Lithium Processing - Spodumene

APPLICATION PAPER

Pall filtration and separation technologies achieve battery grade lithium purity for EV battery production

Introduction

The Electrical Vehicle (EV) market is undergoing a revolution that is transforming the transportation landscape using Lithium-Ion battery technology. The demand for electrical vehicles is projected to increase over five times the current 2022 production values by 2030[1]. To meet this challenge, high purity Lithium Hydroxide and Lithium Carbonate are required as essential materials to formulate these batteries.

The primary sources of Lithium are either brine lakes (Salars) or mineral deposits of mostly Spodumene ore. The Spodumene ore contains up to 6 % weight Lithium and is extracted from the ground in conventional mining operations (see Figure 1) that can be either underground pit excavation or surface strip mining depending on the location of the mineral lode.

Figure 1. *Spodemene ore*

Lithium Processing

The Spodumene concentrate ore is converted into either Lithium Carbonate or Lithium Hydroxide for use as an essential component to make Lithium-Ion Batteries. The process for making Lithium Carbonate is illustrated in Figure 2. A similar flow schematic with different chemical reagents can be used to make Lithium Hydroxide.

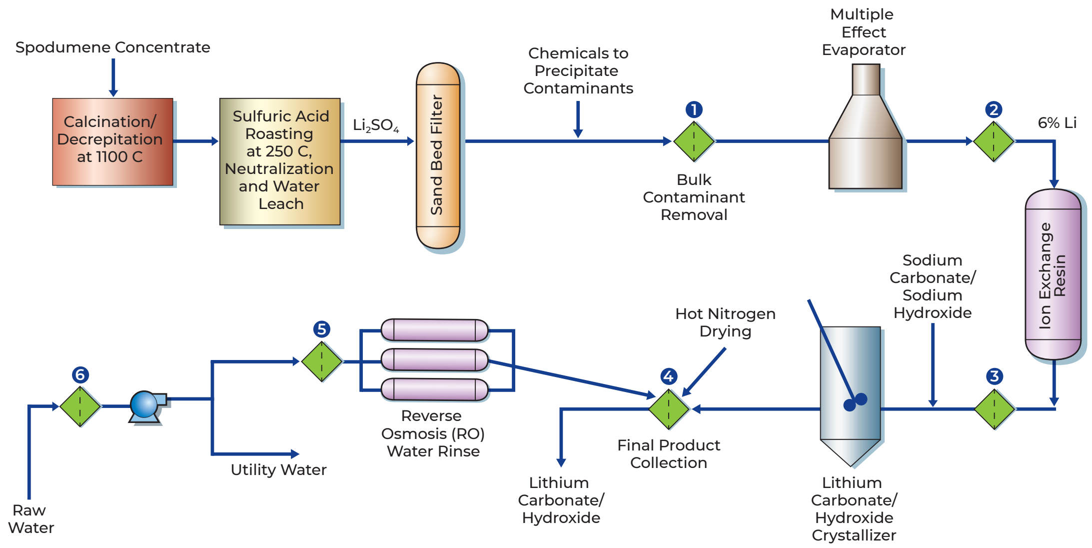
The first step is calcination / decrepitation of the spodumene ore performed at a very high temperature of 1,100 °C (2,112 °F). Here the crystal structure of the spodumene is changed to open the pore structure in the material to a format more easily extracted. The next step involves roasting with sulfuric acid at 250 °C (482 °F) followed by neutralizing and water leaching. The Lithium is now converted into a Lithium Sulfate salt solution and contains other dissolved and particulate impurities. A sand bed can be used to remove bulk particulates and the use of chemical precipitation can remove many of the undesirable dissolved contaminants.

The precipitated contaminants are separated from the Lithium Sulfate stream at location (1) typically through use of either a filter press or a centrifuge type separator. An improved method for this separation step has been developed using automated regenerable cartridge filtration. It is then required to concentrate the stream to about 6% Lithium which is achieved through use of multiple effect evaporators that are staged in series and can use both heat and vacuum to remove water. Following this step at location (2), filter cartridges rated at 1-5 micron are used to separate contaminant particulates.

An ion-Exchange resin bed is deployed to remove dissolved species and another cartridge filter rated at 1 micron is used at location (3) to prevent fine particulates from contaminating the final product. Sodium Carbonate is added to convert the Lithium Sulfate to the desired Lithium Carbonate product which is separated from the liquid stream using a crystallizer. The solid Lithium Carbonate product is collected typically using either a centrifuge or filter press at location (4).

A belt press filter is then used to further separate out and process the Lithium Carbonate particles through a rinse stage using deionized water. An improved method has been developed using automated regenerable cartridge filtration that can replace the centrifuge, filter press and belt filter. A filter cartridge rated at 10 micron is used at location (5) to protect the Reverse Osmosis Membrane System from fouling prematurely. Various process and utility water is needed throughout the plant and a filter cartridge rated at 10-20 microns is used at location (6) to ensure sufficient purity for these operations.

To achieve battery grade final Lithium products, multiple crystallization, re-dissolution and rinsing stages are often required that are not shown in this flow schematic for simplicity. Also, the process depicted is based on conventional operating plants and there are many new methods in development in this emerging industry.



Filter	Filtration Value	Separation Product
1	Bulk removal of precipitated salts - high solid concentrations	Automated regenerable cartridge
2	Protect Ion Exchange (IX) resin and crystallizer from carryover solids	1 - 5 Micron Filter
3	Remove trace solids and Ion Exchange resin fines before crystallizer	1 Micron Filter
4	Collect Lithium Carbonate product	Automated regenerable cartridge
5	Protect RO filtration unit from fouling. RO used to rinse final product	10 Micron Filters
6	Removal of contaminants from raw water for feed to RO and utility water for other uses including flushing out equipment	10 - 20 Mircon Filter

Figure 2. *Lithium Processing*

Material Purity Specifications

Lithium-Ion batteries have strict purity requirements for the materials used in their manufacture. Impurities can lead to poor charging performance including reduced vehicle range of operation, more frequent need to charge, problems with batteries starting at colder temperature and in some extreme cases to the batteries catching on fire. A major issue with the current Lithium conversion practice is reliable operation in producing the high-quality Lithium products. Battery grade purity specs are provided in Table 2 for Lithium Hydroxide and Lithium Carbonate. For Lithium Carbonate the minimum purity requirement is 99.5 wt % and for Lithium Hydroxide Monohydrate (LiOH-H2O) it is 56.5 wt% for Lithium Hydroxide (LiOH) out of a theoretical maximum purity of 57.0 wt % (due to the water monohydrate molecule).

Improved filtration and separation can play an important role in improving both the process reliability for producing consistent high purity products and also for improving the product yields, reducing product re-work, and reducing operation costs.

Grade	$LiOH\text{-}H_2O$	Li_2CO_3
Battery	56.5% LiOH	99.5%

Max Theoretical Purity of LiOH in $LiOH\text{-}H_2O$ is 57.0 wt%

Table 1. *Battery Grade Purity Specifications*[2]

Battery Grade LioH-H_2O (Lithium Hydroxide Monohydride) Purity Specs

LiOH, wt%	*56.5*	*min*
CO_2, wt%	*0.35*	*max*
Cl, wt%	*0.0020*	*max*
SO_4, wt%	*0.010*	*max*
Ca, wppm	*15*	*max*
Fe, wppm	*5*	*max*
Na, wppm	*20*	*max*
Al, wppm	*10*	*max*
Cr, wppm	*5*	*max*
Cu, wppm	*5*	*max*
K, wppm	*10*	*max*
Ni, wppm	*10*	*max*
Si, wppm	*30*	*max*
Zn, wppm	*10*	*max*
Heavy metals as Pb	*10*	*max*
Acid Insolubes, wt%	*0.010*	*max*

EV Battery Value Chain

The various stages in the Electric Battery (EV) value chain are given in Figure 3. For each segment, filtration and separation play a vital role in meeting process goals for yield, purity, and reliability. For base materials, mining and unique material processing are required for Nickel, Cobalt and Aluminum as well as Lithium as described in this paper. Active materials involve treating of chemicals, specialty chemicals and polymers to make the essential battery components consisting of the separator, electrolyte, and anode/cathode. The battery cells also use chemicals and specialty chemicals that must be at rigorous purity levels for preparing the casing, filling operations, and preparing slurries. The battery pack will have micro-electronics and automotive components and require cleanliness monitoring.

Pall Corporation is your partner for filtration and separation needs and has experience throughout the EV battery value chain. Pall has over 400 qualified Engineers and Scientists that can provide: prototype testing, on site pilot testing, best practice training, process optimization, audits, contaminant analysis, application troubleshooting, validation services, presentations at scientific forums, and journal publications.

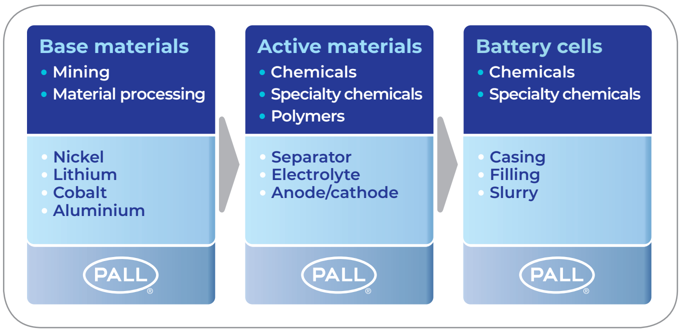


Figure 3. *Applications in the EV Battery Value Chain*

References

1. *Phillips, Piedmont Lithium Company Presentation (Nov 2020) https://www.piedmontlithium.com/*

2. *https://livent.com/wp-content/uploads/2018/09/QS-PDS-1021-r3.pdf*

Supporting the EV revolution from the mine

Mining operations typically involve the quarrying of the lithium rich ore from the surrounding rock followed by crushing & grinding operations, bulk separation by gravity difference or flotation and transport. Various mechanical operations are involved including crusher machines, mechanical shovels, large capacity haul trucks, transport belts, control systems, rotating equipment, and remote generators. Water treatment and fuel conditioning are also essential to the mining operation. Separation applications in mining are described in Table 1 for hydraulic fluids, lube oil fluids, diesel and process fluids, and water treatment.

Application	Filtration Value	Pall Product
Hydraulic	Prevent malfunction of hydraulic control systems due to contaminant fouling and fluid degradation	Particulate Filtration and Dehydration
Lube Oil	Reduce wear and reduced reliability of rotating parts due to contaminant abrasion and sluid viscosity losses	
Diesel and Process Fluids	Break emulsions and prevent damage to internal combustion engines and contamination of other process fluids	Liquid-Liquid Coalescers and High Flow Particulate Filters
Inlet Water, Recycle Process Water and Discharge (Waste) Water	Provide process water, environmental clean up and remote camp drinking water	MF System and High Flow Particulate Filters

Table 2. *Mining Separation Applications*

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