

Overview of Lithium Processing: Evaporative Brine and DLE Technology



APPLICATION PAPER

PIAPLITHBRINEN

Lithium Brine to Lithium Carbonate Process Separation Solutions

Introduction

The Electrical Vehicle (EV) market is undergoing a revolution that is transforming the transportation landscape using Lithium-Ion battery technology. The demand for lithium ion battery is projected to increase to 4900 Gwh in 2030 as compared to 1500 Gwh in 2024¹. 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 that can be either underground pit excavation or surface strip mining depending on the location of the mineral lode.

Brine Applications

Brine is pumped from Salars into surface ponds for evaporation (Figure 1). Initial brine contains Lithium Chloride ranging from 200 to 1,400 ppm. Impurities include Mg, K, SO₄, Ca and others precipitate out of solution before Lithium Chloride. A series of ponds may be used and chemicals to adjust pH and precipitate out impurities.

When the brine is concentrated to 6% Lithium, it is pumped into a processing plant and converted to Lithium Carbonate. The entire process can take up to 18 Months and can be affected by the weather and rainfall.

More than half of the lithium consumed today comes from brines that are located in South America in the Lithium triangle made up of the countries Argentina, Bolivia, and Chile. Other notable brine reserves exist in China and the Western USA.

Extraction of Lithium from brines is a less expensive method compared to processing Spodumene or other ores due to the requirements for mining, grinding, high energy costs to heat and calcinate and the use of sulfuric acid for leaching. However, it does require regional desert like conditions to allow for extended evaporation and takes a much longer period to be processed from raw brine to final product.

Besides the use of primary evaporation ponds, an adsorbent bed can be used to remove Lithium by Direct Lithium Extraction (DLE). A major drawback of this method is the need for fresh water to elute the Lithium off of the adsorbent bed after it has been collected from the brine. The feasibility of using an adsorbent bed can also be affected by the ratio of contaminants in the brine such as high levels of Magnesium compared to Lithium and how selective the adsorbent material is to Lithium.



Figure 1. Lithium Brine Salar Operations

Lithium Processing – Evaporation Ponds

The process for making Lithium Carbonate from brine using evaporation ponds is illustrated in Figure 2.

The first step is pumping the brine (200 -1,400 ppm Li) up from underground lakes called Salars into evaporation ponds. The evaporation process will usually consist of a series of ponds that allow contaminants (Calcium, Magnesium, Potassium, Sulfate, and others) to precipitate out and leave behind Lithium Chloride in solution as it become more concentrated. Ultimately when the brine reaches 6 % Lithium concentration, it is pumped into a processing plant. In some cases a specialized Boron removal unit is used that typically involves a liquid-liquid extraction process when this contaminant is present in the brine.

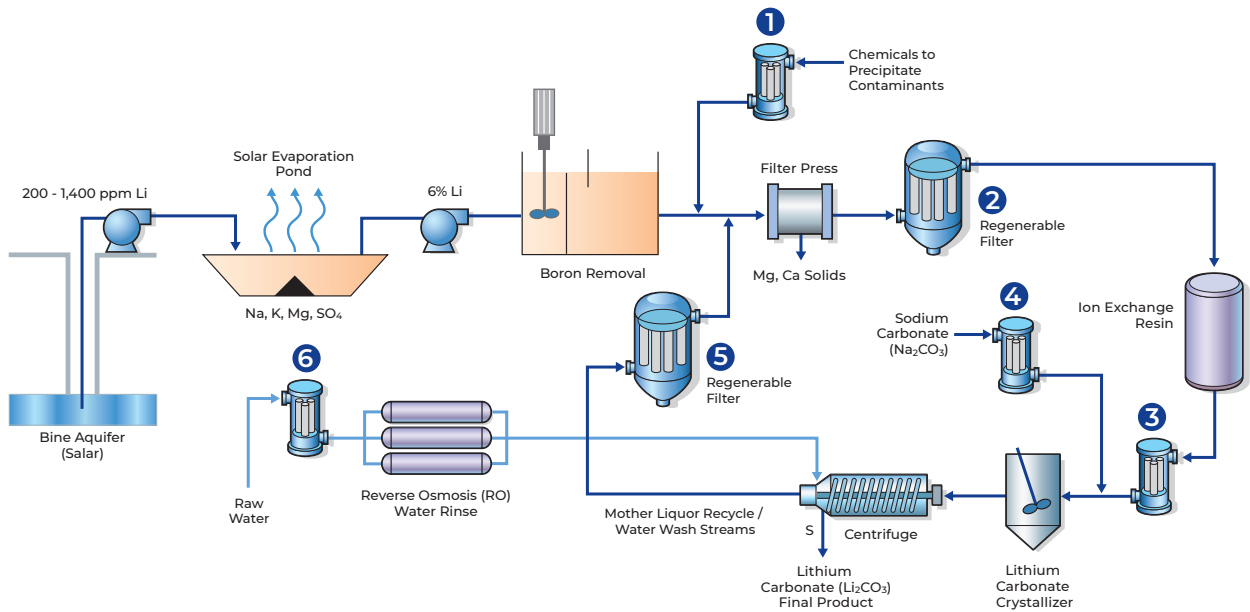
Chemicals are then added to further precipitate out contaminants that are then separated from the brine stream through use of a filter press. Cartridge filters (1-5micron) are used to purify the injected precipitation chemicals at location (1). To capture any carryover solids from the filter press, a regenerable filter (2) can be used that will protect the downstream ion exchange unit and crystallizer. The Ion-Exchange resin bed is primarily used to remove divalent dissolved contaminants remaining

such as Ca^{2+} and Mg^{2+} . Any carryover fragments of resin bed or undissolved salts are separated at location (3) using a 1 micron filter cartridge to protect the crystallizer and any adverse effect on final product purity.

Sodium Carbonate is filtered at location (4) with 1-5 micron filtration and then injected to convert the Lithium brine to the desired Lithium Carbonate product which forms into solid particles using a crystallizer. The solid Lithium Carbonate product is collected using a centrifuge. A regenerable filter at location (5) is used to capture any carryover solids from the centrifuge in the mother liquor before it is recycled and to capture any particles that are present during the water wash process. The water use for rinsing the Lithium Carbonate solids is filtered at location (6) by a 10 micron filter and then further purified using Reverse Osmosis (RO) membrane filtration. To obtain battery grade Lithium Carbonate, two stages of crystallization are normally used.

The final product Lithium Carbonate solids are dried and ground down to a specified particle size before being bagged for shipment.

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	Prevent ingress of impurities into system from injected precipitation chemicals	1 - 5 Micron Filter
2	Protect Ion Exchange (IX) and crystallizer from carryover solids coming from the filter press	Regenerable Filter
3	Remove trace solids and Ion Exchange resin fines before the crystallizer	1 Micron Filter
4	Prevent ingress of impurities into system from injected Sodium Carbonate solution	1 - 5 Micron Filter
5	Recover fine particles of Lithium Carbonate from mother liquor recycle and wash water streams	Regenerable Filter
6	Protect RO filtration unit from fouling enabling consistent supply. RO water used to rinse final product	10 Micron Filter

Figure 2: Lithium Carbonate Process Using Evaporation Ponds

Lithium Processing – DLE

Lithium brine can also be converted into Lithium Carbonate by Direct Lithium Extraction (DLE) using an adsorption bed to separate out the Lithium from the brine. A schematic for making Lithium Carbonate is illustrated in Figure 3.

The first step is pumping the brine (200 -1,400 ppm Li) up from underground lakes called Salars. The raw brine is filtered at location (1) with 20-40 micron filter cartridges to remove silt and undissolved salts before it goes to adsorption beds made up of specialized resins that are intended to adsorb the Lithium and allow the other contaminants (Calcium, Magnesium, Potassium, and others) to remain in the brine which is then sent back to the underground Salar.

When the adsorption bed becomes saturated with Lithium, it is taken offline, and Deionized Water (DI) is used to elute the Lithium from the adsorption bed (2,500 ppm Li). A filter cartridge rated at 10 micron is used at location (2) to protect the Reverse Osmosis Membrane System from fouling prematurely. The elution stream from the adsorbent bed is then treated with injected chemical precipitation chemicals. Cartridge filters rated at 1-5 micron are used to prevent any contaminant particles in these raw chemicals from entering the process at location (3).

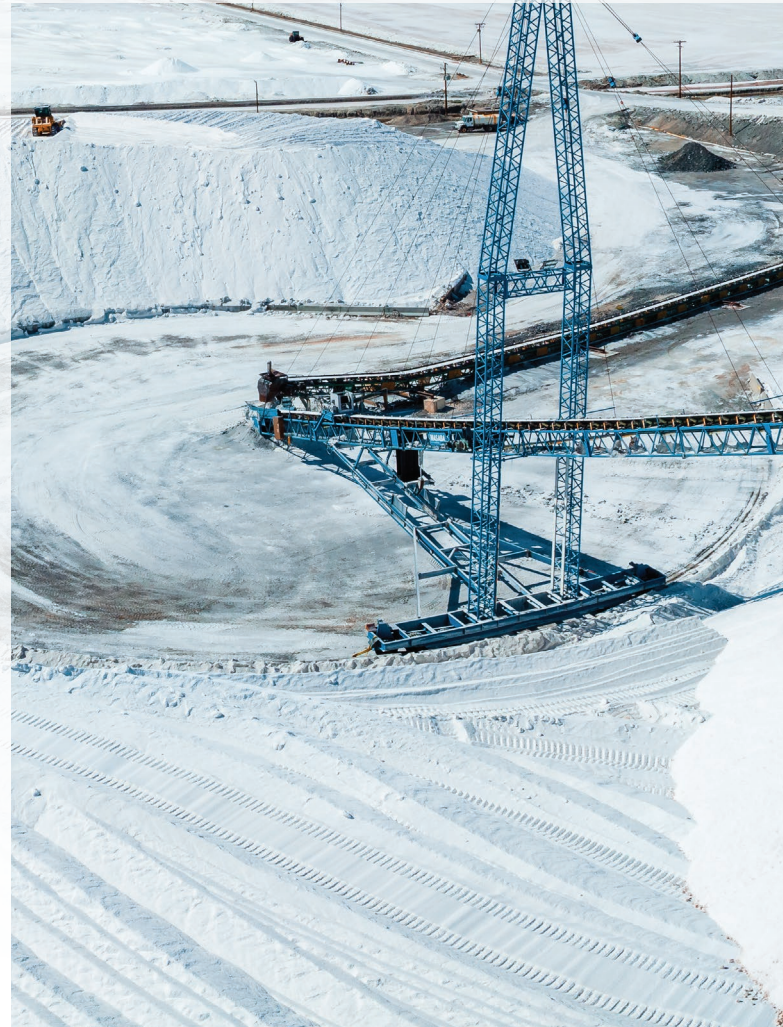
The precipitated chemical contaminants are then separated from the stream by a filter press. Any carryover of solids downstream of the filter press are collected using a regenerable filter at location (4). The process stream is then concentrated up to about 6% Lithium by use of Ultrafiltration, Nano Filtration, Reverse Osmosis (RO) and/ or a combination with a Mechanical Vapor Reactor (MVR). Following this step, any remaining fine contaminants are separated at location (5), using filter cartridges rated at 1-5 micron to protect the Ion-Exchange bed from fouling.

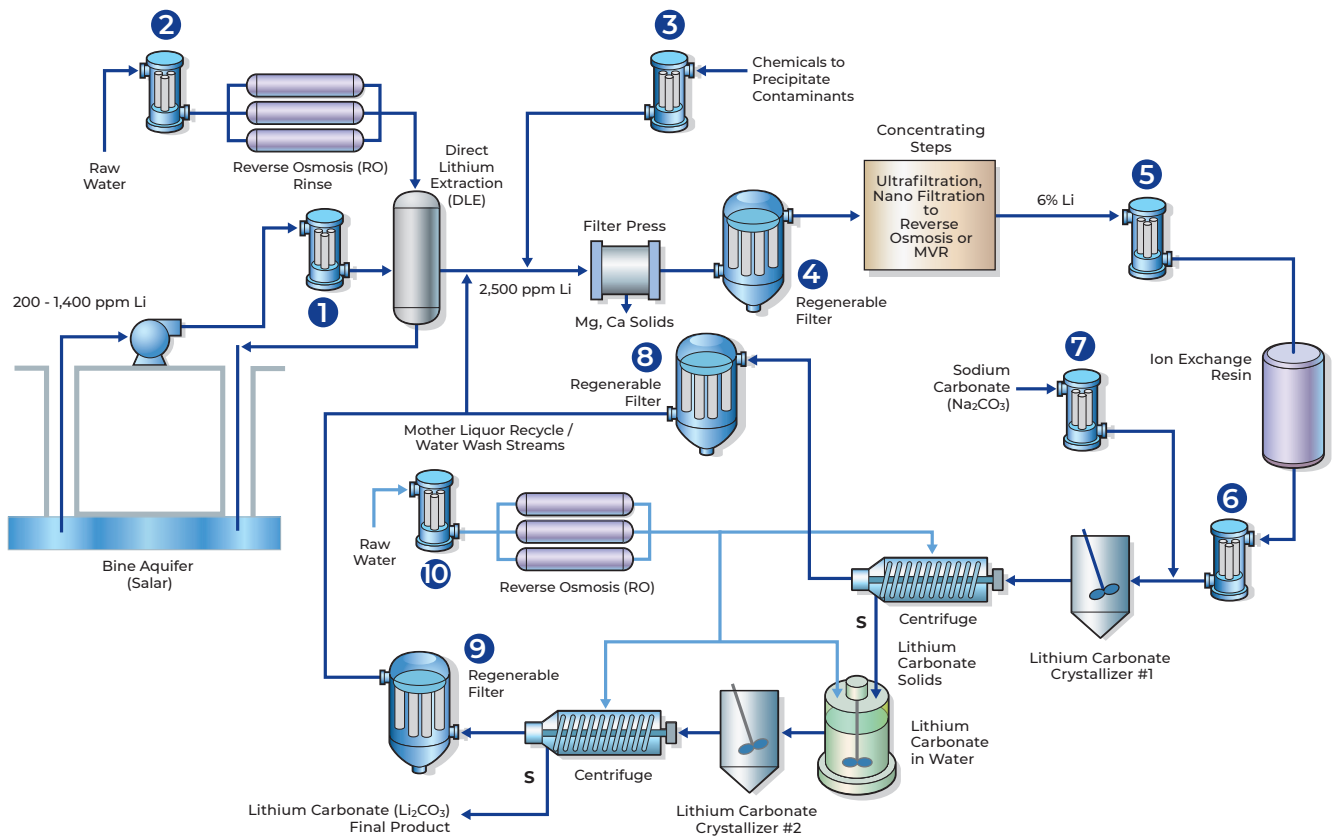
Downstream of the Ion-Exchange resin bed, a cartridge filter rated at 1 micron is used at location (6) to prevent fine particulates from contaminating the final product. Sodium Carbonate is filtered at location (7) by filter cartridges rated at 1-5 microns and added to convert the Lithium brine to the desired Lithium Carbonate product.

The Lithium Carbonate is converted to solid particles in the first stage crystallizer. The solid Lithium Carbonate solids are collected using a centrifuge. A regenerable filter (8) is used to catch any carryover solids from the centrifuge in the recycled mother liquid and during water washing stages.

The Lithium Carbonate is then redissolved in water and recrystallized to improve the purity for battery grade product. A second stage crystallizer is used to create solid particles that are collected in a second stage centrifuge. An additional regenerable filter (9) is used to catch any carryover solids from the centrifuge in the recycled mother liquid and during water washing stages. A filter cartridge rated at 10 micron is used at location (10) to protect the Reverse Osmosis Membrane System from fouling prematurely that is used for the water rinse of the Lithium Carbonate in both stages.

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	Removal of precipitated salts from raw brine – prevent adsorbent bed from contamination	20 -40 Micron Filter
2	Protect RO filtration unit from fouling enabling consistent supply. RO water used to elute captured Lithium from adsorbent bed	10 Micron Filter
3	Prevent ingress of impurities into system from injected precipitation chemicals	1 - 5 Micron Filter
4	Protect Nano-Filter / RO filter membrane systems or MVR from fouling by carryover solids coming from the filter press	Regenerable Filter
5	Protect Ion Exchange (IX) and crystallizer from carryover solids coming from the filter press	1 - 5 Micron Filter
6	Remove trace solids and Ion Exchange resin fines before the crystallizer	1 Micron Filter
7	Prevent ingress of impurities into system from injected Sodium Carbonate solution	1 - 5 Micron Filter
8 9	Recover fine particles of Lithium Carbonate from mother liquor recycle and wash water streams	Regenerable Filter
10	Protect RO filtration unit from fouling enabling consistent supply. RO water used to rinse final product	10 Micron Filter

Figure 3: Lithium Carbonate Process Using Direct Lithium Extraction (DLE)

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 1 for Lithium Carbonate. For Lithium Carbonate the minimum purity requirement is 99.5 wt. and a number of minimum levels are indicated for various contaminants.

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.

Battery Grade LiOH·H₂O (Lithium Hydroxide Monohydrate) Purity Specs

Li ₂ CO ₃ , wt. %	99.5	min
Loss @ 500° C	0.5	max
Na, wt. %	0.050	max
Ca, wt. %	0.040	max
SO ₄ , wt. %	0.1	max
Fe, wppm	5	max
Zn, wppm	5	max
Al, wppm	10	max
Cu, wppm	5	max
Ni, wppm	6	max
Cl, wt. %	0.01	max
Acid Insolubles, wt. %	0.02	max
Malvern D100, µm	40	max
Malvern D90, µm	13	max
Malvern D50, µm	4-6	max
H ₂ O*, wt. %	0.5	max

* Value (as weight percent loss) determined by drying at 500°C for 30 minutes

Table 1: Battery Grade Purity Specifications²

Material Purity Specifications

The various stages in the Electric Battery (EV) value chain are given in Figure 4. 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.

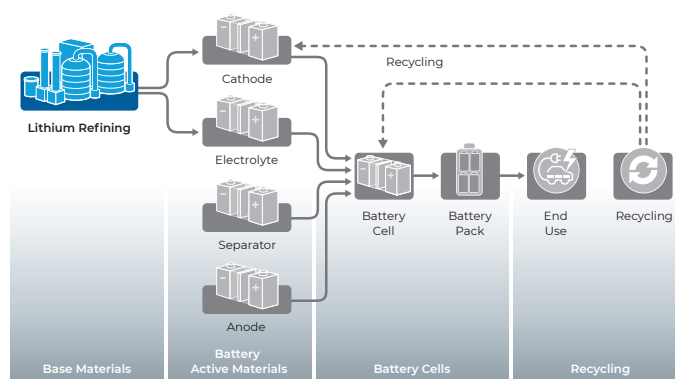


Figure 4: Applications in the EV Battery Value Chain

References

1. Battery monitor 2023 (Roland Berger & RWTH Aachen university)
2. <https://livent.com/wp-content/uploads/2022/09/QS-PDS-1060-r4-Lithium-Carbonate-Micronized-Battery-Grade-Product-Website.pdf>

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