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LITHIUM RECOVERY FROM SPODUMENE: A REVIEW OF THE SULFURIC ACID LEACHING PROCESS

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ABSTRACT

The sulfuric acid process is the primary method for lithium extraction from spodumene, but it is associated with challenges such as high-energy consumption, waste residue generation, and the need for high-temperature pretreatment. This paper provides an evaluation of the sulfuric acid process from operational, economic, and environmental perspectives, focusing on potential optimization strategies. Opportunities for improving the process include the identification of high-value elements within spodumene deposits, recycling residual lithium during mining and processing, and implementing energy-saving measures. The integration of evaporators and crystallizers is also discussed as a means of enhancing process efficiency. Finally, the paper identifies key research areas to make the sulfuric acid process more cost-effective and environmentally sustainable in response to the growing demand for lithium.

Keywords:
Sulfuric acid process, lithium extraction, spodumene, waste residue, energy optimization

INTRODUCTION

In recent years, the demand for lithium extraction has risen due to its use in electric vehicle batteries and other consumer electronic devices (Braga et al. 2019). This increased demand has also been fueled by government incentives and new fuel emissions regulations introduced by various countries to promote environmental sustainability and reduce reliance on fossil fuels (Champion 2019). Chile possesses around 48% of the world's lithium resources. In 2018, Australia was the largest producer of lithium, followed by Chile, China, and Argentina (Barrera 2019; Champion 2019). Since 2017, six new lithium mines have opened in Australia, and numerous large-scale lithium extraction projects are being developed globally (Umar 2020).

Lithium is available in various forms on the market, including lithium carbonate, lithium chloride, lithium hydroxide, and lithium metal (Champion 2019). For comparison, lithium content is often represented in lithium carbonate equivalent (LCE). Lithium is a highly reactive alkali metal and an excellent conductor of heat and electricity (Samco 2018). The applications of lithium have been extensively reviewed (Hu 2012). It is commonly found in both non-rechargeable and rechargeable batteries. Additionally, it has significant use in the production of glass and ceramics (Champion 2019), which were its primary applications until 2005 (Dessemond et al. 2019). Lithium is also used in the production of high-temperature lubricants, pharmaceuticals, and various chemicals (Champion 2019), as well as in psychopharmacological drugs for treating bipolar disorder and related conditions (El Balkhi et al. 2009; Nguyen and Bohle 2004).

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Due to its high reactivity, pure elemental lithium does not occur naturally. Instead, it is found in compounds such as salts (Samco 2018). Lithium is the lightest solid element (Dessemond et al. 2019) and the strongest reducing agent, which contributes to the high energy densities of lithium batteries (Talens Peiro, Villalba, and Ayres 2013). Table 1 shows the physicochemical properties of lithium.

Various authors have thoroughly reviewed lithium extraction and recycling methods (Cheminfo Services Inc 2012; Dessemond et al. 2019; Liu et al. 2019; Liu, Zhao, and Ghahreman 2019; Meng et al. 2019; Meshram, Pandey, and Mankhand 2014; Mishra and Majumdar 2017; Salakjani, Singh, and Nikoloski 2019b; Talens Peiro, Villalba, and Ayres 2013). Lithium is currently extracted from two primary sources: (a) 'salars' (dried salt lakes) and (b) pegmatites (mainly spodumene) (Mishra and Majumdar 2017). Presently, approximately 50% of lithium production comes from mineral ore deposits (Dessemond et al. 2019). Although the lithium concentration in brines is lower than in hard rock, extracting lithium from ore tends to be more expensive due to the energy and materials required (Terence 2020). This process can be more cost-efficient if other valuable elements are present in spodumene deposits, if lithium is recycled during mining and processing, and if energy savings are achieved during extraction.

Lithium extraction from ore is similar to other types of mining. The pollutants and effluents released depend on the type of host mineral (Cheminfo Services Inc 2012). Lithium extraction from mineral ore generates eight times more solid waste compared to lithium produced from brine (Talens Peiro, Villalba, and Ayres 2013). The use of evaporation and crystallization technologies to meet environmental standards and recover by-products is common practice in the mining industry (Condorchem Envitech 2020a).

Various methods are employed to concentrate, crystallize, and enhance the purity of lithium salts through a combination of evaporation, crystallization, and solids handling technologies. For example, evaporators are used to concentrate lithium brines, while forced circulation (FC) crystallizers are commonly applied to precipitate impurities and produce high-purity lithium salts. To ensure that the final product meets quality standards, it is essential to have a solid understanding of multi-component systems, aqueous systems, crystallization growth kinetics, and equipment requirements. As such, bench-scale and laboratory-scale testing play a crucial role in the success of these projects.

Currently, there is a lack of comprehensive technical papers examining the integration of evaporation and crystallization technologies during lithium extraction from spodumene using the traditional sulfuric acid process. This paper seeks to (a) provide a critical review of the sulfuric acid process for lithium extraction from spodumene, considering operational, economic, and environmental factors, (b) assess the potential integration of evaporation and crystallization techniques at various stages of lithium extraction and processing, and (c) highlight opportunities for further research in this area.

Lithium Physico-Chemical Characteristics

Lithium, with an atomic number of 3 and an atomic weight of 6.941, has a melting point of 180.5°C and a boiling point of 1336°C. Its density at 20°C is 0.53 g/cm³, making it one of the lightest elements. The thermal conductivity of lithium is 0.17 cal/(s·cm·°C), and its specific heat at 250°C is 0.849 cal/g. The heat of fusion for lithium is 103.2 cal/g, indicating the energy required for it to change from solid to liquid. Additionally, lithium has an electronegativity of 1.0 on the Linus Pauling scale, reflecting its tendency to attract electrons in chemical reactions. These properties make lithium a highly reactive and efficient material, particularly useful in battery technologies.

2. Lithium Extraction from Spodumene: The Traditional Process

2.1 Resources of Lithium Minerals

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Lithium is found in over 145 different minerals (Talens Peiro, Villalba, and Ayres 2013), with significant sources available in countries like Australia, Canada, and China (Champion 2019). It is primarily located in pegmatites, which are complex igneous rocks formed by the crystallization of magma deep within the Earth's crust (London 2018). There are two types of pegmatites: granitic and non-granitic, with spodumene being a granitic pegmatite mineral (Dessemond et al. 2019). While spodumene is the primary mineral used for lithium production, other minerals such as lepidolite, petalite, amblygonite, and eucryptite can also be utilized (Talens Peiro, Villalba, and Ayres 2013). Australia mainly extracts lithium from spodumene, with average grades of lithium oxide (Li_2O) ranging from 1% to 3%. Nearly all of Australia's economically viable lithium sources are found in Western Australia (Champion 2019).

2.2   Initial Concentration Process

After the spodumene ore is crushed, a flotation process is commonly applied to concentrate the mineral and produce a spodumene concentrate. This involves the use of heavy medium separation, where dense nonaqueous liquids are employed in a froth flotation process. As a result, the lithium ores are concentrated from an initial 1% to 3% Li_2O to a final concentration of 4% to 6% Li_2O (Mishra and Majumdar 2017).

2.3   Sulfuric Acid Process for Lithium Carbonate Production (The Traditional Process)

The sulfuric acid process, also known as the traditional process or acid baking (Salakjani, Singh, and Nikoloski 2019b), was first applied in a continuous plant commissioned in 2012 in China (Meshram, Pandey, and Mankhand 2014). This process begins with heating the spodumene concentrate in a rotary kiln at a temperature of 1,100°C. During this step, α-spodumene is converted to β-spodumene, which is more reactive and suitable for chemical processing (Talens Peiro, Villalba, and Ayres 2013). The initial heating step, known as decrepitation, typically uses a direct-fired kiln, where the ore is exposed to process gas in direct contact. The kiln is set at a slight angle, allowing gravity to assist in moving the material through the rotating drum.

The process continues with a cooling step at 65°C, after which the β-spodumene is ground to less than 149 μm and mixed (Talens Peiro, Villalba, and Ayres 2013). The β-spodumene is then roasted in a second kiln with an excess of concentrated H_2SO_4 at temperatures between 200-250°C (Mishra and Majumdar 2017). This roasting step lasts from 10 minutes to 1 hour, requiring at least 30-40% excess acid to ensure sufficient proton availability to react with impurities (Salakjani, Singh, and Nikoloski 2019b). The primary goal of this acid roasting is to facilitate the extraction of lithium as water-soluble lithium sulfate. This process takes place in an indirectly fired kiln, also known as a calciner, which is externally heated to prevent exposure of β-spodumene to combustion products (Ebbens and Carlson 2020). At this stage, lithium sulfate (Li_2SO_4) is produced, which is soluble in water (SGS 2013), along with an insoluble ore residue, as shown in the following reaction (Mishra and Majumdar 2017):

$$Li_2O.Al_2O_3.4SiO_2+H_2SO_4 \rightarrow H_2O.Al_2O_3.4SiO_2+Li_2SO_4$$
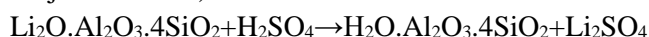
The excess sulfuric acid is neutralized with $CaCO_3$ to adjust the pH to 6-6.5. The kiln contents are then leached with water for 60 minutes (Salakjani, Singh, and Nikoloski 2019b). After filtration of the resulting slurry, a concentrated $CaSO_4$ solution, free of aluminum and iron, is obtained. Lime is added to precipitate magnesium, and soda ash is used to precipitate residual calcium. The lithium solution is then filtered, and its pH is adjusted with sulfuric acid. This solution is concentrated in a multiple-effect (MEF) evaporator (Talens Peiro, Villalba, and Ayres 2013). Achieving a high concentration of lithium in the solution is crucial for high lithium recovery rates (Zhao et al. 2019). Lithium is eventually precipitated as lithium carbonate (Li_2CO_3) by adding soda ash at temperatures between 90-100°C (Talens Peiro, Villalba, and Ayres 2013). To ensure maximum precipitation, about 5% excess soda ash is added, as demonstrated in the following reaction (SME 2019):

$Li_2SO_4 + Na_2CO_3 \rightarrow Li_2CO_3 + Na_2SO_4$
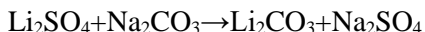
After crystallization, the resulting solution is centrifuged, and the Li_2CO_3 precipitates are washed and dried (Talens Peiro, Villalba, and Ayres 2013). Technical-grade lithium carbonate with a purity of about 95% is produced (SME 2019). Lithium extraction from spodumene is considered a mature technology.

For battery-grade lithium carbonate, which requires a purity of 99.5% and a d90 between 9 and 15 μm (d90 being the diameter at which 90% of the particles are smaller) (Peng et al. 2019), additional purification steps are needed. Various methods exist for purifying lithium carbonate (Chen, Lee, and Ho 2018). When extra purification is required, the following procedure is typically employed: (1) slurrying the impure lithium carbonate in clean water, (2) injecting carbon dioxide to reduce the pH below 8.5 and dissolve $LiHCO_3$, and (3) heating the resulting solution to precipitate pure lithium carbonate.
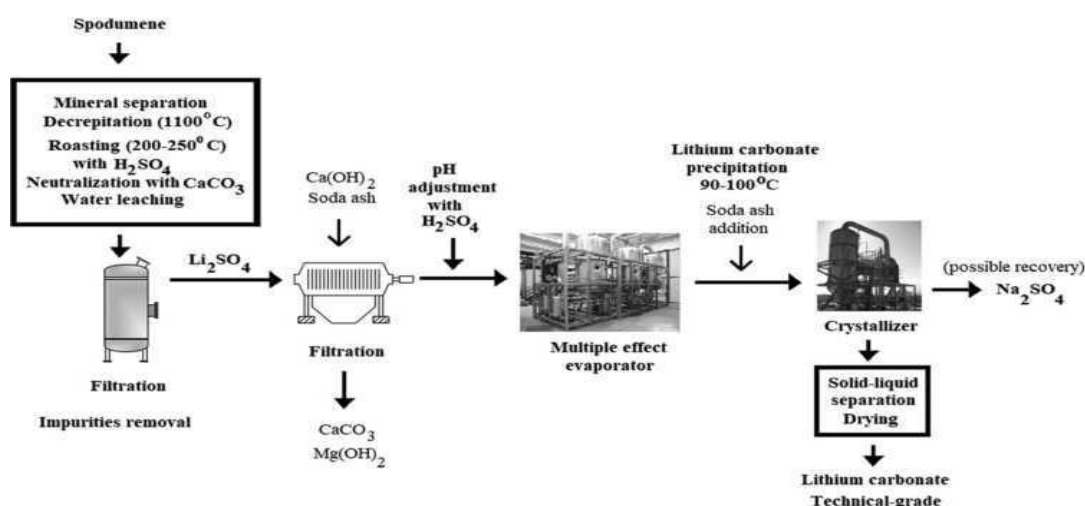

Figure 1. Technical-grade lithium carbonate production layout.

2.4   Production of different lithium salts

The methods described in the provided text refer to various chemical processes used to produce lithium compounds, particularly lithium hydroxide (LiOH) and lithium chloride (LiCl). Here's a brief explanation of each method:

2.4.1   LiOH Production by Reaction of Li_2CO_3 with Lime

This method involves reacting lithium carbonate (Li_2CO_3) with lime ($Ca(OH)_2$) to produce lithium hydroxide (LiOH). The reaction is as follows:

$Li_2CO_3 + Ca(OH)_2 \rightarrow 2LiOH + CaCO_3$
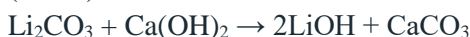
2.4.2 LiOH Production by Membrane Electrolysis (ME) of a Li2SO4 Liquor

This method involves using membrane electrolysis (ME) to produce lithium hydroxide (LiOH) from a lithium sulfate (Li_2SO_4) liquor. The reaction is as follows:

$$2Li_2SO_4 \rightarrow 2LiOH + H2SO_4 + O_2$$

2.4.3 LiOH Production by Reaction of Li_2SO_4 with Caustic Soda

This method involves reacting lithium sulfate (Li2SO4) with caustic soda (NaOH) to produce lithium hydroxide (LiOH). The reaction is as follows:

Li2SO4 + 2NaOH → 2LiOH + Na2SO4

2.4.4   LiCl Production by Reaction of Li_2CO_3 with HCl

This method involves reacting lithium carbonate (Li_2CO_3) with hydrochloric acid (HCl) to produce lithium chloride (LiCl). The reaction is as follows:

$$Li_2CO_3 + 2HCl \rightarrow 2LiCl + H_2CO_3$$

2.5   Li Metal Production

Lithium metal is produced through molten salt electrolysis, which involves using a mixture of LiCl (lithium chloride) and KCl (potassium chloride). This combination forms a molten eutectic electrolyte. The addition of potassium chloride serves two main purposes: (1) to increase the conductivity of the electrolyte and (2) to lower the melting point of the mixture, making the process more efficient. The electrolysis cell operates at temperatures between 400°C and 420°C and is equipped with a steel cathode, cast iron collectors, and a graphite anode.

During the electrolysis, chlorine gas (Cl_2) is released at the anode, while molten lithium rises to the surface, where it is collected. After the initial production, the lithium can be refined through re-melting to reduce the potassium content, achieving a purity level of less than 100 mg/L of potassium.

The electrode reactions involved in the process are:

Cathode reaction (reduction):

$$Li^+ + e^- = Li$$
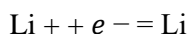
Anode reaction (oxidation):

$$Cl^- = Cl_2(\ ) + e^-$$
$$LiCl(l) = Li(l) + Cl_2\ (g)$$
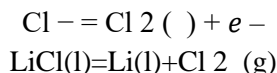
The cost of producing lithium metal from mineral ore, which typically contains 3% Li_2O, is estimated to be around $22,000 per ton. The overall process includes several stages: initial lithium carbonate production via the sulfuric acid process, the conversion of lithium carbonate to lithium chloride, and finally the electrolysis step to produce lithium metal. Further purification and metal casting may be required to obtain battery-grade lithium (Seddon, 2016).

3.   Application of evaporation and crystallization techniques during lithium extraction from spodumene by the traditional sulfuric acid process

3.1   Process applications

Evaporation and crystallization technologies are incorporated in different subprocesses during lithium extraction and processing. For instance, these technologies can be applied for (a) lithium brine concentration, (b) lithium salts crystallization, (c) lithium salts purification by re-crystallization, (d) by products recovery from lithium processing, and (e) water contaminated treatment. Table 3 provides some examples.

Table 1.   Possible applications of evaporation and crystallization technologies during lithium extraction and processing.

Sub-process	Technology
Concentration of lithium sulfate solution & crystallization of technical grade lithium carbonate	Evaporator+Crystallizer
Purification of lithium carbonate	Crystallizer

Recovery of sodium sulfate as a byproduct during lithium carbonate production	Crystallizer
Lithium chloride production	Crystallizer
Lithium hydroxide production	Crystallizer
Water contaminated (tailings) treatment	Evaporator+Crystallizer

Condensate water produced during evaporation/crystallization treatment can be recycled into the process. As shown in Section 2.3, 24 tons of water are spent to produce one ton of lithium carbonate from spodumene.

3.2 Evaporation Technology

3.2.1 Evaporation Concepts

Evaporation occurs when a liquid's vapor pressure exceeds atmospheric pressure. Liquids with high vapor pressure evaporate easily, while those with low vapor pressure do so more slowly. Boiling takes place when the liquid is heated to a temperature at which its vapor pressure equals atmospheric pressure, leading to rapid evaporation. Conversely, if external pressure is reduced (as in a vacuum), liquids like water can boil at lower temperatures. This principle is applied in processes such as lithium extraction, where evaporation is used to concentrate solutions. By lowering the pressure in the evaporation chamber, the boiling point and energy consumption are reduced. As salinity increases, the boiling point rises due to a decrease in vapor pressure, making evaporation less efficient. To maintain effective performance, regular chemical treatment is necessary to clean the heat transfer surfaces.

3.2.2 Types of Evaporators

In the mining industry, **Multiple Effect (MEF)** and **Mechanical Vapor Recompression (MVR)** evaporators are commonly used.

- **MEF Evaporators**: These systems are powered by hot water, steam, or diathermic oil. One key advantage is the ability to use residual heat streams. MEF units can operate with one or more stages, with heat transferred from the evaporated product being recycled in the subsequent stages. The first-stage condensate is typically the purest. MEF configurations include external heat exchangers and submerged tube exchangers. The number of effects is determined by the trade-off between operating costs and capital investment, as well as the evaporation capacity and boiling point elevation of the solution.

- **MVR Evaporators**: MVR systems are known for their low energy consumption. They work by preheating the brine using vapor or electrical resistors before compressing the evaporated vapor with a root pump, raising its temperature and pressure. This vapor is then used to heat the raw brine again. MVR evaporators come in various configurations, such as **Forced Circulation (FC)** and **Falling Film (FF)** types:
  - **FC Evaporators**: These use forced water circulation to optimize heat exchange and energy efficiency. Operating temperatures typically range around 60°C, with a vacuum maintained at approximately 200 mbar.

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- o **FF Evaporators**: In this design, brine is recirculated and flows down the tube walls, evaporating as it does so. The compressed vapor condenses on the outside of the tubes, transferring heat to the brine inside. The operating temperature is around 90°C, with a vacuum maintained at about 700 mbar.

Energy consumption in FC evaporators is approximately 50 kWh/m³ of distillate, while in FF evaporators, it can range from 36 to 100 kWh/m³.

The selection between MEF and MVR depends on factors such as the cost of natural gas, available power, and the presence of residual heat streams on-site.

3.3 Crystallization Process

3.3.1 Crystallization Concepts

Crystallization requires supersaturation, a state in which the solute concentration exceeds the normal solubility. Supersaturation can be achieved through evaporation, cooling, or chemical precipitation. The process begins with an "induction period" before crystals start forming, influenced by factors like supersaturation level, agitation, presence of impurities, and viscosity.

For salts with solubility that is largely independent of temperature, **evaporative crystallization** is recommended. However, if solubility strongly depends on temperature, **vacuum cooling crystallization** might be more suitable. In vacuum cooling, cooling surfaces are not needed, and incrustation (build-up of solid material) is avoided. The crystallization method chosen depends on the desired final product specifications, such as purity, particle size, and processing time.

Crystallization involves two main stages: nucleation (the formation of new crystals) and crystal growth. Nucleation can be primary (without prior crystals) or secondary (when crystals are already present in the solution). Secondary nucleation is more common in industrial processes, as it is induced by the presence of impurities or by the physical interaction between crystals and surfaces like other crystals or equipment. Crystal-agitator contacts are the most effective for generating secondary nuclei.

This detailed description of crystallization and evaporation technologies is crucial for understanding how these processes can be optimized for industrial applications such as lithium extraction, where both concentration and crystallization of solutions are essential.
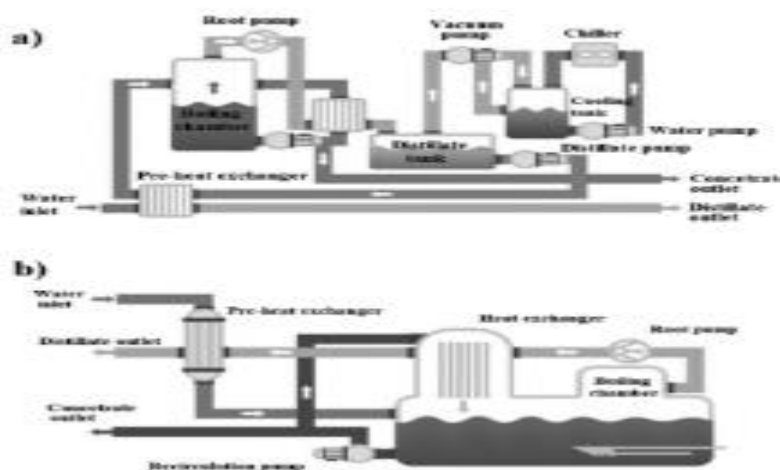

Figure 2. MVR evaporator lay-out: a) Forced circulation and b) Falling film (Condorchem Envitech 2020b).

Secondary nucleation (Bo) is influenced by factors such as dissipated mixing energy (ε), the degree of supersaturation (ΔC), and suspension density (m), with the nucleation rate being more sensitive to changes in

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ΔC. The equation $B_0 = kN(\epsilon r \cdot m_i)^T \Delta C^n$ $B_0 = kN(r \cdot m_i \epsilon)^T \Delta C^n$ expresses how these factors contribute to nucleation, highlighting that controlling mixing energy, suspension density, and supersaturation can effectively manage secondary nucleation. The crystal growth rate, which determines how fast crystals grow, is also affected by ΔC, though it is less sensitive to it than nucleation. To form larger crystals, it is essential to limit ΔC to avoid surpassing the metastable region, set ΔC at a point where the growth rate is maximized, and optimize mixing energy to minimize secondary nucleation. Impurities play a significant role by altering crystal growth, morphology, and nucleation, often promoting secondary nucleation and leading to smaller crystals or altered crystal shapes. Therefore, careful control of supersaturation, mixing energy, and impurities is crucial to optimizing crystallization processes and achieving the desired crystal size, purity, and morphology.

3.3.2 Types of Crystallizers

Forced Circulation (FC) crystallizers are highly effective for applications requiring high evaporation rates and are particularly suitable for salts that crystallize readily, such as lithium carbonate, lithium hydroxide, and sodium sulfate. These crystallizers consist of several key components: an evaporation (boiling) chamber, a recirculation pump that provides mixing energy, and an external heat exchanger. The recirculation pump, often a centrifugal type, is used to circulate the slurry within the system. This circulation must be carefully controlled to maintain the crystals in suspension, ensuring that the crystallization process proceeds efficiently. By regulating the circulation rate, FC crystallizers can effectively manage the conditions necessary for crystal growth and prevent unwanted issues such as settling of the crystals.
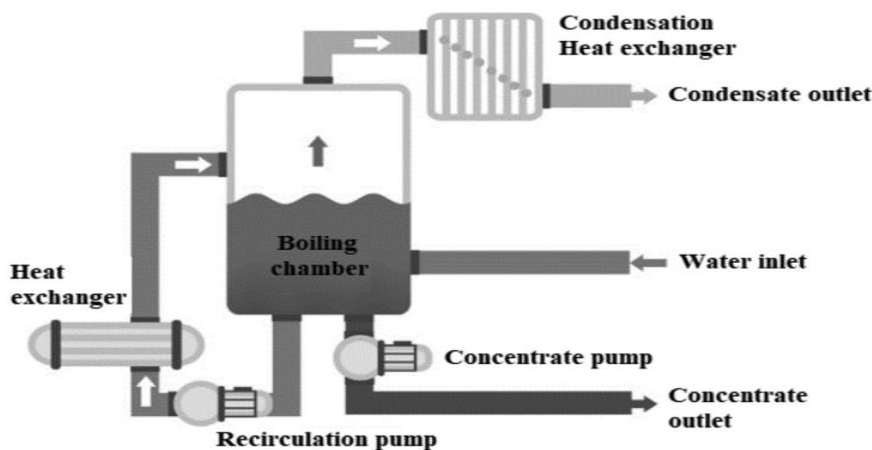

Figure 3. FC crystallizer (Condorchem Envitech 2020b).

FC crystallizers can operate: (a) under vacuum or (b) at low super-atmospheric pressure (Gea 2012). A particle size lower that 0.5 mm is achievable using FC crystallizers (Mullin 2001). During the crystallization step, the crystals must be separated from the mother liquor. This can be achieved with a pusher centrifuge, if the crystal size is big enough, following pre-concentration in a thickener. Different brands specialize in providing centrifuges for this purpose (Andritz 2020; Riera Nadeu 2018). Other types of crystallizers available in the market are: (a) draft tube baffled crystallizers, (b) Oslo crystallizers, (c) vacuum crystallizers, (d) reactive crystallizers, (e) adiabatic flash crystallizers and (f) calandria crystallizers. Draft tube baffled crystallizers and Oslo crystallizers can produce crystals of higher particle size than FC crystallizers.

3.4 Waste Energy Recovery

The traditional lithium extraction process from spodumene is energy-intensive, but waste heat recovery offers a potential solution to reduce costs. A comprehensive energy-mass balance is essential to model on-site energy use. One option is to recover waste heat from the high-temperature decrepitation process (1100°C) by passing the kiln's waste gases through boilers to generate steam. This steam can then drive a turbine-generator, which powers downstream units like evaporators and crystallizers. Although initial investment costs may be high, calculating the payback period is necessary for evaluating economic feasibility. Alternatively, steam or hot water can be utilized as a power source in various evaporators, such as MEF (Multiple Effect) and MVR (Mechanical Vapor Recompression) systems, to facilitate the evaporation process. In these systems, steam helps generate water vapor, which is used in successive evaporation stages, and is also employed in pre-heating in MVR evaporators. FC crystallizers, similar to other technologies, can also be powered by hot water or steam, optimizing energy use across the extraction process.

3.5 Opportunities for Future Research

As lithium demand rises, there is an increasing push for developing new extraction methods and improving existing ones. Research could explore lithium recovery and recycling during mining, where residual lithium carbonate (Li2CO3) in washing waters could be recycled, potentially reaching concentrations up to 1,500 mg/L. One approach might involve returning these residuals to the water leach step after impurity removal. Additionally, there is potential for using a combination of membrane and evaporation technologies to minimize or concentrate tailings, which represent a significant environmental challenge. Evaporative crystallization could offer an advantage over traditional membrane filtration by minimizing saline wastewater volume to zero and enabling the recovery of solid residues such as pure salts. Research into the integration of membrane distillation (MD) with crystallization has also been proposed as a promising alternative for lithium recovery. Despite high capital and operational costs, MD technology could enhance the efficiency of lithium extraction, particularly when coupled with traditional processes. The utilization of residual heat on-site, such as waste heat from decrepitation, could also improve energy efficiency, especially in arid regions where water reuse is crucial. Further research into solar energy use could also help cut costs and reduce emissions in the extraction process. Overall, achieving Zero Liquid Discharge (ZLD) and improving the integration of new technologies like MD with traditional extraction methods are key areas for future development.

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