number of the existing pilot plant equipment items will need to be modified to accommodate the new Pilot Plant facilities. This is considered a simple activity as the existing pilot plant has been assembled with components which can be modified. New equipment items required for the pilot plant operation will be housed in this facility.

The new equipment items have been selected as skid mounted facilities so that they be placed in the building facilities with minimal engineering and modifications. Services such as compressed air, water and electrical power will be required to be directed to the new equipment items as required.

5.6.2 Support facilities

Associated with the existing pilot plant facilities are the following support facilities which will be vital for the pilot plant operation, monitoring and control:

- CSIRO laboratory support facilities, along with trained staff, which are generally able to cater for a broad range of sample analysis. Laboratory space is available should any specialised analysis equipment be required.
- Amenities for operating personnel such as ablutions, kitchen and crib rooms.
- Communication facilities such as phone and internet.
- Pilot Plant control facilities which can be programmed to suit operation to manage, monitor and control the Pilot Plant via a dedicated control system. The control system can also be used to store historical operating data and records for future reference.
- Workshop facilities to allow for daily maintenance requirement for equipment and minor modifications.
- Storage facilities for the precursor feed materials and process reagent requirements.

6 Value-Add Estimates

6.1 Basis of Estimate

The value-add estimate has focused on identifying the benefits generated through converting the precursor feed materials into NCM 622 CAM. The estimate of the value added in producing the NCM 622 CAM is the difference between the potential market value of the cathode active material minus the cost to produce the CAM.

The cost to produce the CAM is summarised by the following operating cost items:

- The battery grade precursor feed materials (NiSO_4 , CoSO_4 , MnSO_4 and LiOH).
- Reagents (sodium hydroxide and ammonium hydroxide solutions).
- Electrical power and/or fuel.
- Labour.
- Consumables (filter cloths, lubrication, material handling items, packaging, etc.).
- Maintenance of the facility.
- General and Administration.

For Pilot Plant operation the various fixed costs, such as labour and general and administration costs, would be high compared to an industrial scale facility and this would add significantly to the overall treatment cost. Pilot plant facilities as a batch operation are not designed to operate at maximum efficiency and are not designed to take advantage of the economies of scale generally resulting from a full-scale industrial facility design. For these reasons the evaluation of the value added from the normal development of a full operating cost model will not make economic sense.

Evaluation of the potential of the value-add of producing CAM from the precursor feed materials will instead be estimated on the basis of the feed material and reagent consumption costs per batch of prepared material from the Pilot Plant and utility prices for electricity and steam versus the current CAM market price information available in the public domain.

The Pilot Plant works in a batch process as stated in the mechanical design. Two or three batches are assumed to be produced every 24 hours. Material losses from transfers and equipment are negligible. Estimates for reagents and feed precursor materials have been based on literature where possible, and alternatively suppliers taking into account current market conditions. Where possible Australian supply rates were selected, however, in some instances American and Chinese suppliers were selected due to limited distribution of the chemicals. In these cases, an exchange rate of 1AUD = 0.65USD and 1AUD = 4.31CNY were chosen.

The summary of the unit purchase cost for the precursor and reagent requirements for the process operation basis figures are shown below in Table 8.

Table 8 Feed pricing

Feed	Price (\$AUD)	Units
Caustic soda (50%)	757	As 50% solution w/w
Ammonium solution (30%)	678	As 30% solution w/w
Cobalt sulphate	13,000	per tonne
Nickel sulphate	6,500	per tonne
Manganese sulphate	1,525	per tonne
Lithium hydroxide monohydrate	12,700	per tonne

Utility prices shown in Table 9 are based off current government rates in Western Australia.

Table 9 Utility prices

Feed	Price (\$AUD)	Units
Demineralised water	1.43	Per kilolitre
Electricity	0.31	Per kWh
Gas (natural gas) usage	0.12	Per kWh

The current market price (@ 1AUD = 4.31CNY) for P-CAM, CAM and associated raw materials is listed in Table 10.

Table 10 Current market prices for P-CAM, CAM and precursor materials (HCA, 2020)

Material	Price (\$AUD)	Units
NCM 523 CAM	27.26	Per kg
NCM 622 CAM	32.48	Per kg
NCM 811 CAM	43.50	Per kg
NCM 523 P-CAM	17.75	Per kg
NCM 622 P-CAM	19.26	Per kg
NCM 811 P-CAM	19.84	Per kg
Nickel sulphate	5.34	Per kg
Manganese sulphate	1.45	Per kg
Lithium hydroxide	49.30	Per kg

6.2 Estimate Exclusions

In the major utilities covered electricity does not factor into account peak and off-peak rates. Instead opting for an average of the two rates. It was found that for a small pilot plant of this size it would have inconsequential effects.

Delivery costs associated with the supply of reagents and feed are not included as these are dependent on where these are purchased from. Distribution is highly dependent on plant location and these costs can be accounted for when investigations into the purchase costs of reagents and feed precursor materials have been advanced.

Environmental costs relating to the disposal of waste filtrate are handled off site by third parties such as the Water Corporation of WA. The waste is currently sent to be treated offsite and as such is highly dependent on the local geography of the plant.

6.3 Value-Add for NCM 622 CAM

- The current market price for NCM 622 CAM is \$32.48 AUD/kg (@ 1AUD = 4.31CNY)(HCA, 2020).
- The cost of reagents and metal, including lithium, to produce NCM 622 CAM in the Pilot Plant facility is estimated to be approximately \$19.10 AUD/kg.

As the costs of processing for the Pilot Plant are currently unknown, actual margins for value added cannot currently be calculated. However, based on raw materials cost and the current market price, it can be estimated that Pilot Plant processing costs to produce NCM 622 CAM would need to be less than \$13.38 AUD/kg (minus whatever mark-up is present on the current market price, estimated to be very small) in order to have any added value margin.

7 Recommendations Summary

Based on the data collated in this Report, combined with current market trends, this section will detail a recommended roadmap forward for the FBI CRC CAM Pilot Plant, including targeted CAM materials and their required properties. As significant testing is required to verify and optimise the key process parameters (see Table 5-1) it is the recommendation of this Report that a lab-scale phase be implemented before pilot-scale production is commenced. Although parameters are likely to change when moving from lab-scale to pilot-scale, lab-scale testing will provide validation and reasonable starting values to commence pilot-scale operation. As much smaller quantities of material are required at lab-scale, this will greatly reduce operating costs and increase the speed of initial testing while also minimising risk.

7.1 Cathode Active Material Targets

As mentioned previously, due to the complexity in processing of NCM 811, it is recommended that initial trials focus on the synthesis and optimisation of polycrystalline NCM 622 (65/15/20) to first develop process and technical expertise. This material has a high uptake for the EV market in bi-modal form; that is a dual particle size distribution consisting of 4 and 14 μm D50 secondary particle agglomerates. This requires that separate batches of 4 μm and 14 μm P-CAM be generated and taken to final coated CAM stage separately also. They are then combined for final use.

Based on current market trends the below progression for NCM CAM types should be implemented for FBI CRC CAM Pilot Plant production. NCM market demand is changing rapidly, and this progression will need to be revised regularly. It is not recommended that NCA production be pursued until after NCM 811 production has been mastered due to similarities in complexity of processing.

- NCM 622 (65/15/20) polycrystalline bi-modal 4-14 μm
- NCM 811 (80/10/10) polycrystalline bi-modal 4-14 μm
- NCM 622 (65/15/20) single crystalline ~3-4 μm
- NCM 811 (83/12/05) polycrystalline ~12 μm
- NCM 811 (83/12/05) single crystalline ~3-4 μm
- Further increased nickel content materials

7.1.1 NCM 622 performance requirements

Cell manufactures will have a list of CAM performance requirements that must be met before they will procure material. This will vary between cell manufactures, but in general requirements for NCM 622 are:

- Cell testing occur at 45 °C with a 2.5-4.2 V cycle profile in coin half-cells and then single layer pouch cells.
- 160-170 mAh/g capacity.
- 95% capacity retention after first 30 cycles.
- Coulombic efficiency (CE) of 90% initial discharge.
- Press density of 3.2-3.5 g/cm³.

7.2 Lab-Scale

Initial trials at lab scale will require the procurement of a low volume (2-10 L) capacity reaction vessel that can meet the process design criteria specified in sections 5.2.3.1 and 5.2.3.2. This will require stirring and heating control, ability to control dosing rates of mixed sulphate, NaOH and NH_4OH solutions as well as pH monitoring capability and control of reactor atmosphere.

A glass reactor is recommended so that Si leaching can be first tested to ascertain whether the existing BHP/CSIRO reactor vessels are feasible for use. The exact same composition glass as the existing vessels must be used. If Si contamination occurs, a small scale 316 stainless reactor will need to be procured, as well as replacement of the existing pilot-scale glass reactor vessels. Another option is to perform a simpler leach test with more rudimentary equipment first to assess Si contamination prior to purchasing the lab-scale reactor. Recommended lab-scale progression is as follows:

- Determine feasibility of glass reactor vessels. This will require elemental analysis (XRF, EDS, ICP) of rudimentary P-CAM formed in the same material vessel as the existing reactors. Must be conducted at stirring, temperature and pH conditions matching those quoted in sections 5.2.3.1 and 5.2.3.2.
- Optimise P-CAM for ~14 μm secondary agglomerate size (P-CAM14). It is not recommended to aim for exact control of particle size at this stage as parameters are likely to change when transferred to pilot-scale. This will require mainly scanning electron microscope (SEM) and particle sizing analysis.
- Processing of P-CAM14 to final CAM stage with dopant and coating materials aimed at improving structural stability (Al_2O_3). QUT currently has the capability to perform the Lithiation, Calcination and Coating stages at lab-scale, and it is recommended that these trials take place there. To ensure that the Lithiation, Calcination and Coating stages are functioning correctly, 'control' CAM will be generated at QUT from commercially available P-CAM of similar secondary agglomerate size. This CAM material will be electrochemically characterised and serve as the benchmark for lab-scale P-CAM14 generated. As well as electrochemical characterisation, this step will require structural (XRD), elemental (XRF, XRD, ICP) and morphological (SEM, particle sizing) characterisation. QUT has this capability and it is recommended that this analysis occur there to minimise downtime and complications from shipping of material. This process is intensive and would require significant allocation of QUT staff and resources.
- Once the ~14 μm P-CAM performance has been verified it may move to pilot-scale trials and production.
- Optimise P-CAM for ~4 μm secondary agglomerate size (P-CAM4). It is not recommended to aim for exact control of particle size at this stage as parameters are likely to change when transferred to pilot-scale.
- Processing of P-CAM4 to final CAM stage with dopant and coating materials aimed at improving structural stability (Al_2O_3). This material can then be mixed with CAM generated from P-CAM14 to form bi-modal 4-14 μm NCM 622 CAM.

- Perform electrochemical testing of this material against commercial bi-modal NCM 622 CAM as a benchmark.
- Once performance has been verified production of NCM 622 can switch entirely to pilot-scale, and lab-scale testing of NCM 811 P-CAM production can commence.

7.3 Pilot-Scale

Once P-CAM14 has been verified electrochemically against benchmarks, pilot-scale production should begin. This will require some tweaking of processing conditions for P-CAM formation due to the increase in scale. Similar to lab-scale trials, the P-CAM, once formed in pilot-scale quantity, should then be processed in lab-scale quantity to final doped and coated CAM material for electrochemical benchmarking and characterisation at QUT.

By this point, arrangements for procurement and installation of the appropriate pilot-scale equipment required for the Lithiation, Calcination and Coating stages should be complete. Once this infrastructure is in place pilot-scale production of NCM6 22 CAM can commence, with accompanying electrochemical benchmarking and characterisation in a similar progression as described for lab-scale testing. NCM development should then proceed as per section 7.1, or as per current market demand dictates.

8 Acknowledgments

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About the Research Team



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PRICIPAL INVESTIGATOR AND PROJECT LEAD

Peter is a Professorial Fellow at QUT and an AQ Founders Fellow. He established QUT's Advanced Battery Facility (ABF), the first lithium ion battery pilot manufacturing facility in Australia. His expertise includes the management of skilled scientific and engineering teams in collaboration with both local and international R&D organisations to solve industrially relevant problems. He has a multi-disciplinary academic background and is regarded as a materials science expert and leader in the fields of lithium-ion batteries, nano-materials manufacture and high temperature superconductors. Professor Talbot holds numerous patents in the fields of solid-state chemistry, automated chemical manufacture and nano-materials.



Dr Joshua Watts

CHIEF INVESTIGATOR

Joshua is a Research Fellow at QUT with a PhD in energy and process engineering specialising in nano-materials synthesis, optimisation and characterisation. He has over 10 years of research and development experience in both industry and academia, primarily focused in the fields of renewable energy generation and energy storage. Joshua's experience includes the synthesis and optimisation of Li-ion battery cathode materials at pilot plant scale as well as the synthesis of non-oxide ceramic materials for high performance applications. Joshua has several peer reviewed publications in the fields of materials synthesis and advanced characterisation.

9 About the Future Battery Industries Cooperative Research Centre

FBICRC is an independent centre where industry, government and researchers can come together to create the tools, technologies and skills to grow the role of battery storage in Australia's electricity grids, and make Australia a larger player in global battery value chains. Australian minerals are critical to the global battery technology revolution. Our resources, thinking, innovation and expertise will enable us to seize this once in a generation, national opportunity to develop the next wave of battery industries.

10 About the Queensland University of Technology Institute for Future Environments

The IFE is a transdisciplinary research and innovation institute that addresses social and economic challenges in a range of key sectors, including primary industries, the environment, resources, manufacturing, services and information communication and technology. The IFE's mission is to generate knowledge, technology and practices that make our world more sustainable, secure and resilient by addressing global challenges in a resource-constrained world.

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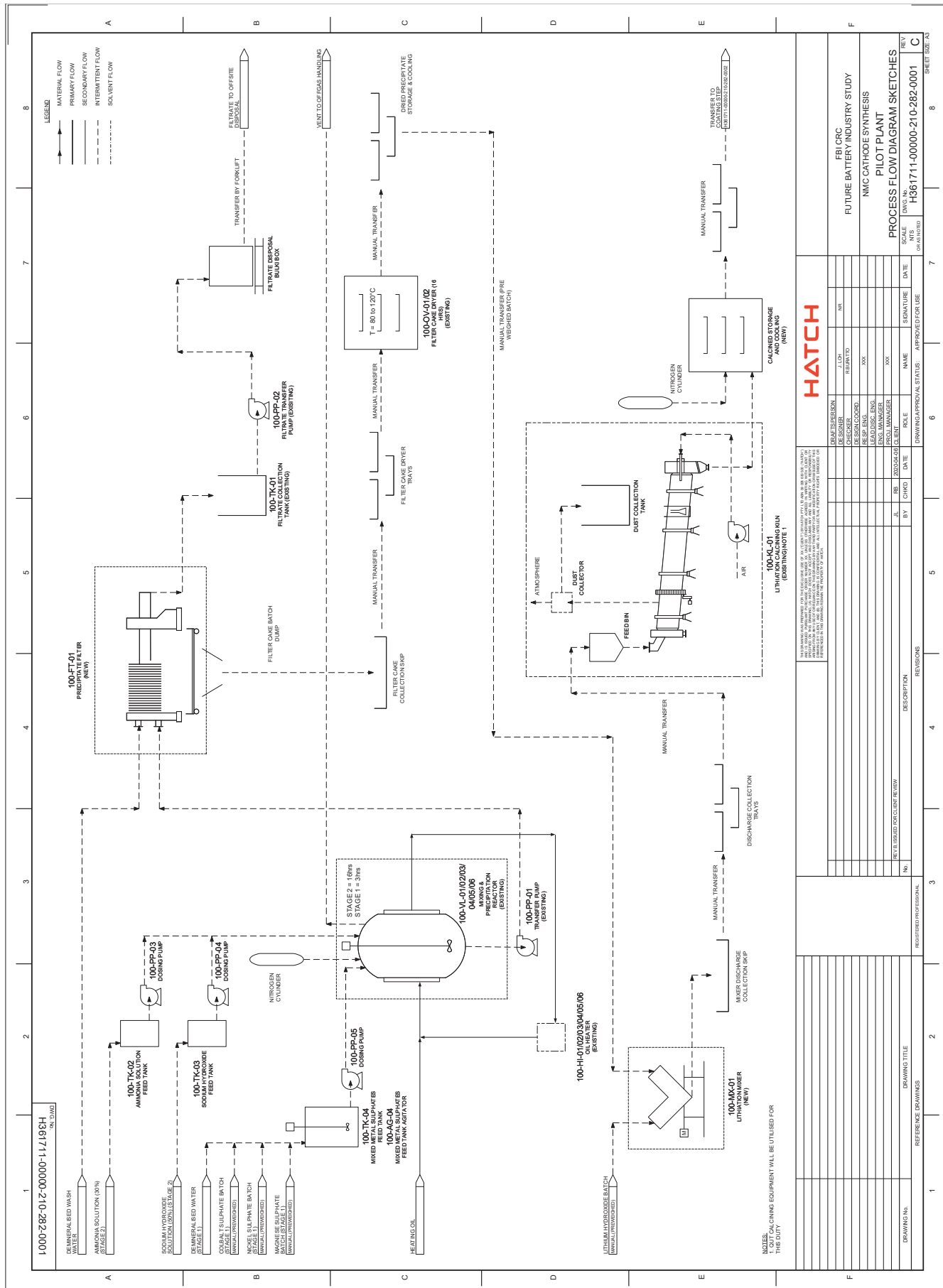
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Appendix

Appendix A	Process Flow Diagram Sketches	92 – 93
Appendix B	Process Design Criteria	94 – 102
Appendix C	Equipment List	103
Appendix D	Pilot Plant Capacity Estimate	104 – 106
Appendix E	Filtration Equipment	107 – 133
Appendix F	Calcine Kiln Equipment	134 – 142



Appendix A Process Flow Diagram Sketches





Appendix B Process Design Criteria



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PARAMETER	VALUE	UNITS	REF/CONF.	SOURCE	MASS & ENERGY BALANCE METHODOLOGY	REV
The following reference codes are used in this document						
Assumed / Indicative / Estimated:	A		4	FEL 4, Execution / Implementation Phase:	Chemical compound codes The following suffixes are used: (s) = solid phase (a) = aqueous phase (g) = gaseous phase (l) = liquid phase Note: Reactions and their extents are modelled in the order as stated in this document.	
Client's Data:	C		3	FEL 3, Feasibility Studies:		
Process Design Basis & Capacity Basis:	D		2	FEL 2, Pre-Feasibility Studies / Definition Phase:		
Engineering Calculation:	E		1	FEL 1, Scoping Studies / Preliminary Data:		
Hatch Recommended:	H		0	Order-of-Magnitude Studies / Sighter tests:		
Literature:	L					
Mass Balance:	M					
Plant Data:	P					
Regulatory Standards and Codes:	R					
Standard Industry Practice:	S					
Test Work:	T					
Vendor Originated Data:	V					
AREA 00000 PILOT PLANT						
General Area Parameters						
Availability	Variable	%		H1		0
Operating hours per day	24	hr/d		H1		0
Pilot plant throughput (nominal case)	Variable	dry kg/d		E1		
Battery cathode active material basis of design	NMC622	-		C2		0
100-HI-01/02/03/04/05/06 Oil Heater						
Energy Balance						
Temperature range	25 to 120	°C		H1		
Fuel source	Electric	-		P2		
Sizing Basis						
Supply	Vendor package	-		P2		

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PARAMETER	VALUE	UNITS	REF/CONF.	SOURCE	MASS & ENERGY BALANCE METHODOLOGY	REV
100-VL-01/02/03 Mixing						
Batch Chemical Mixing Mass Balance						
Deionized Water added per batch	10.3	L	E2			
Battery grade CoSO ₄ added per batch (CoSO ₄ ·7H ₂ O)	0.869	kg	E2			
Battery grade NiSO ₄ added per batch (NiSO ₄ ·6H ₂ O)	3.520	kg	E2			
Battery grade MnSO ₄ added per batch (MnSO ₄ ·H ₂ O)	0.696	kg	E2			
Mass & Energy Balance Chemistry						
Target mixed sulphate in solution	2.0	mol/L (aq)	L2			
Energy Balance						
Normal operating temperature	25 to 50	°C	L2			
Sizing Basis						
Number of reactor vessels	3	-	P2			
Reactor vessel volume (100%)	100	L	P2			
Reactor vessel volume used per batch	85	L	H2			
Residence time	3	h	L2			
Agitator speed	1000+	rpm	L2			
100-VL-01/02/03 Precipitation						
Batch Co-Precipitation Mass Balance						
NaOH solution (50% w/w) added per batch	20.4	kg	E2			
NH ₄ OH solution (30% w/w) added per batch	49.6	kg	E2			
Target pH	11.1 to 11.6	-	L2			
Target concentration of NaOH	2.0 to 4.0	mol/L (aq)	L2			
Target concentration of NH ₄ OH	0.36 to 17.0	mol/L (aq)	L2			
Co-precipitation atmosphere	inertN ₂	-	L2			



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PARAMETER	VALUE	UNITS	REF/CONF.	SOURCE	MASS & ENERGY BALANCE METHODOLOGY	REV
Mass & Energy Balance Chemistry						
Precipitation reactions taking place						
$\text{CoSO}_4(\text{aq}) + 2\text{NaOH}(\text{aq}) = \text{Co}(\text{OH})_2(\text{s}) + \text{Na}_2\text{SO}_4(\text{aq})$	100	%	L2			
$\text{NiSO}_4(\text{aq}) + 2\text{NaOH}(\text{aq}) = \text{Ni}(\text{OH})_2(\text{s}) + \text{Na}_2\text{SO}_4(\text{aq})$	100	%	L2			
$\text{MnSO}_4(\text{aq}) + 2\text{NaOH}(\text{aq}) = \text{Mn}(\text{OH})_2(\text{s}) + \text{Na}_2\text{SO}_4(\text{aq})$	100	%	L2			
Mass of Co-precipitate ($\text{Ni}_x\text{Mn}_y\text{Co}_z(\text{OH})$) per batch	8	kg	E2			
Energy Balance						
Normal operating temperature	50 to 60	°C	L2			
Sizing Basis						
Number of reactor vessels	3	-	P2			
Reactor vessel volume (100%)	100	L	P2			
Reactor vessel volume used per batch	85	L	H2			
Residence time	12 to 32	h	L2			
Agitator speed	1000+	rpm	L2			
100-FI-01 Precipitate Filter						
Batch Co-precipitation Filtration Mass Balance						
Feed solids concentration	8.8	%	E1			
Solids throughput	8.24	kg/batch	E1			
Deionised wash water	2	L/kg solids	A			
Wash efficiency (single volume)	90	%	A			
Filter cake moisture	15	w/w%	A			
Filtrate solids concentration	0	ppm	A			
Sizing Basis						
Number of filters	1.0	-	E1			
Type	pressure	-	H2			
Feed time	variable	min	H2			
Specific filtration rate	15	kg/m ² /h	A			
Wet pulp density	0.988	t/m ³	E2			
100-TK-01 Filtrate Collection Tank						
Mass Balance						

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PARAMETER	VALUE	UNITS	REF/CONF.	SOURCE	MASS & ENERGY BALANCE METHODOLOGY	REV
Filtrate solids concentration	0	ppm	A			
Litres per batch Co-precipitation	81	L/batch	E2			
Sizing Basis						
Batch filtration volumes (Based on 1m ³ bulki box)	10	No.	E2			
100-OV-01/02 Filter Cake Dryer						
Energy Balance						
Temperature range	80-120	°C	L2			
Heating medium	Electric	-	P2			
Sizing Basis						
Type	Pilot plant Vacuum Oven	-	P2			
Capacity	multi batch trays	No.	P2			
Number of drying ovens	2	No.	P2			
Drying residence time	12 to 24	hours	L2			
100-MX-01 Lithiation Mixer						
Mass Balance						
Mass of dried Co-precipitate per batch	8.2	kg	E2			
Mass of battery grade LiOH per batch	2.1	kg	E2			
Target stoichiometric excess LiOH	2.0	%	L2			



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PARAMETER	VALUE	UNITS	REF/CONF.	SOURCE	MASS & ENERGY BALANCE METHODOLOGY	REV
Sizing Basis						
Type	V mixer	-	H2			
Mixing time	Variable	hr	H2			
Mixer batch capacity	25	kg	H2			
100-KL-01 Lithiation Calcining Kiln						
Calcination Mass Balance						
Type of operation	Batch/Continuous	-	H2			
Oxygen addition	variable	L/h	L2			
Chemical Reaction Extent						
$\text{Ni}_x\text{Mn}_y\text{Co}_z(\text{OH})(\text{s}) + \text{LiOH}(\text{s}) = \text{Li}_x\text{Ni}_x\text{Mn}_y\text{Co}_z\text{O}_2(\text{s})$	Variable	%	L2			
Energy Balance						
Stage 1 Calcination Temperature	450 to 480	°C	L2			
Stage 2 Calcination Temperature	750	°C	L2			
Maximum/design calcination temperature	1000	°C	H2			
Cooled calcine temperature	100	°C	H2			
Sizing Basis						
Type	Electric rotary kiln c/w dust extraction system	-	H2			
Capacity	25	kg/h	H2			
Supply	Vendor package					
100-MX-02 Coating Mixer						
Mass Balance						
Mass of dried Co-precipitate per batch		kg	C1			
Mass of battery grade $\text{Al}(\text{OH})_3$ per batch		kg	C1			
Mass & Energy Balance Chemistry						
Temperature of operation	25	°C	A			
Sizing Basis						
Type	V mixer	-	H2			

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PARAMETER	VALUE	UNITS	REF/CONF.	SOURCE	MASS & ENERGY BALANCE METHODOLOGY	REV
100-TK-03 Filtrate Collection Tank						
100-KL-01/02 Lithiation Calcining Kiln						
100-ML-01 Coated Product Deagglomerator						
100-KL-04/05 Coating Calcining Oven						

Appendix C Equipment List



FBI CRC - Future Battery Industry Scene Setting Study

Pilot Plant Equipment List - Preliminary Equipment Specification

Existing = Existing equipment items at the BHPB Nickel Sulphate pilot plant

New = New equipment items required for the FBI CRC pilot plant

Note: Equipment item numbers are as noted on the Process Flow Diagram Sketches

Equipment Number	Equipment Name	Make/ Model	Equipment Description/Preliminary Specification
100-HI-01/02/03/04/05/06	OIL HEATER (EXISTING)		Type: Circulating oil electric heater. Heating capacity 7.5KW installed. Complete with circulating oil pump
100-VL-01/02/03/04/05/06	MIXING & PRECIPITATION REACTOR (EXISTING)		Type: Enclosed double skin glass vessel with elliptical top and bottom. Agitated. Capacity 100L.
100-AG-01/02/03/04/05/06	MIXING & PRECIPITATION REACTOR AGITATOR (EXISTING)		Type: Double flat blade. Stainless steel PTFE coated. Variable speed pneumatic drive. Speed range TBC.
100-PP-01	TRANSFER PUMP (EXISTING)		Type: Peristaltic electric drive. Capacity 320L/h at 7.5 bar. Electric drive 0.5 kW. Model Flowrox Atex
100-FT-01	PRECIPITATE FILTER (NEW)		Type: Plate and frame pressure type filter. Manually operated. Plate size 200mm x 200mm. Estimated filter area required 1m ² . Filter opening, closing and cake release: manual operation. Polypropylene plates.
100-TK-01	FILTRATE COLLECTION TANK (EXISTING)		Type: Standard 1m ³ plastic bulk box with built in forklift lift pallet
100-PP-02	FILTRATE TRANSFER PUMP (EXISTING)		Type: Peristaltic electric drive. Capacity 320L/h at 7.5 bar. Electric drive XX kW. Model Flowrox Atex
100-OV-01/02	FILTER CAKE DRYER (16 HRS) (NEW)		Type: Existing pilot plant/laboratory drying ovens. Multiple shelf capacity. Drying temperature range 60 to 120°C. Electric heating capacity XX kW.
100-MX-01	LITHIATION MIXER (NEW)	AWE Group SVP-0.05	Type: V-Mixer. Capacity 25L per chamber. Manual operation. Electric power 0.75 kW. Overall volume = 50L. Effective Volume = 20L.
100-KL-01/02	LITHIATION CALCINING OVEN (X2) (NEW)		Type: Horizontal rotary kiln, complete with feed bin, screw feeder, discharge cooler, discharge screw feeder, discharge hopper and exhaust gas extraction baghouse. Electric power requirement ZZ KW. Capacity 25dry kg per hour. Residence time 6 hours. Variable temperature control to range maximum operation 1000°C.
100-MX-02	COATING MIXER (NEW)	AWE Group SVP-0.05	Type: V-Mixer. Capacity 25L per chamber. Manual operation. Electric power 0.75 kW. Overall volume = 50L. Effective Volume = 20L.
100-KL-02	COATING CALCINING KILN (EXISTING) NOTE 1		Type: Horizontal rotary kiln, complete with feed bin, screw feeder, discharge cooler, discharge screw feeder, discharge hopper and exhaust gas extraction baghouse. Electric power requirement ZZ KW. Capacity 25dry kg per hour. Residence time 6 hours. Variable temperature control to range maximum operation 1000°C.

Appendix D Pilot Plant Capacity Estimate

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Engineering Report
Process Engineering
Future Battery Industry Scene Setting Study

FBI CRC Future Battery Industry Scene Setting Study

Pilot Plant Capacity Estimate

Basis: NMC622 Cathode Active Material

Mixing and Coprecipitation Reactor = 100L Nickel Sulphate pilot plant reactor vessel

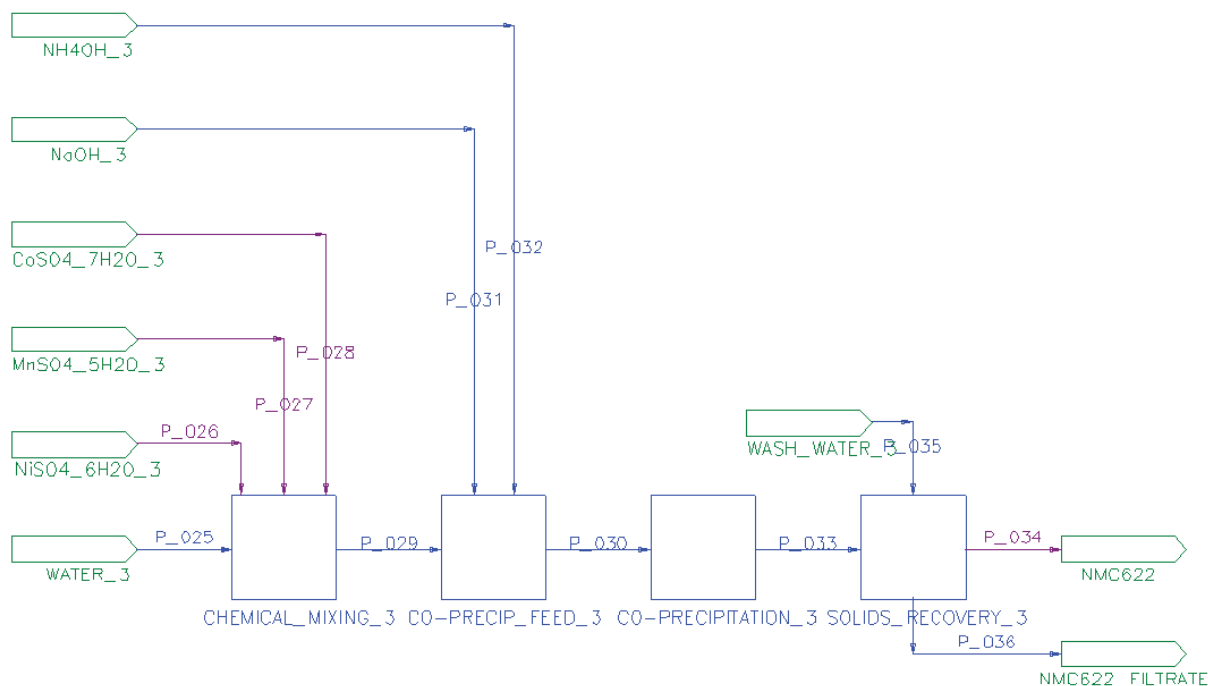
Batch Operation

Mass Balance Model Objective:

The key objective is to understand the process required to form NCM622 before lithiation. The model will focus on the chemical mixing and co-precipitation stages, which are the most complex. With additional attention on the batch mass balance and system operating temperatures.

The model is based on utilizing the batch volume of the 100L reactor vessels at the BHPB nickel sulphate pilot plant, CSIRO, Waterford. The mass balance model has been developed to utilize the reactor vessel selected operating volume of 85L.

To simulate the batch process in SysCAD a continuous model was developed. An hour was made equivalent to one NCM622 batch. A layout of the model is shown below.



Process stage boxes have been used to simulate each step of the process. It has been assumed in each step that satisfactory residence time has been given for a complete reaction to occur.

- Chemical mixing – Mixed sulphate dissolution reactions
- Co-precipitation feed – Mixing of reactants before the reaction takes place. No reaction occurs at this stage. Identification of feed composition before hydroxide reaction.
- Co-precipitation – Hydroxide reactions with sodium hydroxide and ammonium hydroxide addition.
- Solids Recovery – Filtering of precipitates from the co-precipitation mixture. A wash ratio of 1kg per kg of dry solids equivalent has been assumed for solids washing. The stream split is set with all solids to the NCM622 stream and remaining liquid to NCM622 filtrate.

Basis:

- 85L active volume in reactor
- NaOH reagent solution concentration: 50 w/w%
- NH₄OH reagent solution concentration: 30 w/w%
- Filtered solids wash ratio of 1kg demineralized water/ kg dry solids

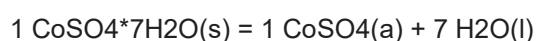
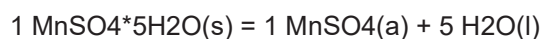
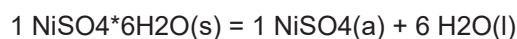
Assumptions:

- Nickel, manganese and cobalt precipitate as individual hydroxides as opposed to Ni₆Mn₂Co₂(OH)
- Negligible reaction energies for nickel & cobalt dissolution
- All reactions reach extent of 100%
- Room temperature: 25°C
- Separation is ideal (all solids to one stream; liquids to another stream)

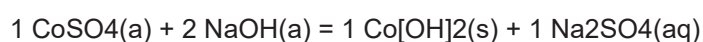
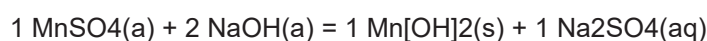
Assumed Reactions:

Modelled as individual reactions due to the lack of information on Ni₆Mn₂Co₂(OH). All reactions achieve full completion and it has been assumed there are no other side reactions. Metal hydroxides do not react further with water after co-precipitation.

Mixed Sulphate Reactions:



Hydroxide Reactions:



Results: The results of the model are summarised in the following table.

Material	Units	NMC622
Aqueous sulphate concentration	mol/L	2
NaOH concentration	mol/L	3
NH ₄ OH concentration	mol/L	2
Ni _x Mn _y Co _z (OH) per reactor batch operation	kg	8.2

Outcomes:

- Exothermic Manganese dissolution reaction will require cooling from 65°C to 50-60°C
- Volume restricted in producing NMC 622 material as a high NH₄OH concentration is required (17mol/L). In the calculation this was restricted to 5mol/L resulting in a small batch of Ni_xMn_yCo_z(OH) product.

Appendix E Filtration Equipment

HATCH

Queensland University of Technology
Future Battery Industry Scene Setting Study
H361711

Engineering Report
Process Engineering
Future Battery Industry Scene Setting Study

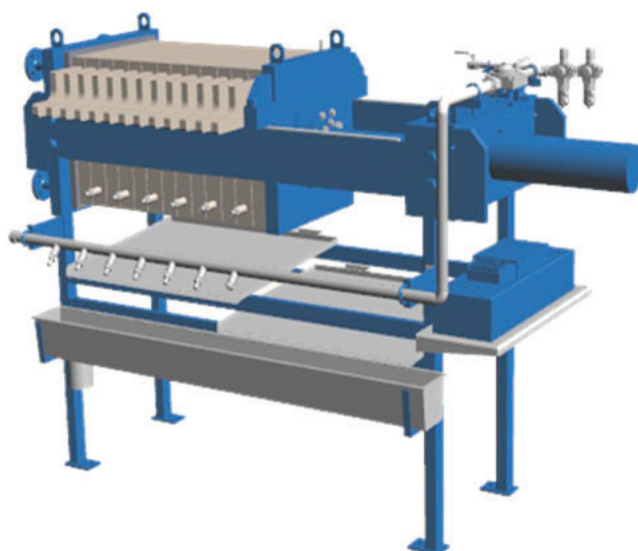
E.1 IFS Consultants

From: Wilfried Wimmeler <wilfried@ifs-consultants.com.au>
Sent: Wednesday, April 1, 2020 12:12 PM
To: Buratto, Rob <rob.buratto@hatch.com>
Subject: RE: Pilot Plant Filtration Duty

Hello Rob,

Thank you for your email. I am in home quarantine for 2 more days after a visit to Chile and a challenging few days to get back home... The smallest possible press with diaphragm / membrane plate is a 470 x 470 mm plate format.

We build a number of these presses like the one on the photo – with changing number of plates.





Most times such a press gets fed with an Air Driven diaphragm pump, which does not require pressure volum regulation – it is self regulating....

We also 'clad' some of the filters in stainless and can offer to make a spray cover as attached.

In this case you may wish to consider maybe 3-6 chambers – and extendable to 10 – as the few more centimeters in length do not contribute to an additoonal price.

4 chambers = 19 L unsqueezed and 1.3 m2

Happy to help

Regards

Wilfried Wimmmler



Innovative Filtration Solutions
Consultants & Equipment Suppliers

Wilfried@ifs-consultants.com.au

www.ifs-consultants.com.au

Mobile: +61 (0) 408 526 018

Ph.: +61 (0)8 6555 1807

From: Buratto, Rob <rob.buratto@hatch.com>
Sent: Wednesday, 1 April 2020 8:55 AM
To: Wilfried Wimmeler <wilfried@ifs-consultants.com.au>
Subject: Pilot Plant Filtration Duty

Wilfred
Hi

I trust you are keeping well and safe with the current coronavirus situation.
Here at Hatch (Perth) we are now all working from home.

I have a query about a filtration duty which you may be able to assist with.

We are working on a pilot plant design for lithium ion battery materials preparation (CAM – cathode active material).

I wanted to enquire about the smallest pressure (plate and frame?) type filter units which you may have in your standard range of equipment?

The duty is to filter a precipitation process product (NiXCoyMnz.OH).

Volume of slurry = 100 litres

% solids = 8 to 12 % w/w

Solids sg = 2.2

Mass of solids is approximately 7 to 12 dry kg.

Estimated particle size is approximately 5 to 10 micron.

Feed rate (based on precipitation discharge pump capacity) 200 to 400 litres per hour. Peristaltic type pump. This feed rate can be reduced if necessary because the operation is batch and there is about 6 hours between batches.

No filtration data is available, but approximating this material to something similar I estimate we would need approximately 0.6 to 1.0 m² filter area.

Do you have any standard equipment type small enough for this duty? We are looking for a simple filter operation, manually operated as much as possible to suit the pilot plant type operation.

Or, what would you suggest is the best option for this duty.

Please let me know if you have any questions.

Regards

Rob Buratto

NOTICE - This message from Hatch is intended only for the use of the individual or entity to which it is addressed and may contain information which is privileged, confidential or proprietary. Internet communications cannot be guaranteed to be secure or error-free as information could be intercepted, corrupted, lost, arrive late or contain viruses. By communicating with us via e-mail, you accept such risks. When addressed to our clients, any information, drawings, opinions or advice (collectively, "information") contained in this e-mail is subject to the terms and conditions expressed in the governing agreements. Where no such agreement exists, the recipient shall neither rely upon nor disclose to others, such information without our written consent. Unless otherwise agreed, we do not assume any liability with respect to the accuracy or completeness of the information set out in this e-mail. If you have received this message in error, please notify us immediately by return e-mail and destroy and delete the message from your computer.



E.2 Outotec

H361711-00000-210-066-0003, Rev. D,

Ver: 04.03

From: Victor Ortega <victor.ortega@outotec.com>

Sent: Wednesday, April 1, 2020 9:04 AM

To: Buratto, Rob <rob.buratto@hatch.com>

Subject: RE: Pilot Plant Filtration Duty

Hi Rob,

I attached some information about the test filters.

- The datasheet for the PF 0.4
- The Manual for the PF 0.1 that it will give you a clear view of the things that you will require for the PF 0.4

Thanks

Victor

From: Buratto, Rob <rob.buratto@hatch.com>

Sent: Wednesday, 1 April 2020 8:47 AM

To: Victor Ortega <victor.ortega@outotec.com>

Subject: RE: Pilot Plant Filtration Duty

Victor

Hi

Thanks for the response.

The Larox PF 0.4 Test Filter is an option that can be made to work. The filtration times can be extended as the operation will be batch basis and we have about 6 hours between batches. At this stage we do not need a price as we are trying to find an equipment option which can be made to work.

So, can you provide further technical detail about the test filter?

Do you have a brochure/specification sheet/GA drawing/photos of the test filter for us to assess this, services required, power, physical dimensions, whether this is skid mounted etc.

Regards

Rob Buratto

From: Victor Ortega <victor.ortega@outotec.com>

Sent: Wednesday, April 1, 2020 8:39 AM
To: Buratto, Rob <rob.buratto@hatch.com>
Cc: Shane Freeman <shane.freeman@outotec.com>
Subject: RE: Pilot Plant Filtration Duty

Hi Rob,

Thanks for your email.

Probably an option, it will be a Test Filter Larox PF 0.4 (0.4 m²) but I do not have price for this one and the delivery time is around 16 weeks (probably with the Covid-19, there will be some delays) Ex works.

If you are interested in the test filter, let me know so I can get a price for you.

Thanks

Victor

From: Buratto, Rob <rob.buratto@hatch.com>
Sent: Tuesday, 31 March 2020 8:35 AM
To: Victor Ortega <victor.ortega@outotec.com>
Subject: Pilot Plant Filtration Duty

Victor

Hi

You emailed me a week or so ago about meeting to discuss equipment/projects/etc that we are working on at Hatch. Unfortunately events associated with the coronavirus have overtaken things.

I trust you are keeping well and safe with the current coronavirus situation.
Here at Hatch (Perth) we are now all working from home.

I have a query about a filtration duty which you may be able to assist with.

We are working on a pilot plant design for lithium ion battery materials preparation (CAM – cathode active material).

I wanted to enquire about the smallest pressure (plate and frame?) type filter units which you may have in your standard range of equipment?

The duty is to filter a precipitation process product (Ni_xCo_yMn_z.OH).

Volume of slurry = 100 litres

% solids = 8 to 12 % w/w

Mass of solids is approximately 7 to 12 dry kg.

Estimated particle size is approximately 5 to 10 micron.

Feed rate (based on precipitation discharge pump capacity) 200 to 400 litres per hour. Peristaltic type pump.

No filtration data is available, but approximating this material to something similar I estimate we would need approximately 0.6 to 1.0 m² filter area.

Do you have any standard equipment type small enough for this duty?

Or, what would you suggest is the best option for this duty.

Please let me know if you have any questions.

Regards

Rob Buratto

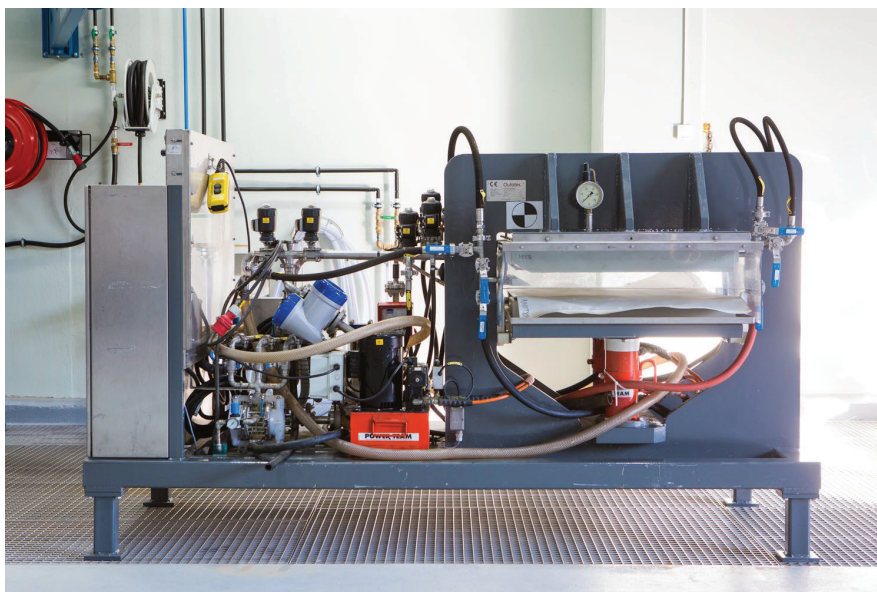
OUTOTEC LAROX® PF 0.4**Features**

The Outotec LAROX® PF 0.4 is used for collecting the data required for the sizing, specification and warranty of the Outotec LAROX® pressure filter series. This filter has one plate with an area of 0.4m² and can carry out all the process steps that can be done on a production scale unit.

The instrumentation of the test unit provides detailed information about each test filtration cycle. The test cycle process parameters such as flows and quantities, pressures, temperatures and conductivities are shown on-line in the unit's process Operator interface unit (OIU).

Benefits

- Chamber thickness 33 or 45 mm
- Identifies the total cycle time including individual steps [min].
- Confirms the filter capacity [L/m²h or kg/m²h D.S.].
- Shows the residual moisture in the cake [%].
- Shows the amount of solids in the filtrate [mg/L].
- Indicates the amount of washing water needed [L/kg of wash liquid per dry solids content in the cake, l/l of wash liquid per the feed volume].
- Shows the efficiency of washing, with the result for material removed from or remaining in the washed cake.
- Identifies the air consumption [m³ /m²min].
- Identifies the correct choice of filter cloth.





Operation Instructions

LABORATORY UNIT

LAROX

PF 0.1

Outotec (Filters) Oy
Tukkikatu 1, P.O. Box 29
FI-53101 Lappeenranta

TABLE OF CONTENT

FOR SAFETY REASONS	3
1.1 PURPOSE OF USE	4
1.2 TECHNICAL DESCRIPTION	4
1.2.1 Slurry tank	4
1.2.2 Mixer for slurry tank	4
1.2.3 Feed pump	5
1.2.4 Laboratory pressure filter PF 0.1 H2	5
1.2.5 Pressure water station	5
1.2.6 Piping	5
1.2.7 Cake washing pump	5
1.2.8 Chamber rings 15mm x Ø 450mm and 30mm x Ø 450mm	5
1.3 PREPARATIONS FOR THE TEST	6
1.3.1 Defining the test aims	6
1.3.2 Preparation of the slurry	6
1.3.3 Preparing the test filter for operation	7
1.3.4 Test devices	7
1.3.5 Choice of the filter cloth	7
1.4 TEST PROCEDURE (Short program)	8
1.4.1 Filtration	8
1.4.2 Pressing	8
1.4.3 Air drying	9
1.4.4 Discharge	9
1.4.5 Summary	9
1.5 TEST PROCEDURE (Long program)	10
1.5.1 Filtration	11
1.5.2 Pressing I	11
1.5.3 Washing	11
1.5.4 Pressing II	12
1.5.5 Air drying	12
1.5.6 Discharge	12
1.5.7 Summary	12
1.6 RECORDING THE TEST RESULTS, ANALYSING AND REPORTING	13
1.6.1 Data recorded before the test	14
1.6.2 Measuring results recorded during the test	14
1.6.3 Analysis and calculations	15
1.6.4 Complementary remarks	16
1.7 USE OF TEST RESULTS	16

FOR SAFETY REASONS

BEFORE STARTING THE FILTER, READ THROUGH AND MAKE SURE YOU UNDERSTAND THE OPERATION AND MAINTENANCE MANUAL.

NEVER START ANY MAINTENANCE OR REPAIR WORK DURING THE OPERATION OF THE FILTER.

REMEMBER THIS:

1. Before starting the filter, make sure there is nobody in the vicinity of the filter.
2. Before starting the filter, make sure that there are no strange objects between the plates.
3. Take into account that the operation of the filter requires the use of pressing air and water as well as hydraulic oils. The slurry hoses operate under pressure as well. A damage in any of these pressure pipelines may be dangerous.
4. Take into account that the filter operates automatically and thus different parts may move without any separate measures.
5. When working close to the filter, always use a goggles and overalls with long sleeves. When filtering corrosive or other dangerous materials, take into account special protection requirements.
6. During maintenance, repair or adjustment work, switch off the electric and make sure there is no pressure in the pipelines.
7. Never remove the cake, cloth or other objects from between the plates before the plates are totally open and the movement of the plates is stopped. Always mind your hands.
8. Make sure that the assembly of the unit is steady. Lock the wheels (optional) under the feet. The location of the unit should be remote avoiding passing through traffic to disturb unit or persons working with it.
9. Slurry tank is equipped with an agitator. Mind your hands and do not drop any irrelevant objects into the tank while the agitator is running.
10. Do not take this filter to ex proof area.
11. This test filter is not meant for continuous production use. Do not leave the test filter unattended during the operation.

1.1 PURPOSE OF USE

The Outotec Larox laboratory pressure filter type PF 0.1 H2 is meant to be used in laboratories, which get samples for the filtration tests. The expected results are:

- finding out the suitability of the Larox automatic pressure filter for a particular slurry filtration
- finding out the preliminary working measurements for this slurry
- finding out the best possible cycle length for test runs with PF 1.7 m² mobile filter unit at the customer's plant

Tests on the Outotec Larox PF 0.1 H2 laboratory pressure filter give data for the preliminary ascertaining of the following necessary measurements:

- the total length of cycle time including individual phases (min)
- filter capacity (l/m²h or kg/m²h)
- residual moisture in the cake (% H₂O)
- solids in the filtrate (mg/l)
- amount of washing water needed (l/kg for the dry solids content in the cake, l/l for the slurry)
- the result for the washed cake (% dry solids)
- air consumption (m³/m² min)
- choice of the cloth

1.2 TECHNICAL DESCRIPTION

The Outotec Larox laboratory pressure filter PF 0.1 H2 is manufactured according to the drawing F000966. The different parts are placed on a U-profile base. The main dimensions are 1650 x 600 x 1300 mm (L x W x H).

The components are as follows:

1.2.1 Slurry tank

- volume 100 l
- drain pipe connection, size DN 50
- material AISI 316

1.2.2 Mixer for slurry tank

- mixer, electrically operated drive
- drive gear, ration 1:18
- type BONFIGLIOLI MVF/P49
- electric motor, rating 0,25 kW, 220-480 VAC, 50/60 Hz, rotation speed 1500/1800 rpm
- shaft and mixer of material AISI 316

1.2.3 Feed pump

WILDEN P2R, compressed air operated. The pump is made of polypropylene, which withstands most applications well. When corrosive liquids are being filtered, it is advisable to consult the importer about the durability of the pump.

1.2.4 Laboratory pressure filter PF 0.1 H2

- filter area 0.1 m²
- the frame and all parts in contact with the liquid are made of material AISI 316, polypropylene grid
- pressing diaphragm and sealing made of rubber
- the closing of the filter is effected by a hydraulic cylinder. The pressure gauge of the hydraulic pump must show at least 600 bar

1.2.5 Pressure water station

- the pressure water station is compressed air operated, volume 10 l
- operation pressure max. 16 bar
- construction material AISI 304

1.2.6 Piping

PF 0.1 H2 is piped as follows:

- slurry pipe from the slurry tank through the pump to the pressure filter, materials AISI 316/rubber hoses
- compressed-air piping to the driving devices (mixer, pumps) AISI316
- water pipes AISI 316

Parts of the frame are sandblasted and painted red with an epoxy paint, except for parts made of stainless steel, pump and hydraulic mechanism.

1.2.7 Cake washing pump

- Wilden P1
- made of polypropylene, which withstands most liquids well
- maximum working pressure 8 bars

1.2.8 Chamber rings 15mm x Ø 450mm and 30mm x Ø 450mm

- standard chamber thickness is 45 mm
- chamber thickness can be transformed by means of chamber rings as follows:

45 – 12	= 33 mm
45 + 15	= 60 mm
45 + 30	= 75 mm
45 + 30 + 15	= 90 mm

1.3 PREPARATIONS FOR THE TEST

A thorough preliminary preparation is essential for a successful test.

1.3.1 Defining the test aims

Before starting the test procedure the prevailing circumstances concerning the slurry must be defined very clearly:

- slurry D.S. content and specific gravity
- slurry temperature
- slurry pH
- wash water quality
- wash water pH and temperature

These circumstances should be kept in the test.

In addition, the test filtration aims should be defined: Is the aim:

- the best possible dry solids content
- the lowest possible cake moisture
- the cleanest possible filtrate
- the most economical cake washing result
- the right filter cloth
- or optimizing some of these together

When the aim is known, it is easier to make a test filtration plan, which helps in reaching the aim as directly as possible.

1.3.2 Preparation of the slurry

There must be enough slurry for the test procedure (re-pulping should be avoided, because the slurry from re-pulping is not always homogenous with the original slurry).

The slurry needed for one test depends on the slurry D.S. content and its filtration characteristics.

The aim is that to do so many tests will be carried out that pumping, pressing and drying times will become optimized. Washing time and results will also be optimized in the washing tests.

If the sample has arrived in dry form or if its solid contents must be changed, it has to be mixed carefully and the mixing procedure and the quality of the liquid must be known.

It is essential to mix the slurry sample carefully. Make sure to collect all the settled solids into the mixing tank.

1.3.3 Preparing the test filter for operation

Place the filter on a suitable base to make the working conditions pleasant. The most suitable height near the chamber is 90 – 100 cm.

Connect the pressure air connections (6-7 bar) for air drying and actuator.

If cake washing is included in the test, connect the wash water tank and pressure air coupling in their places. See enclosure T-5191.

Fill in the pressing water tank (7) with clean water.

Pour the slurry to be mixed into the mixing tank (2). The mixing capacity of the mixer (1) is not always sufficient. Therefore let the diaphragm pump circulate the slurry ->the circulation valve (A) open and the slurry valve (B) closed.

1.3.4 Test devices

- 3 pcs of filtrate vessels (5 l), equipped with scale (5 pcs in the test that requires washing)
- thermometer 0-100 °C
- pH indicator / pH papers
- scale 0-15 kg
- stop watch
- calculator
- sample bags and bottles
- moisture measuring set or oven + precision scale
- slurry density scale

1.3.5 Choice of the filter cloth

Factors that effect the choice of the cloth

- slurry temperature
- slurry pH
- particle size distribution of slurry
- the solids content requirements for the filtrate

1.4 TEST PROCEDURE (Short program)

After the preparations the first test filtration is carried out.

The slurry must be in the mixing phase (the mixer is in operation and the diaphragm pump keeps the circulation on), the filtration temperature and pH must be right and the possible additives added.

Place the chosen and weighed filter cloth in its place and close the filter using the foot treadle (4) on the filter side.

The hydraulic pressure meter must be at least 600 bars before the filtration can be started.

Make sure before starting that

- the drying air valve (C) is open
- close the slurry discharge valve (E)
- air should now be flowing into the filter, penetrate the filter cloth and come out of the filtrate hose
- close the drying air valve (C)
- place the filtrate vessel under the filtrate hose

1.4.1 Filtration

Take a sample from the slurry. Measure the slurry level height in the slurry tank before pumping. Adjust the diaphragm pump pumping speed (normally from medium speed to maximum).

Close the circulation valve (A) and open the slurry valve (B) immediately and start the stopwatch.

Let the pump go on pumping the slurry long enough. (If the slurry can be filtered very easily, the pumping must be stopped when the cake thickness is near maximum, otherwise the pump and the pipes may get blocked).

Close the slurry valve (B) and stop the stopwatch, open the circulation valve (A) and lower the pump speed for reaching a suitable circulation.

Read the pumping time and write it down.

Measure the slurry level height after pumping, minus it from the height before pumping and write down the pumped slurry amount in the data sheet (this measuring should be done during the diaphragm pressing).

1.4.2 Pressing

Put the filtrate hose into the following filtrate vessel. Start the diaphragm pressing by turning the three-way valve (P) to position ON. The pressing water pump (8) starts and pumps the water above the diaphragm. When the pressure raises over 5 bars, start the stopwatch.

Note how the filtrate flows out and the point where the filtrate flow weakens very essentially and where it flows very little.

With this information the pressing time can be optimized in the following tests (the diaphragm pressing pressure can be changed by adjusting the water pump air pressure.)

When the filtrate flow stops, stop the stopwatch and open the manifold drain valve (E) about 15-20 seconds before the end of pressing time.

1.4.3 Air drying

Open the drying air valve (C) carefully. Air flows now out of the manifold drain valve and cleans the pipes.

Adjust the airflow speed. Close the slurry discharge valve (E). Turn the diaphragm pressing three-way valve (P) to position OFF. Stop the stopwatch. Water flows back into the pressing water tank (7). Start the stopwatch when opening the valve P. Watch the filtrate flow as the drying proceeds. This helps in optimizing the drying time in the following tests.

When the filtrate flow stops, close the drying air valve (C) and stop the watch.

Open the manifold drain valve (E) to remove the pressure from the filtration chamber. After this the chamber can be opened safely.

1.4.4 Discharge

Remove the pressure from hydraulic closing pump (4) and the filter chamber opens. Lift the cake with cloth on the scale and weigh it. Clean the cloth and the filter chamber for the following test. Note if the cake sticks to the diaphragm or the cloth. Note also how easily the cloth is cleaned.

1.4.5 Summary

The test that is carried out in the prescribed way gives by far the best possible cake moisture but the dry solids capacity is not the best possible.

A detailed test plan has to be made for the following tests and the slurry to be used and the test aims have to be taken into consideration.

If there is enough slurry to be used it is worth making a test plan where many variables can be changed.

If there is so little amount of slurry that it is enough only for a few tests then the most essential factors i.e. the drying and pressing times are recommended to be used as variables.

When several tests are made it is very important to repeat all standard phases and the test procedure as well as possible. In this way the test results can be compared to each other in the best possible way.

The following list shows in the order of importance those variables that should be kept constant during the test. If there is only a little amount of slurry for the test, keep constant as many variables as possible from the beginning of the below mentioned list. If there is enough slurry all these can be regarded as variables.

- slurry pH
- slurry temperature
- pressing pressure
- pumping pressure
- air pressure
- slurry D.S. content
- drying air flowing speed
- pumping time
- pressing time
- drying time

Every variable should be handled test-by-test or based on what is essential for a successful test.

1.5 TEST PROCEDURE (Long program)

After preparations start the first test filtration.

The slurry must be mixed (the mixer in operation and the diaphragm pump keeps the circulation on), the filtration temperature and pH must be right and the possible additives added.

The wash liquid must be ready and its temperature adjusted.

Place the chosen and weighed filter cloth in its place and close the pressure filter with the foot treadle on the filter side. The hydraulic pressure meter must show at least 600 bars.

Make sure before the filtration the right position of the valves:

- open the drying air valve (C)
- close the manifold drain valve (E)
- air should now be flowing into the filter, through the filter cloth and coming out of the filtrate hose
- close the drying air valve (C)
- place the filtrate vessel under the filtrate hose

1.5.1 Filtration

Take a sample from the slurry. Measure the slurry level height in the slurry tank before pumping. Adjust the diaphragm pump pumping speed (normally at maximum).

Close the circulation valve (A) and open the slurry valve (B) immediately and start the stopwatch. Let the pump go on pumping the slurry long enough (if the slurry can be filtered easily, the pumping must be stopped when the cake thickness is near maximum, otherwise the pump and the pipes may get blocked).

Close the slurry valve (B), stop the stopwatch, open the circulation valve (A) and lower the pump speed for reaching a suitable circulation.

Read the pumping time and write it down. Measure the slurry level height after pumping, minus it from the height before pumping and write down the pumped slurry amount in the data sheets (this measuring should be done during the diaphragm pressing).

1.5.2 Pressing I

Put the filtrate hose into the following filtrate vessel. Start the diaphragm pressing by turning the three-way valve (P) to position ON. The water pump (8) starts operating and pumps the water above the diaphragm. When the pressure raises over 5 bars, start the stopwatch. Note how the filtrate flows out and the point in which the filtrate flow weakens essentially and where it flows very little. With this information the pressing time can be optimized in the following tests.

When there is no more filtrate, stop the stopwatch and open the slurry discharge valve (E).

With some slurries I pressing time is very short to ensure successful washing and sometimes it must be left out. Also the lower diaphragm pressing pressure can be proved during I pressing (the diaphragm pressing pressure can be adjusted by changing the water pump air pressure).

1.5.3 Washing

Check the wash liquid level height. Make sure that the suction end of the wash pump hose is in the wash liquid vessel. Open the air valve (G) to the wash pump. Close the manifold drain valve (E). Put the filtrate hose into the following filtrate vessel. Open the wash liquid valve (J) and turn the three-way valve (P) for diaphragm pressing to position OFF.

Start the stopwatch and check the lowering of wash liquid level height. Close the wash liquid valve (J) when the planned liquid amount has flowed into the filter.

Stop the stopwatch. If the intended wash liquid amount does not go into the filter, measure the amount that flowed into the filter and the flowing time.

1.5.4 Pressing II

Put the filtrate hose into the following filtrate vessel. Start the diaphragm pressing by turning the three-way valve (P) to position ON. The water pump (8) starts and pumps the water above the diaphragm. When the pressure raises over 5 bars, start the stopwatch.

Note how the filtrate flows out and the point in which the filtrate flow weakens essentially and the point where it flows very little. With this information the pressing time can be optimized in the following tests.

When there is no more filtrate, stop the stopwatch and open the manifold drain valve (E).

1.5.5 Air drying

Open the drying air valve (C) carefully. Air flows now out of the slurry discharge valve (E) and cleans the pipes. Adjust the airflow speed. Close the slurry pipe discharge valve (E). Turn the diaphragm pressing three-way valve (P) to position OFF.

The water flows back into the water tank (7). Start the stopwatch when the air flows through the filtrate hose. Watch the filtrate flow as the drying proceeds. This helps in minimizing the drying time in the following tests.

When the filtrate flow stops, close the drying air valve (C) and stop the watch.

Open the manifold drain valve (E) to remove the pressure from the filtration chamber. Only after this the chamber can be opened safely.

1.5.6 Discharge

Remove the pressure from hydraulic closing pump (4) and the filter chamber opens. Lift the cake with cloths on the scale and weigh it. Rinse the cloth and the filter chamber for the next test. Note if the cake sticks on the diaphragm and the cloth. Also note how easily the cloth is cleaned.

1.5.7 Summary

The test that is carried out in the prescribed way gives by far the best possible cake moisture but the dry solids capacity and the cake washing result are not the best possible.

A detailed test plan has to be made for the following tests and the slurry used and the test aims have to be taken into consideration.

If there is enough slurry to be used it is worth making a test plan where many variables can be changed.

If there is so little amount of slurry that it is enough only for a few tests then the most essential factors i.e. the drying and pressing times are recommended to be used as variables.

When several tests are made it is very important to repeat all standard phases and the test procedure as well as possible. In this way the test results can be compared to each other in the best possible way.

The following list shows in the order of importance those variables that should be kept constant during the test. If the amount of slurry for the test is small, keep constant as many variables as possible from the beginning of the below mentioned list. If there is enough slurry all these can be regarded as variables.

- slurry pH
- slurry temperature
- pressing pressure
- pumping pressure
- air pressure
- slurry D.S. content
- drying air flowing speed
- pumping time
- I pressing time
- washing time
- II pressing time
- drying time
- wash liquid amount

Every variable should be handled test by test or based on what is essential for a successful test.

1.6 RECORDING THE TEST RESULTS, ANALYSING AND REPORTING

The tests that are carried out are written down in the enclosed data sheets. For bigger test series it is necessary to make a separate description, chart, graphic, etc. of the research subject in question.

The information written down in the data sheets can be divided in four groups:

- data before tests
- measuring results during the test
- results during analysis and calculation
- complementary remarks

1.6.1 Data recorded before the test

- customer data
- test filter type and area
- the exact name and chemical formula of the slurry to be filtered
- place and date of the test and name of the person who carried out the test
- filter cloth
- slurry pH, temperature, slurry D.S content and density

1.6.2 Measuring results recorded during the test

- pumping time (min) = time used for pumping (write down minimum 1.0min)
- I pressing time (min)
- washing time (min)
- II pressing time (min)
- air drying time (min)
- "dead time" = 4 min
- feed pressure approx. 0.9 times the used air pressure for diaphragm pump
- the used maximum pressure is written down as the pressing water pressure
- the wash liquid pressure is the same as the pressure of the used air write down the rotameter show as air flow speed during the drying phase (try to keep the flowing speed constant during the entire drying phase, max 300 l min)
- write down the difference of the tank level height before and after pumping as the slurry amount
- the wash liquid amount is the amount which flowed into the filter
- the filtrate amount is a total amount, but it must be measured separately:
 - during pumping
 - during I diaphragm pressing
 - during washing
 - during II pressing
 - during air drying
- slurry temperature and pH
- wash liquid temperature and pH
- filtrate temperature and pH
- cake average thickness
- cake weight

1.6.3 Analysis and calculations

Measure the moisture and washing result if it is possible with the existing equipment. Otherwise leave it for the customer to be measured.

$$\text{Cake moisture [\%]} = \frac{(\text{wet weight} - \text{dry weight})}{\text{wet weight}} \times 100$$

Measure the slurry liter weight from the slurry sample and the dry solids content and the grain size distribution. To measure the dry solids content weigh the slurry sample (e.g. 250 ml) and dry it.

$$\text{Slurry D.S. content [\%]} = \frac{\text{dry weight}}{\text{wet weight}} \times 100$$

The dry solids content can be measured in the filtrate sample. Minus the moisture from the cake weight.

The dry solids capacity is calculated on dry weight C (kg/m²h) :

$$C = \frac{W \times 60}{A \times T}, \text{ where}$$

W = cake weight (kg)

A = filter area (m²)

T = total cycle time (min)

The total cycle time T = pumping time (minimum 1.0 min) + I pressing time + washing time + II pressing time + drying time + 4 min (cake discharge, cloth washing etc. "dead time").

The slurry capacity is calculated with the same formula. The slurry amount (l) replaces the cake dry weight W and the result is (l/m²h).

In the filter capacity W = total amount of filtrates -wash liquid amount.

$$\text{Wash liquid consumption } Q = \frac{V \times 60}{A \times C \times T (m^3 / t \text{ D.S.})}$$

where

V = used wash liquid(l)

A = filter area (m²) C = dry solids capacity (kg/m²h) T = total cycle time (min)

1.6.4 Complementary remarks

The chemicals and the amounts of chemicals used for slurry improving.
 How well the cake keeps together and how fragile or glutinous it is.
 Cake sticking on the diaphragm and cloth.
 How easily the cloth is washed.
 How clear the filtrates are by visual inspection.

1.7 USE OF TEST RESULTS

The primary aim of Outotec Larox PF 0.1 test filter is to find out the suitability of the automatic pressure filter for the slurry in question. The dimensioning should also be made sure with a 2.5 m² test filter at the customer's plant.

If a preliminary estimate is given of the necessary filtration area in the customer's process, the following should be known:

G_a = yearly filtrated dry solids amount (kg)
 T = yearly operation hours (h) in continuous three shift work (8000 h)
 C = based on the test the filter dry solids capacity (kg/m²h)
 n = utilization rate (90 %)

$$\text{The necessary filtration area } A \left[m^2 \right] = \frac{G_a \times 100 \%}{T \times C \times n}$$

All test filtration results are recorded in Outotec (Filters) Oy Data Bank file. This file is for making the following test filtrations easier. To make this file as perfect as possible, the data sheets should be filled in as carefully as possible and send them together with the written report to be recorded in the file.

E.3 GBL Process

H361711-00000-210-066-0003, Rev. D,

From: gb@gbprocess.com <gb@gbprocess.com>

Sent: Tuesday, March 31, 2020 10:26 AM

To: Buratto, Rob <rob.buratto@hatch.com>

Cc: pn@gbprocess.com

Subject: [QT19-104] RE: Pilot Plant Filtration Duty

Dear Rob,

Our existing pilot plant filters are most likely OK for your duty. See further below.

From the info given, I believe the DrM FUNDABAC filter is the most suitable due to the relative low feed solids, the high product value (and thus high recovery required). Thus, we strongly recommend to do bench scale TSD (true scale down) testwork (as what we are doing for RTA) which requires only a few litre and provides all required scale up data.

A few questions:

- A. Where do you plan to run the pilot plant? City? Lab?
- B. Solids SG?
- C. Continuous operation of 200 to 400L/h or only batches per 100L? How many batches per shift?
- D. MoC or corrosiveness of slurry?
- E. Temperature of slurry?

We have available as pilot plants:

- A. TH filter press with 0.3m² filter area. One (1) chamber which is more a demo unit with the same ancillaries and process as the full scale TH filter. See picture below:

Pilot Tests for Filter Presses



* Requires 415V, 20 NI/min at 6 bar air, weight approx 1.6t, 2m x 2.4m x 2m

A TH Minerals high performance pilot scale filter press is available for on site tests:

- ▶ Single chamber
- ▶ 0.3 m² filter area
- ▶ Skid mounted
- ▶ Special features include:
 - Hydraulic drive
 - Integrated piston pump
 - 100 litre feed tank
 - Membrane squeeze system (if required)
 - PLC controlled

- B. DrM FUNDABAC filter with 0.13m² Filter area.

See picture attached.

From: Buratto, Rob <rob.buratto@hatch.com>
Sent: Tuesday, 31 March 2020 11:11
To: Goetz Bickert (GBL Process) <gb@gbprocess.com>
Subject: Pilot Plant Filtration Duty

Goetz

Hi

I trust you are keeping well and safe with the current coronavirus situation.
Here at Hatch (Perth) we are now all working from home.

We are working on a pilot plant design for a lithium ion battery materials preparation (CAM – cathode active material).

I wanted to enquire about the smallest pressure (plate and frame?) type filter units which you may have in your standard range of equipment?

The duty is to filter a precipitation process product (NiXCoyMnz.OH).

Volume of slurry = 100 litres

% solids = 8 to 12 % w/w

Mass of solids is approximately 7 to 12 dry kg.

Estimated particle size is approximately 5 to 10 micron.

Feed rate (based on precipitation discharge pump capacity) 200 to 400 litres per hour. Peristaltic type pump.

No filtration data is available, but approximating this material to something similar I estimate we would need approximately 0.6 to 1.0 m² filter area.

Do you have any standard equipment type small enough for this duty?

Or, what would you suggest is the best option for this duty.

Please let me know if you have any questions.

Regards

Rob Buratto



Appendix F Calcine Kiln Equipment

Vendor Contact Details

Batch Rotating Kiln:

Tetlow Kilns and Furnaces Pty Ltd
12 George St, Blackburn, Victoria, 3130
Phone: (03) 9877 4188
Email: info@tetlow.com.au

The vendor has been contacted to enquire about their ability to supply a kiln for the specific requirements of the pilot plant. The vendor is yet to respond.

This furnace type is not suitable for future NCM811 production as a fully sealed, complete oxygen atmosphere is required which cannot be achieved. Precise control of temperature and environment would also be unlikely/difficult, and the unit would not be suitable for NCM622 either.

Continuous Rotating Kiln:

A rotary kiln capable of accepting a continuous feed with a nominal capacity of 25 kg of material has been estimated to suit the requirements of the pilot plant. The kiln would be electrically heated and come complete with feed bin, feed screw conveyor, discharge mechanism, product cooling mechanism, product discharge bin and exhaust gas handling system. The intention would be to have the complete facility skid mounted. The feed material would be manually loaded into the kiln feed bin. A control panel, monitoring process control variables, would be provided as part of the package.

Two potential equipment suppliers for the continuous rotating kiln package to suit the pilot plant design have been contacted. These are as follows:

Calcine Kiln Vendor 1: Specialises in fabricating pilot plant facilities and package calcining equipment packages for the uranium industry.

Adelaide Control Engineering Pty Ltd
10 Peekarra St, Regency Park, South Australia, 5010
Phone: (08) 8118 6460
Email: glenn@adlcontrol.com.au

The vendor has been contacted to enquire about their ability to supply a kiln for the specific requirements of the pilot plant.

The vendor has responded with various queries concerning the kiln duty and has indicated that they are capable of supplying the equipment package.

The detailed information has not yet been received from the vendor.

Calcine Kiln Vendor 2: Specialises in fabricating rotary kiln equipment for activated carbon calcining in the gold industry, small scale rotary roasting kilns for the rare earth processing industry and small-scale pilot facility equipment.



ANSAC

Lot 2 Estuary Drive, Bunbury, Western Australia. 6230

Phone: (08) 9724 9000

Email: tim.obyrne@ansac.com.au

The vendor has been contacted to enquire about their ability to supply a kiln for the specific requirements of the pilot plant.

The vendor has responded with various queries concerning the kiln duty and has indicated that they are capable of supplying the equipment package. The detailed information has not yet been received from the vendor

Tetlow Kilns & Furnaces Pty Ltd

ABN 55 127 133 546

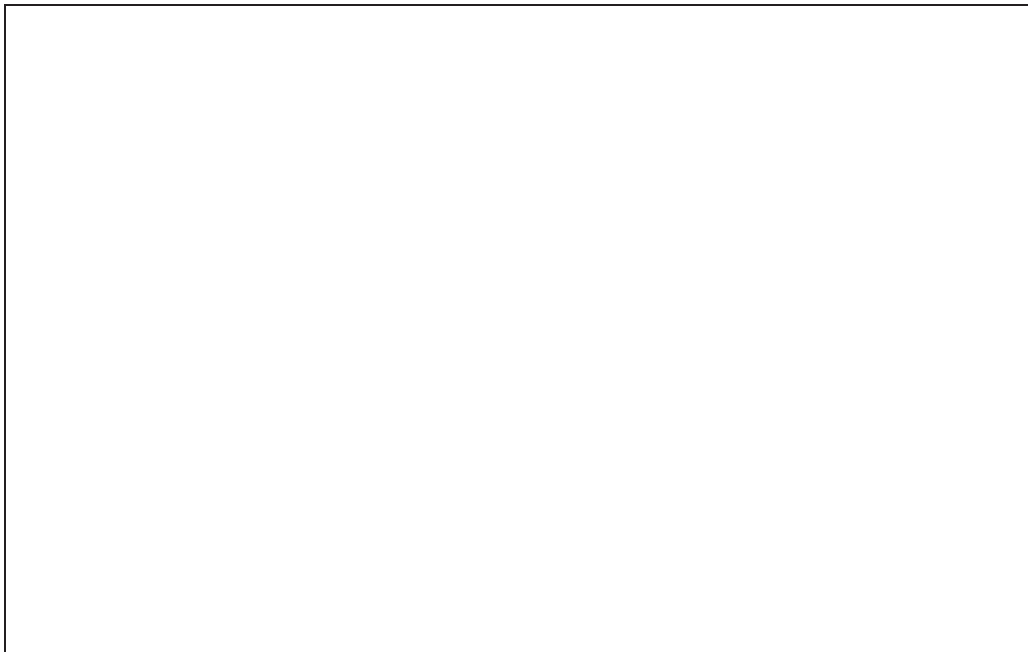
12 George Street, Blackburn 3130 Victoria Australia

Telephone: (03) 9877 4188 Fax: (03) 9894 1974

Telephone + 61 3 9877 4188 Fax: + 61 3 9894 1974

E-MAIL: info@tetlow.com.au WEBSITE:www.Tetlow.com.au

- **Furnaces**
- **Electric in air to 2000°C**
- **Vacuum**
- **Heat Treatment**
- **Controlled Atmosphere**
- **Melting**
- **Gas to 2300°C**



Quotation:

Rotary Calcining Furnace

Date: 4th October 2011

**Kilns, Furnaces & Ovens for Industry
Manufactured in Australia**

Hi Grant

1.0 **Re: Quotation for Rotary Calcining Furnace**

2.0 We have much pleasure in submitting herewith our quotation for the above mentioned, details and costs are as follows:

3.0 **Construction**

Main rotating body will be rolled from heavy section mild steel plate with rolled angle iron and RHS, stiffening rings on both ends and rolled stiffening ring in from the ends and in the middle.

4.0 **Body Support Frame**

Rolled body will be supported on a frame fabricated from heavy duty RHS. This frame will carry the support rollers, motor gear box assembly and burner.

5.0 **Main Base Frame**

The main base frame will be fabricated from heavy duty RHS. This frame will carry the pivot for the body support frame. This frame will carry the tilting rams that allows the furnace to be tilted.

6.0 **Door**

The door on the furnace will be fitted with a feed chute for loading of product. The burner would also be fitted into and connected with flexible gas and air lines. The door opening and closing will be by way of electric actuators.

7.0 **Tilting of Furnace Lip Axis Type**

The furnace will be supplied with 2 hydraulic rams mounted onto the tilting frame. The pivot will be as close as practical to the pouring position to keep the arch of the pouring position to a minimum. The hydraulic rams will be operated by a motorized power pump. Tilting by a single joystick for raising and lowering. The joy stick will be mounted at the front of the furnace in the vicinity of the pivot.

We also incorporate hose burst valves into the hose lines (they activate when instant pressure loss occurs) if a hose ruptures the valves hold the rams at the point where pressure loss occurred. The furnace will tilt forward to empty out product by 40° and tilt back by 20° for loading of product.

8.0 **Insulation**

The rotary furnace will be insulated with high density refractory such as Calderys cast M32 or similar. The refractory will be backed up by 1400° vacuum formed ceramic fibre board and microporous insulation. The refractory will be anchored back to the case with hi temperature metallic anchors. The refractory is to be heated to operating temperature at a slow rate of climb.

9.0 **Burner**

The furnace will be fitted with a single burner that fires through the door insulation into the chamber. The burner will be designed to keep the velocity to a minimum but still be effective.

10.0 **Valve Train and Flame System – Main Chamber**

A gas manifold will be mounted to the burner, and will be supplied with the appropriate flame surveillance and safety equipment, double block solenoid, full sequence flame programmer, ignition transformer, pressure switches for gas and air control, valves, regulators, modulating motor, ratio regulator and separate industrial combustion blower that is to be mounted outside the building in a weather proof noise attenuation enclosure. Connection of combustion blower is the responsibility of the purchaser. All equipment supplied and tested to AGA standards.

11.0 **Flue/Load Chute**

The furnace will be flued out through the door. The flue will also incorporate a load chute that will be fitted with an insulated door to blank off when not in use.

12.0 **Rotating System**

The furnace rotating system will be by way of a gear drive direct onto the furnace drum body. The drive will be a motor gearbox combination control with a variable speed drive with soft start. The speed will be adjustable by way of the control panel on the control cubicle. The control panel will allow for changing of RPM by way of push buttons and digital read out operating range .6 to 3 RPM.

13.0 **Over Temperature Controlling**

This is designed to prevent an inadvertent over firing. It is a complete separate circuit each with their own thermocouple, wiring and relays and has the capability of overriding all other control equipment.

14.0 **Communications**

The temperature programmer will be fitted with RS485. The RS485 from the programmer will be wired to a multi pin socket mounted on the control cubicle. A multi pin plug will be fitted to the 10 mt cable that will in turn be connected to the converter fitted with USB plug to connect to a computer.

Shimaden light software is available and can be downloaded from the internet **FREE**. This system is compatible with Windows XP. The purchaser can then develop a spreadsheet in Excel for data logging.

15.0 **Shimaden FP93 Temperature Controller/Programmer**

Shimaden FP93 96 mm² type R microprocessor based controller/programmer with display of process and set values and auto tune function of PID parameters with multi segment program firing for both heating and cooling as long as the firing times are within the parameters of the furnace and its load. The controller/programmer has 4 programs with 10 segments in its memory. All 4 programs can be linked to achieve 1 program of 40 segments.

16.0 **Specification – Rotary Calcining Furnace**

Model:	Rotary Calcining Furnace
Internal size (nominal):	700 mm ID 950 + 200 mm internal taper down to 460 mm Total length 1150 mm
Load:	50 kg with 20° tilt back
External size:	2400 mm Wide 3100 mm Deep 2500 mm High
Maximum Operating Temperature:	0 – 1300°C
Fuel:	LPG
Heating Capacity:	600 MJ
Gas Pressure required:	7 KPA
Power:	3 phase 415 volt 25 am 18 kW
Nominal operating kW per hour:	6 kW

17.0 **Price:**





18.0 **Manufacturing Time**

14 to 18 working weeks (manufacturing time) from receipt of official order and depending on prior work commitments.

Note: Closed for annual leave from 23rd December 2011 until date to be advised.

19.0

[REDACTED]

20.0 An official order made out to **TETLOW KILNS & FURNACES PTY LTD** will be required

21.0 All prices are firm for a period of ninety days only.

22.0 Goods supplied subject to GST

If you require any further information, please do not hesitate to contact the undersigned.

Yours faithfully

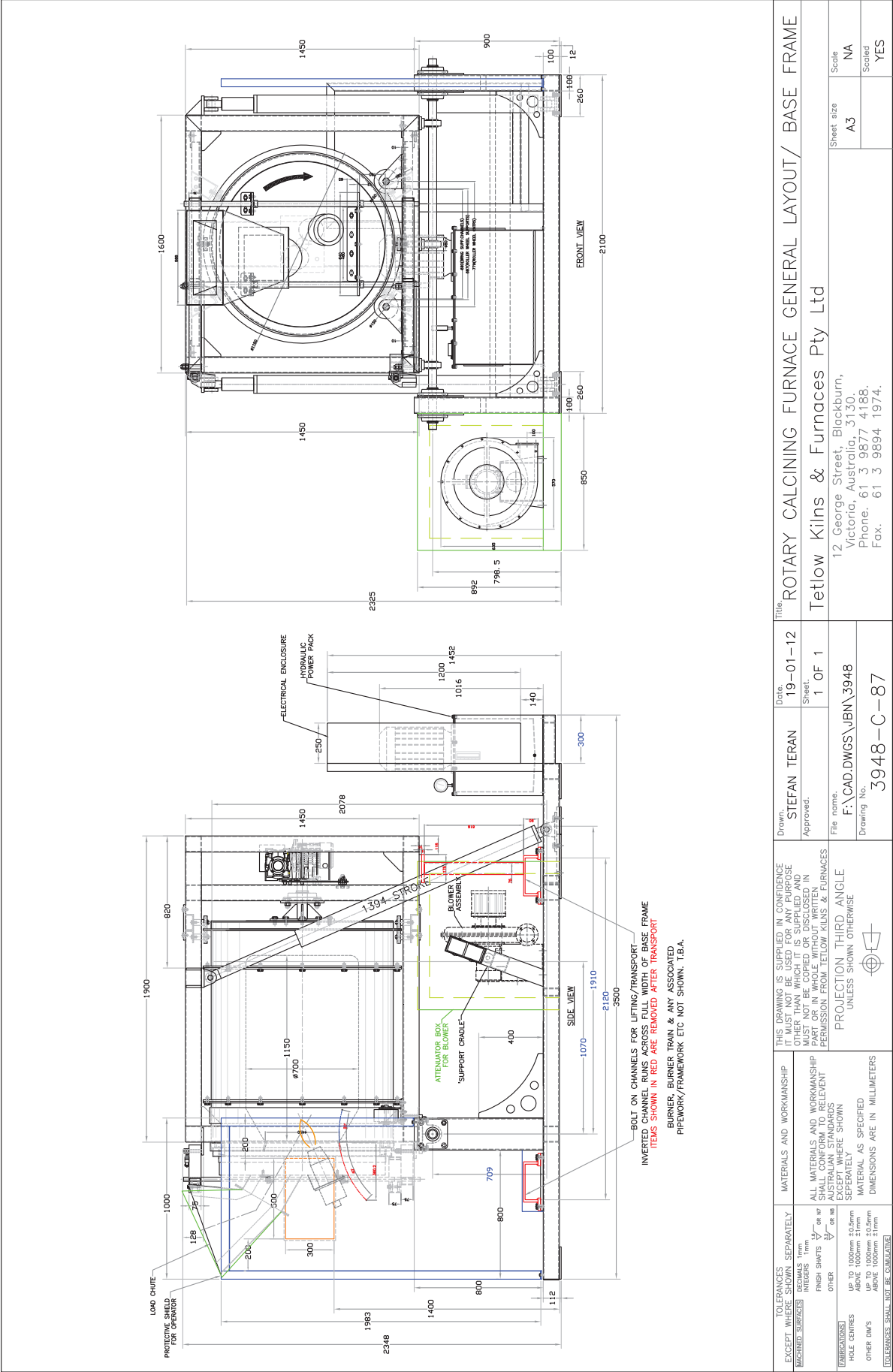
Tetlow Kilns & Furnaces Pty Ltd

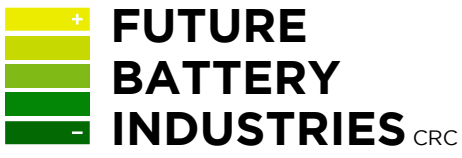
Roger I Brockway

Roger I Brockway

General Manager

Note: For more information for standard furnaces & kilns including specials manufactured to customers requirements see our website: www.tetlow.com.au





Li-ion battery cathode manufacture in Australia

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