



DIRECT EXTRACTION OF LITHIUM FROM A-SPODUMENE



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PARTNERSHIP HIGHLIGHTS



Patent-Pending process for Direct Extraction of lithium from a-spodumene



PCT/US2021/018727 applications filed by Penn State in 2021



Efficient process design allowing for reduced energy consumption, significantly lower CO2 emissions, reduced chemical costs and fewer facility components



Strategic partnership with Penn State University

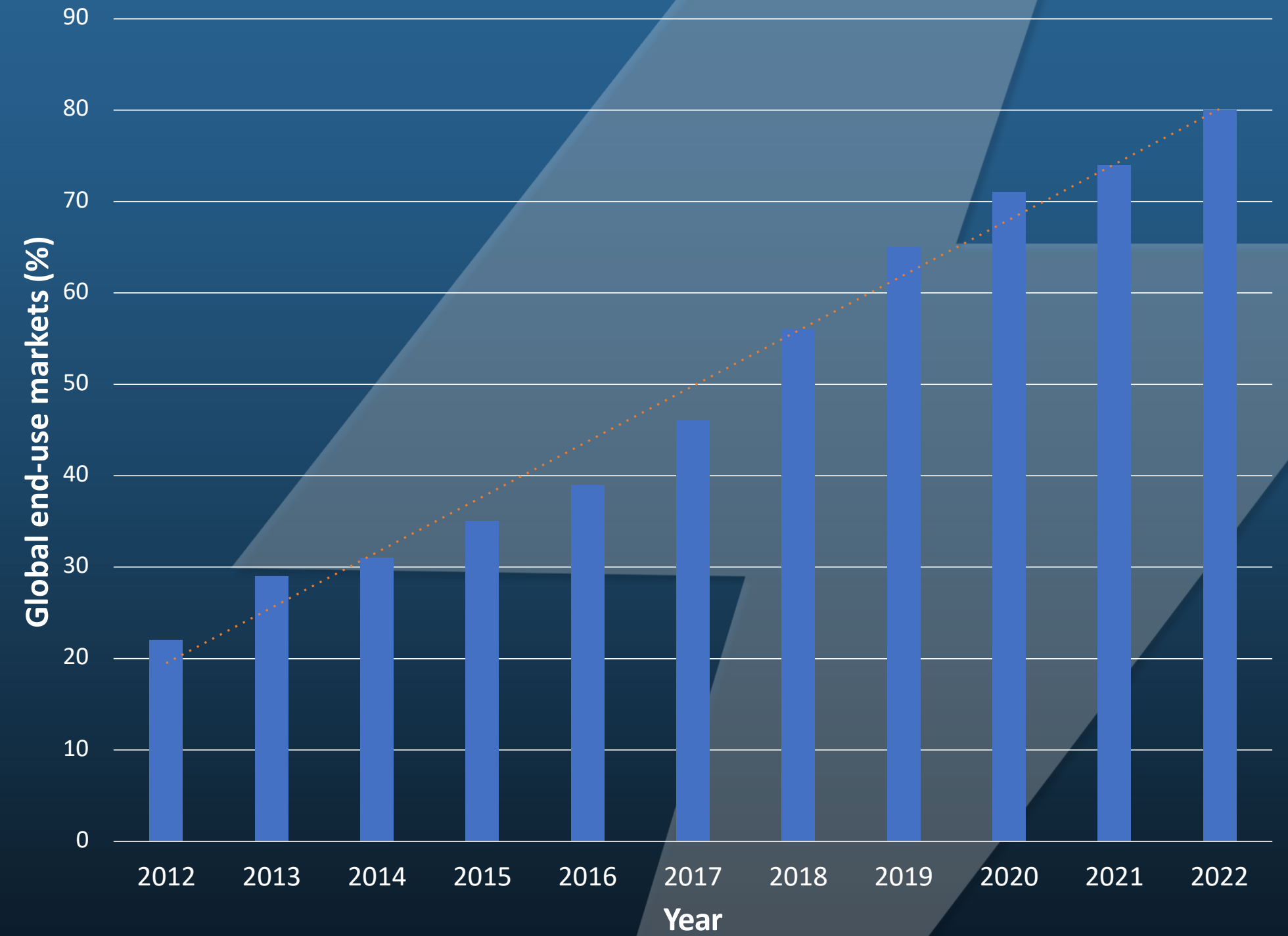
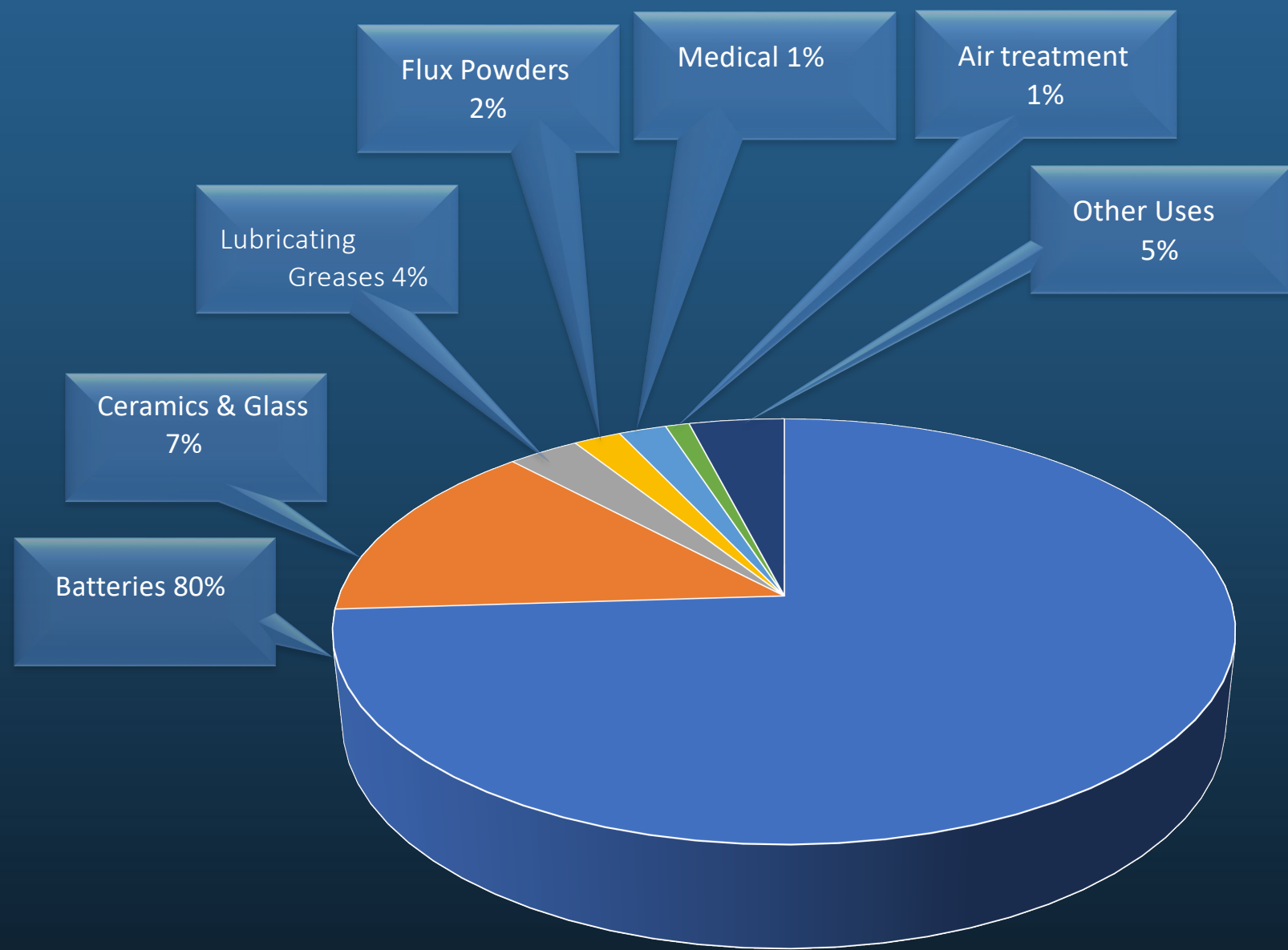


Technology ready for pilot-scale testing

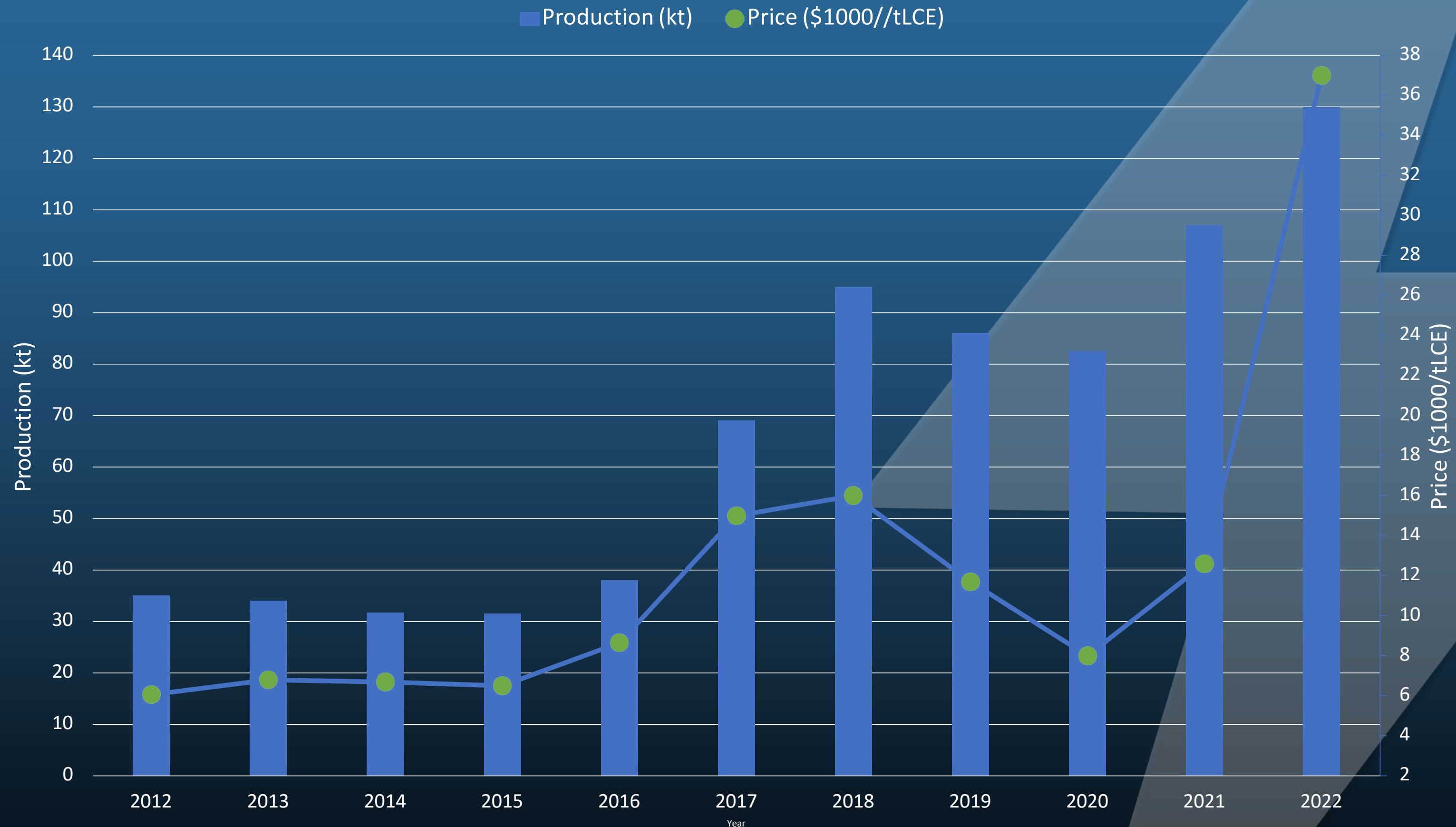


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LITHIUM APPLICATIONS



LITHIUM PRODUCTION



LITHIUM SOURCES

Brines: ~50% (ABC triangle, US, China)

- Using solar energy

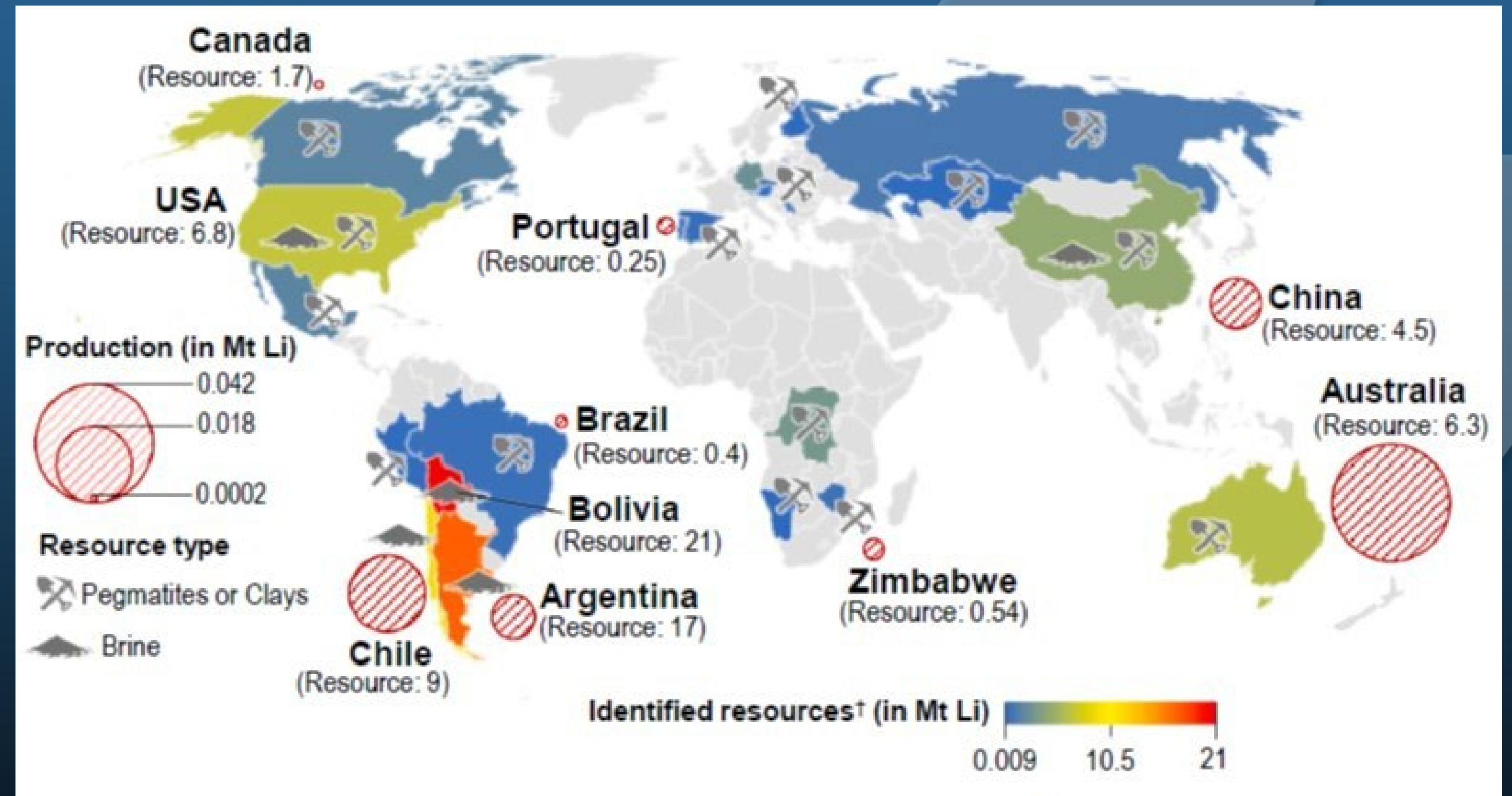
- 12-18 months

Minerals: ~40%

- Distributed around the world
- Higher concentration
- More reliable
- Faster to production

Others: ~10%

- Clays
- Secondary resources



CURRENT INDUSTRIAL PROCESS

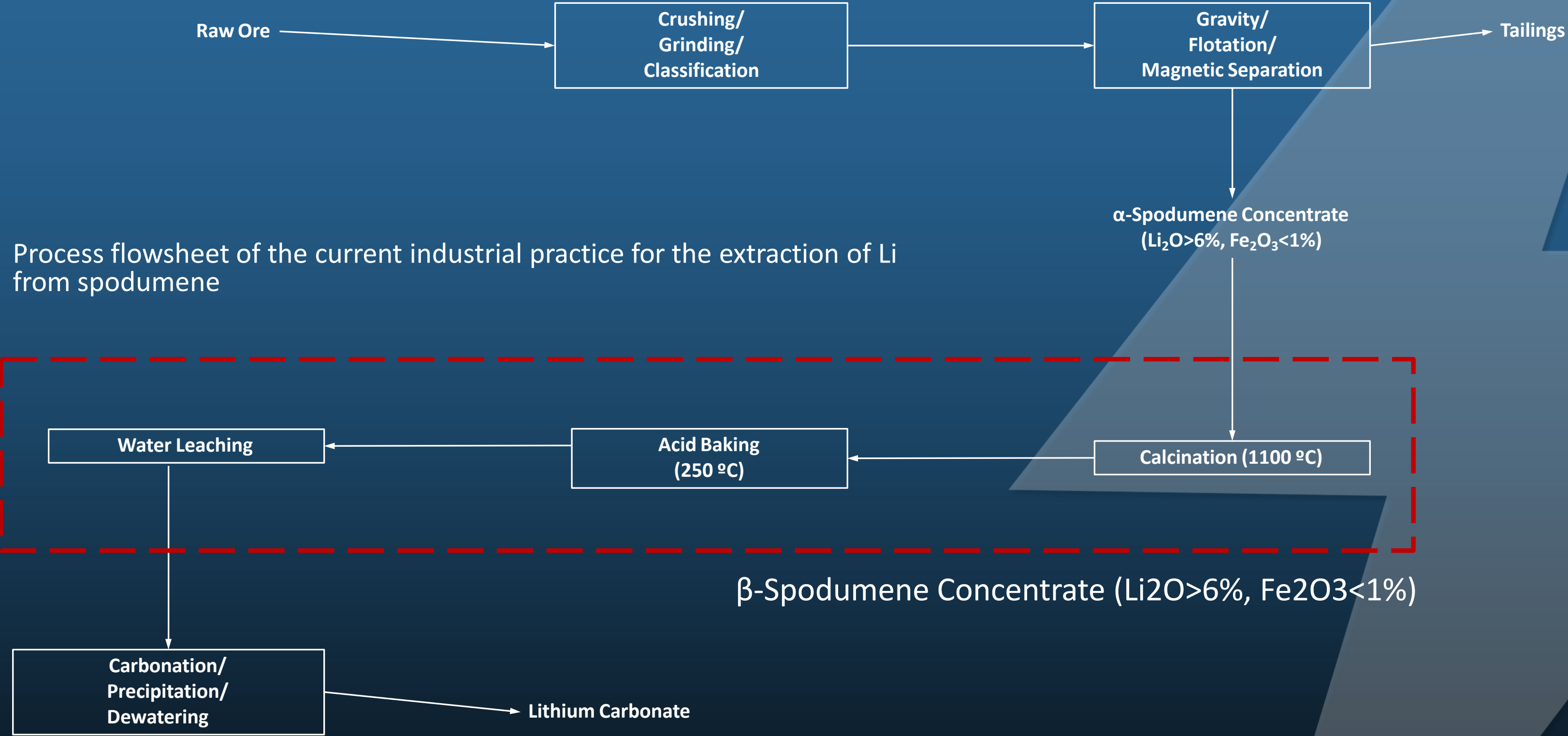
- The spodumene concentrate, which typically has about 6% Lithium Oxide (Li_2O), is obtained through physical separation.
- Spodumene mineral naturally occurs in the α phase, a monoclinic compact crystal structure with low reactivity, and thus cannot be easily leached.
- To extract Lithium (Li) from spodumene through conventional methods, the concentrate goes through high-temperature calcination at $1100\text{ }^\circ\text{C}$ for 2 hours, during which α -spodumene is converted to the relatively porous, more reactive and leachable β -spodumene.
- This product then undergoes the sulfuric acid baking process at $250\text{ }^\circ\text{C}$, producing Lithium Sulfate (Li_2SO_4) through ion exchange between H^+ and Li^+ , which can be readily dissolved in water.
- The pregnant leach solution obtained from the water leaching then goes through a purification process to produce the final product such as lithium carbonate (Li_2CO_3).

DRAWBACKS

- High cost required for the energy- extensive high-temperature calcination process for the phase transformation of α - to β - spodumene.
- Significant environmental impacts as it contributes to the high greenhouse gas (GHG) emission of Li extraction from spodumene (i.e., 9 tonnes of CO₂ per tonne of lithium carbonate equivalent (LCE) produced, nearly triple that from the brine sector).
- Concentrated sulfuric acid baking of β -spodumene in this method requires significant equipment investment and results in acid gas emissions.
- Temperature control and energy recovery are also challenging when a rotary kiln is used for this purpose. Furthermore, the large amount of precipitated Iron (Fe) and Aluminum (Al) impurities causes Li losses.
- Purification of Li from the acidic leachate requires significant chemicals to adjust pH.

LITHIUM EXTRACTION FROM SPODUMENE

Spodumene $\text{LiAlSi}_2\text{O}_6$: 8% Li_2O



Adopted from Critical Elements Corporation 43-101 Report (2011)

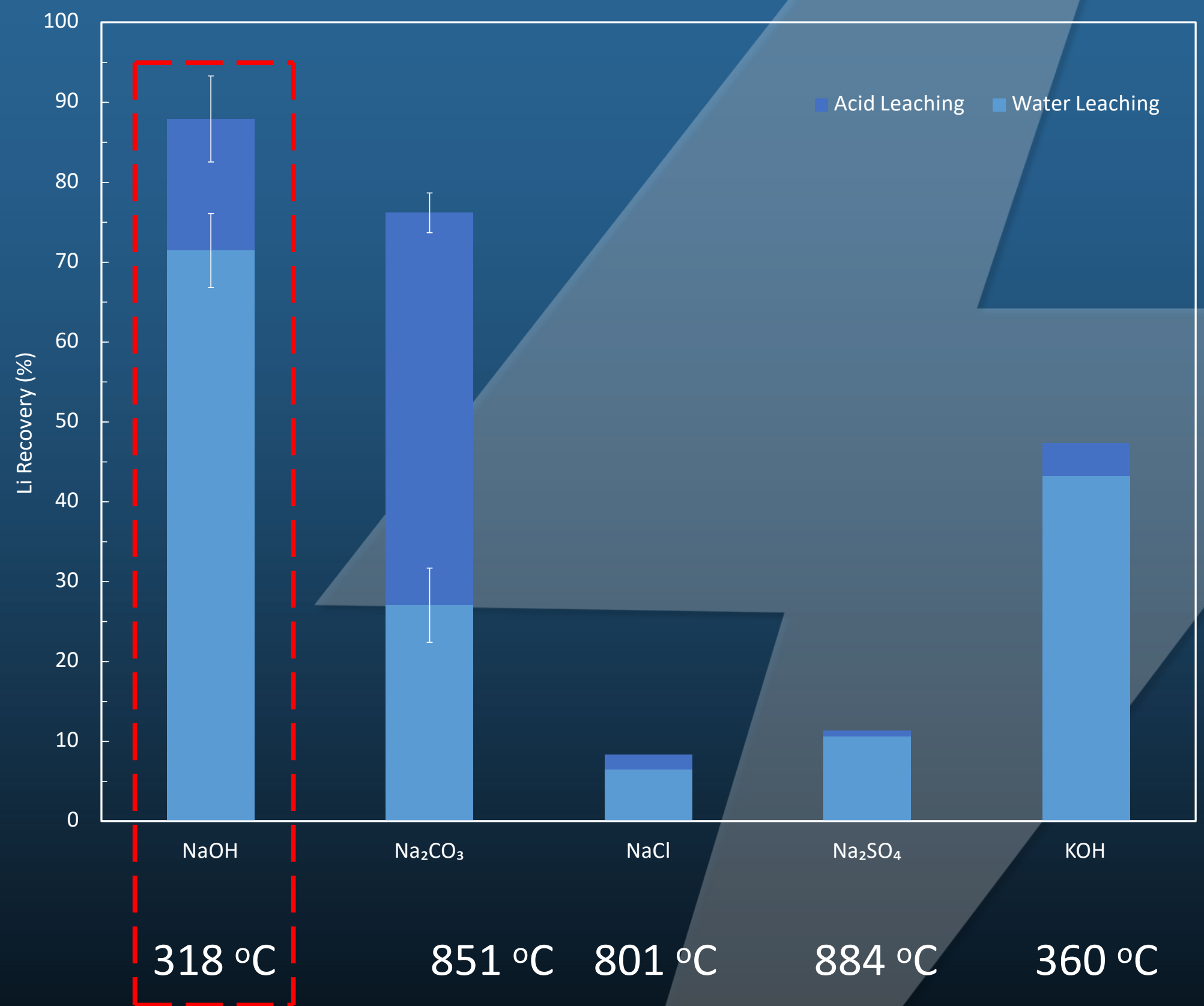
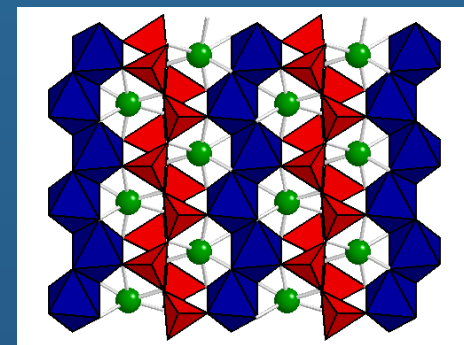
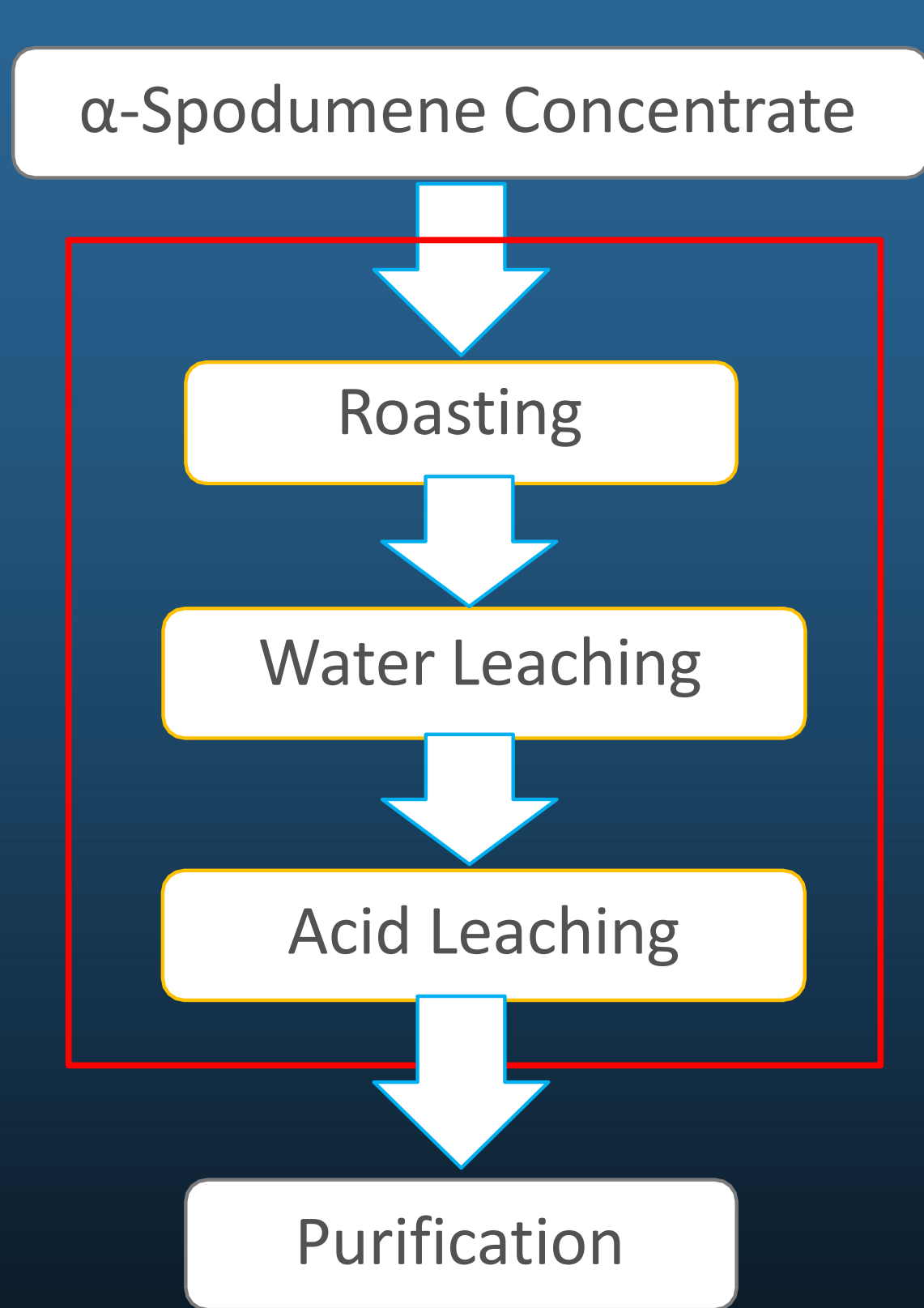
PATENT-PENDING DISCOVERY

CHEMICAL ROASTING

- To eliminate the need for high-temperature calcination and acid-baking processes, Penn State research focused on direct extraction of Li from α -spodumene.
- Research was focused to transfer the structure of α -spodumene to water or acid soluble phases, that can be recovered by water or acid leaching.
- Various industrial roasting agent were examined for this phase transformation. The chemicals were mixed with α -spodumene and the mixture was heated at the melting point of each chemical and was then subjected to a sequential water and acid leaching process.

*For further information regarding the scientific process please see the appendix

SALT ROASTING AND LEACHING – TESTING PHASE



SALT ROASTING AND LEACHING OUTCOME

- Sodium Hydroxide (NaOH) was found the most effective reagent for the phase transformation of α -spodumene to water-soluble phases.
- 71% and 88% of water leaching, and total leaching recovery values, correspondingly, were achieved by NaOH roasting, water leaching, acid leaching, under the non-optimized conditions of roasting temperature of 320 °C, NaOH : spodumene ratio of 1.5 : 1, and roasting time of 2hr.
- The majority of Li was recovered in water leaching, while keeping Al in solid residue. Al can be further recovered through acid leaching as a by product.
- The optimization study is undergoing to maximize Li recovery (>90%) through NaOH roasting using conventional heating followed by water leaching, thereby removing the acid leaching process.
- Thermodynamics analysis by FactSage demonstrated that α -spodumene was decomposed to Na_2SiO_3 , Na_4SiO_4 , NaAlO_2 , and $\text{Li}_3\text{NaSiO}_4$ by NaOH.

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(54) Title: PROCESS FOR EXTRACTION OF LITHIUM

(57) Abstract: Disclosed herein are methods for the recovery of lithium from lithium-bearing materials. More specifically, disclosed herein are methods comprising heating the lithium-bearing material with a solid roasting agent, forming a water suspension to allow to leach at least a portion of lithium into the water, separating a liquid and solid phase, and then exposing the collected solid phase to acid to allow acid leaching of the remaining amount of lithium.

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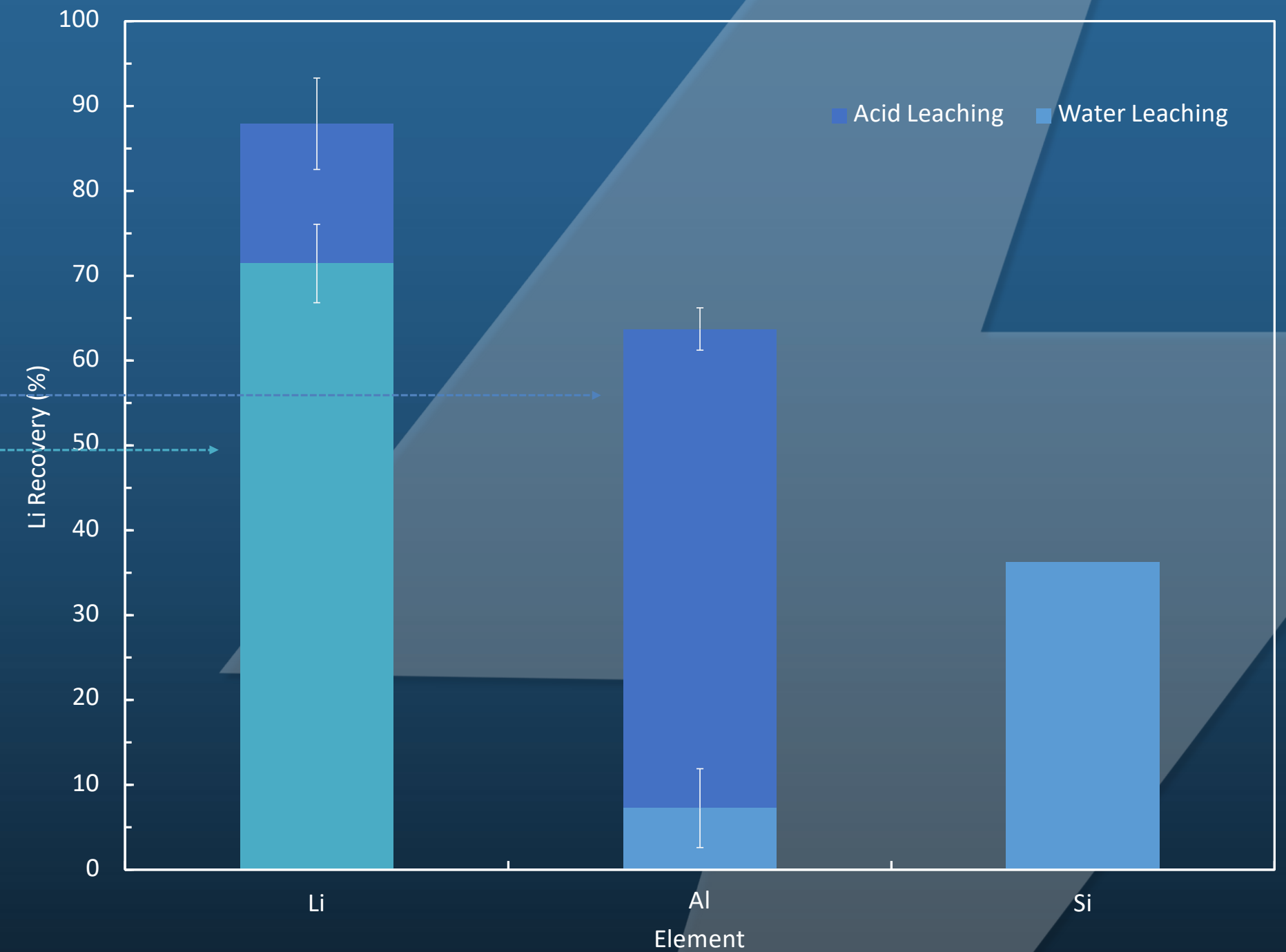
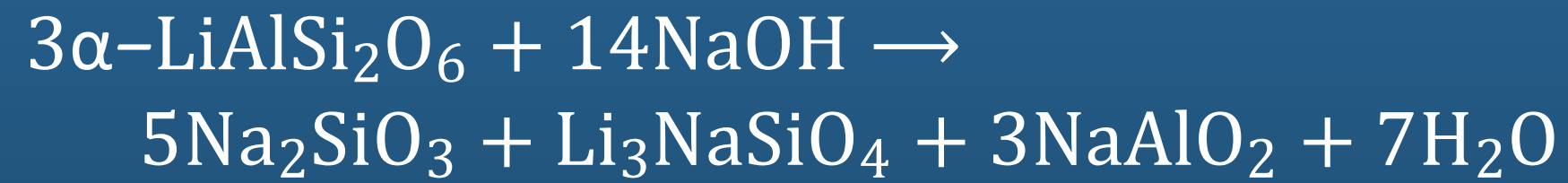
Research Article

Direct Extraction of Lithium from α -Spodumene by Salt Roasting–Leaching Process

Shihua Han, Daulet Sagzhanov, Jinhe Pan, Behzad Vaziri Hassas, Mohammad Rezaee,* Hamid Akbari, and Robert Mensah-Biney

ELEMENTAL RECOVERY BY NaOH ROASTING

- Equilibria calculated by FactSage

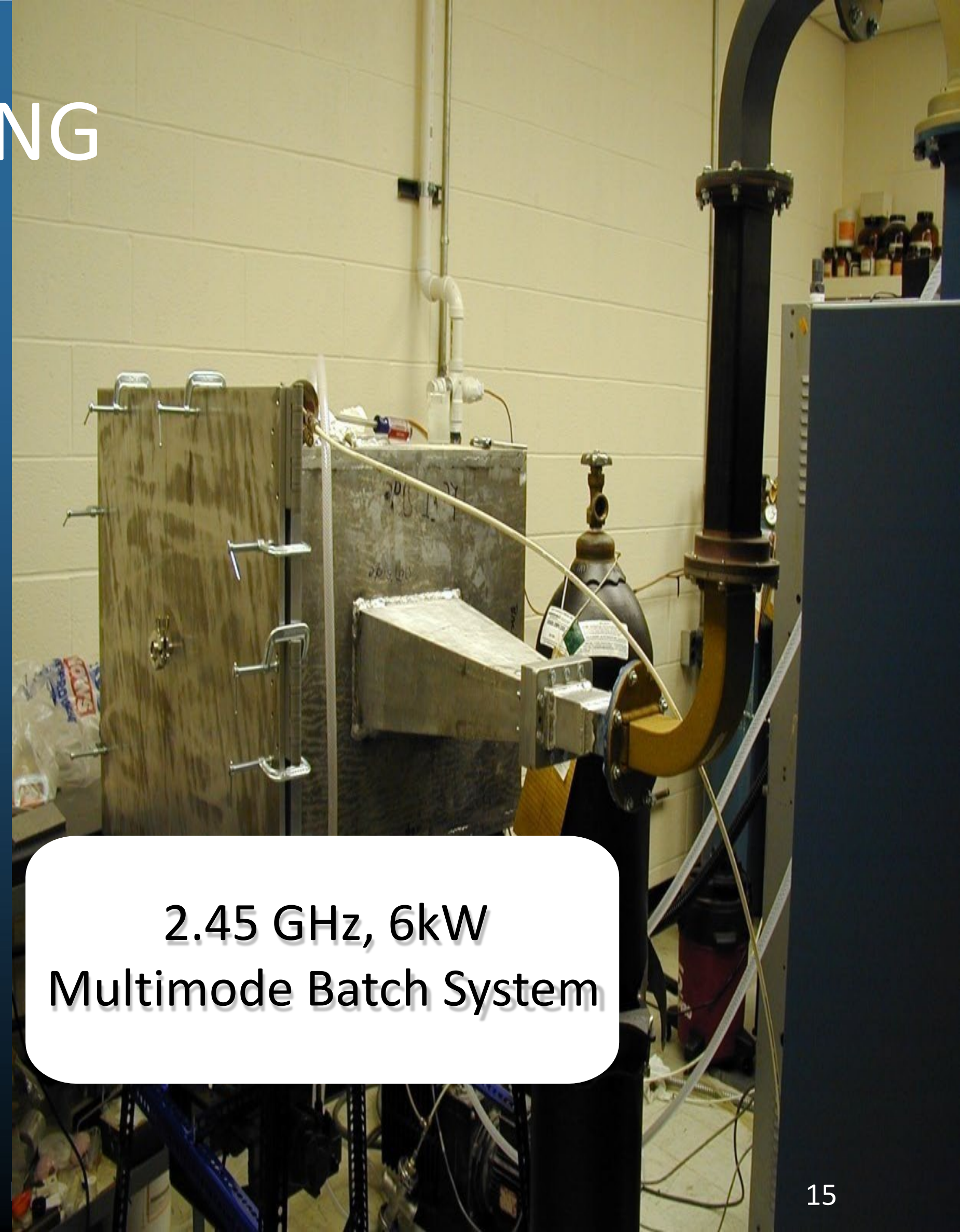
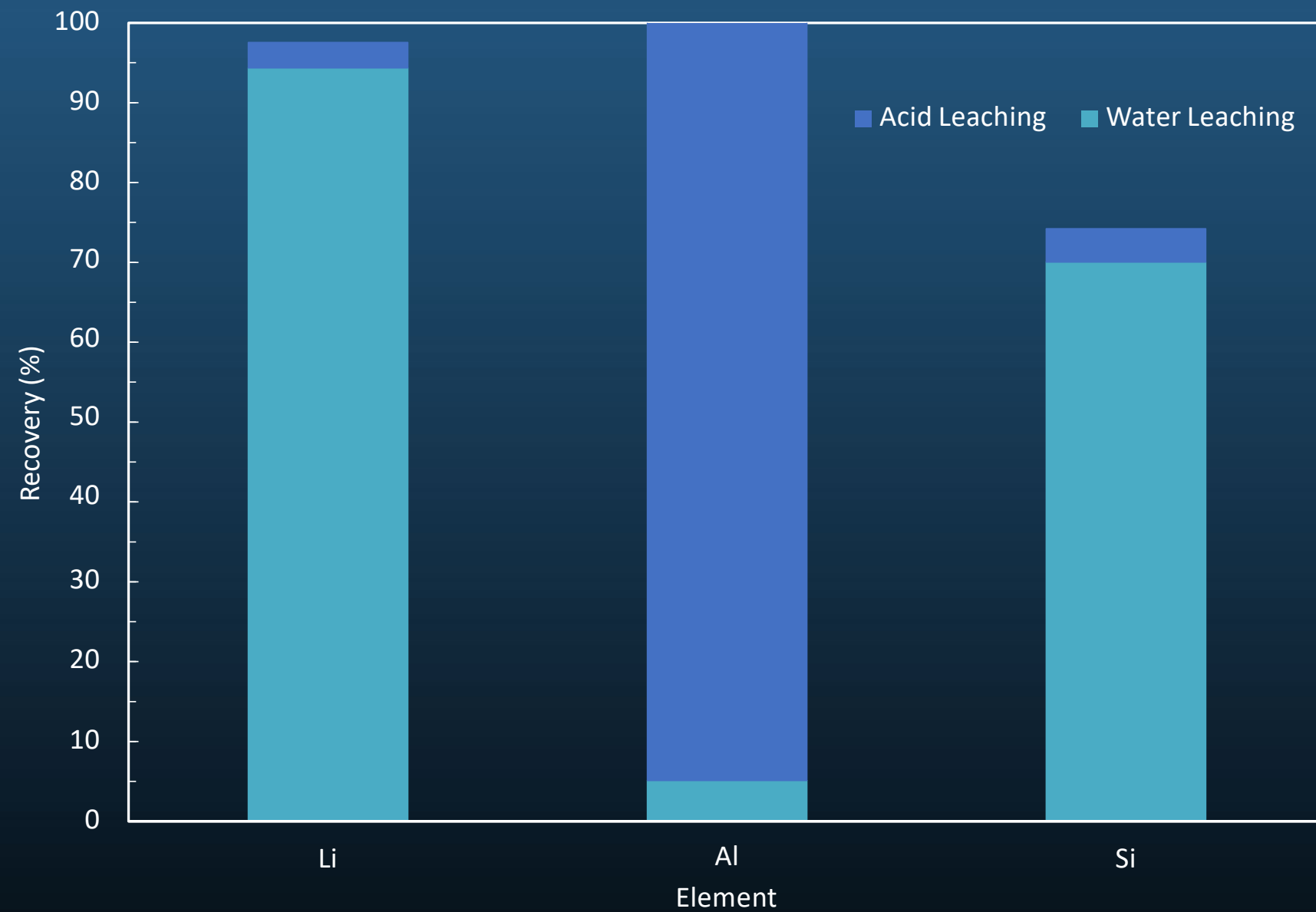


PATENT-PENDING NaOH ROASTING

- Penn State Process developed by Dr. Rezaee's research group eliminated the high temperature calcination and acid baking processes, and directly extracted Li from α -spodumene through NaOH roasting (at ~ 320 °C) followed by leaching.
- To further reduce the energy consumption and utilize the microwave heating advantages and polarity of NaOH, microwave NaOH roasting was implemented as NaOH quickly absorbs the microwaves.
- Compared to the conventional heating the benefits include cost-savings, low processing times, better controllable heating process with no greenhouse gas emissions, and direct, non-contact, selective, and volumetric heating are tangible benefits of microwave processing. The latter results in enhancing the porosity of the host mineral, thereby reducing the required sintering time, and chemical reactions time and temperature, while enhancing the lixiviants diffusion in the mineral and improving elemental recovery values.
- Microwave NaOH roasting of α -spodumene followed by water leaching recovered more than 95% of Li, while leaving Al in solid.

PATENT-PENDING NaOH ROASTING

- Microwave roasting of spodumene with NaOH was conducted at 1.5 kW and 400 °C



2.45 GHz, 6kW
Multimode Batch System

PENN STATE TEAM

Penn State stands as a hub of comprehensive expertise covering the entire spectrum of the critical minerals supply chain. This encompassing knowledge spans across pivotal domains including geology, mining, metallurgy, and alloy production. The university proudly accommodates a diverse range of cutting-edge facilities and pioneering research hubs, such as the renowned Center for Critical Minerals, the innovative Battery and Energy Storage Technology (BEST) Center, and the trailblazing Microwave Processing and Engineering Center. It's important to note that while Penn State serves as a fertile ground for groundbreaking advancements, the exclusive results of these groundbreaking efforts are a testament to the dedicated contributions of the research group led by Dr. Mohammad Rezaee.

Dr. Mohammad Rezaee

Associate Professor of Energy and Mineral Engineering

The Pennsylvania State University



Mohammad Rezaee is an Associate Professor of Mining Engineering and the holder of the Centennial Career Development Professorship in Mining Engineering in the College of Earth and Mineral Sciences. Dr. Rezaee's research focuses on developing sustainable mining waste disposal practices from which valuables such as critical elements can be recovered for commercial use while the remaining materials are processed to generate environmentally benign materials. His work centers around the extraction of critical elements from primary and secondary sources, coal preparation, mineral processing, environmental management, and computational fluid dynamics and scale modeling.

TECHNOLOGY READINESS LEVEL TIMELINE

- Penn State and Hertz Lithium will optimize the process parameters for maximizing Lithium recovery in water and conduct the kinetic study to obtain scale-up parameters for pilot-scale testing. By completion of this research project (within 6 months) the technology will be at TRL 4 at minimum.
- Pilot-scale prototype demonstration (~\$2.5M) within a period of 1 year will conclude the process at TRL 6.
- The economic assessment conducted in TRL 4 through 6 will provide the necessary information to understand the CAPEX required to bring the technology to TRL 9 (commercialization) as well as the day-to-day operating cost of the facility.

PROCESS SUSTAINABILITY ADVANTAGES

- The Penn State process directly recovers Li from α -spodumene through NaOH and water leaching.
- The developed process eliminates the high-temperature (1100 °C) calcination process, and associated energy cost as well as Green House Gas emissions.
- The process eliminates the high-temperature acid-baking process, and associated chemical cost, acid gas emission, and maintenance cost.
- The leachate obtained from NaOH roasting and water leaching will have a basic pH, reducing the chemical consumption and cost in downstream purification process.
- The process is fast, reducing the number and size of the process units, and associated cost.

THANK YOU!

FOR MORE INFORMATION, PLEASE CONTACT:

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 **HERTZ
LITHIUM**



APPENDIX I - ACKNOWLEDGMENTS

- Penn State Center for Critical Minerals (C²M)
- Penn State EMS Energy Institute (EI)
- Penn State Department of Energy and Mineral Engineering
- Penn State Microwave Processing and Engineering Center (MPEC)

APPENDIX II – SCIENTIFIC PROCESS

Microwave-Assisted Calcination

- Microwave-assisted calcination of spodumene offers significant advantages over calcination through conventional heating.
- Microwaves are electromagnetic waves with frequencies between 300 MHz and 300 GHz from which 915 and 2450 MHz are the most commonly used for industrial/commercial applications.
- Compared to the conventional heating, cost-saving, low processing time, better controllable heating process with no greenhouse gas emission, and direct, non-contact, selective, and volumetric heating are tangible benefits of microwave processing. The latter results in enhancing the porosity of the host mineral, thereby reducing the required sintering time, and chemical reactions time and temperature, while enhancing the lixiviants diffusion in the mineral and improving elemental recovery values.
- To utilize the potential benefits of microwave and hybrid processes, we studied the microwave-assisted calcination of spodumene, evaluated the effect of microwave power on phase transformation and leachability of this mineral, and compared the results with that of calcined via conventional heating.

APPENDIX III – SCIENTIFIC PROCESS CONT'D

Microwave-Assisted Calcination

- The optimum lithium recovery through the microwave calcination-acid roasting-leaching process reached 97% at 2.0 kW microwave power, comparable with that of conventional heating. The complete phase transformation in microwave calcination occurred at less than 20 minutes, compared to 2 hours for the conventional heating.
- Besides a more rapid, less energy-consuming, and free greenhouse-gas-emission calcination, microwave heating also resulted in lower recovery values of Fe, Na, and Ca in the leaching process, gaining an advantage in downstream purification processes.
- The critical temperature after which α -spodumene absorbs microwave energy was found to be around to be around 570 °C.

APPENDIX IV – SCIENTIFIC PROCESS CONT'D

Microwave-Assisted Calcination

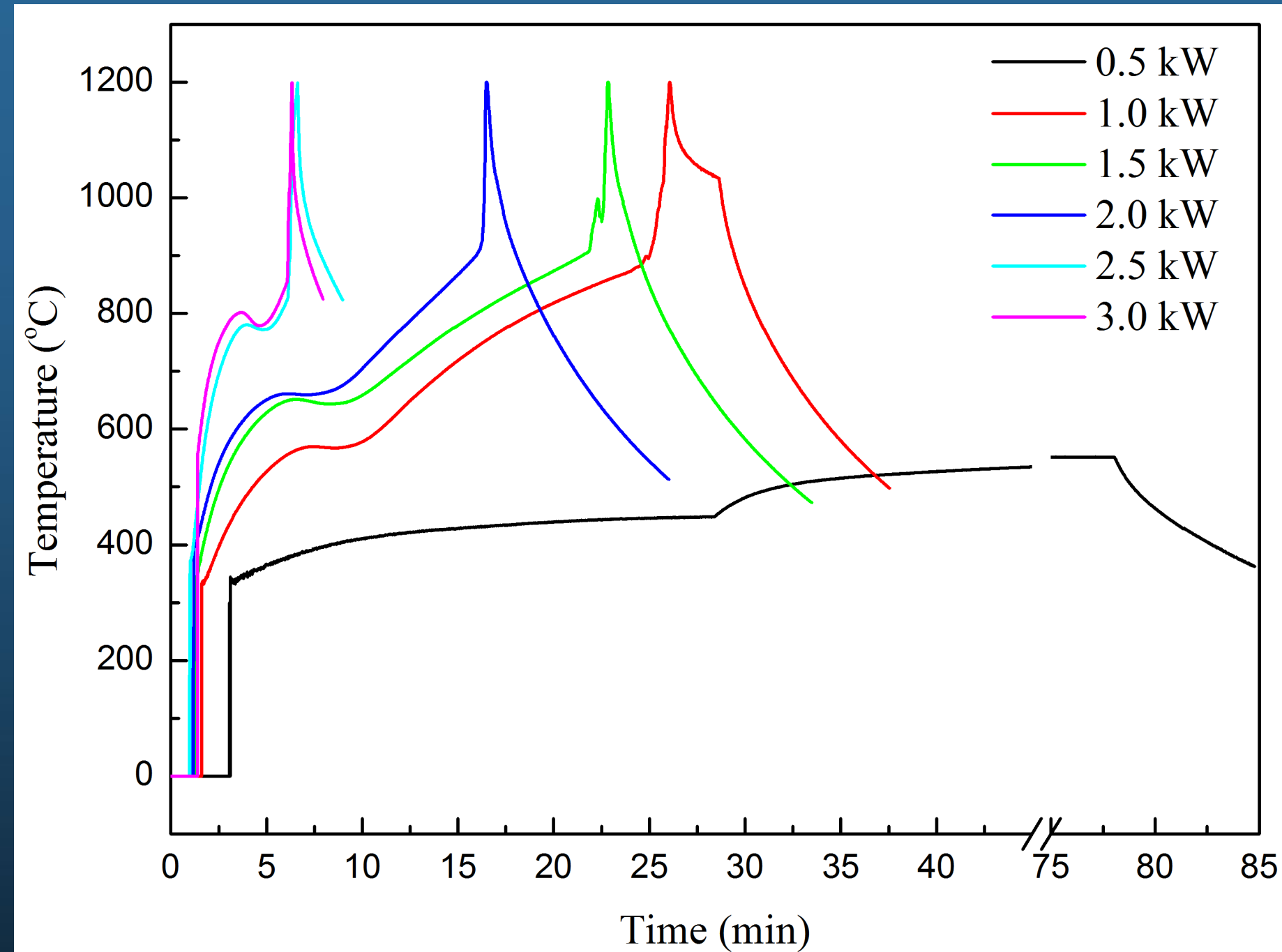
Spodumene Concentrate

Microwave Calcination

Acid Baking

Water Leaching

Purification



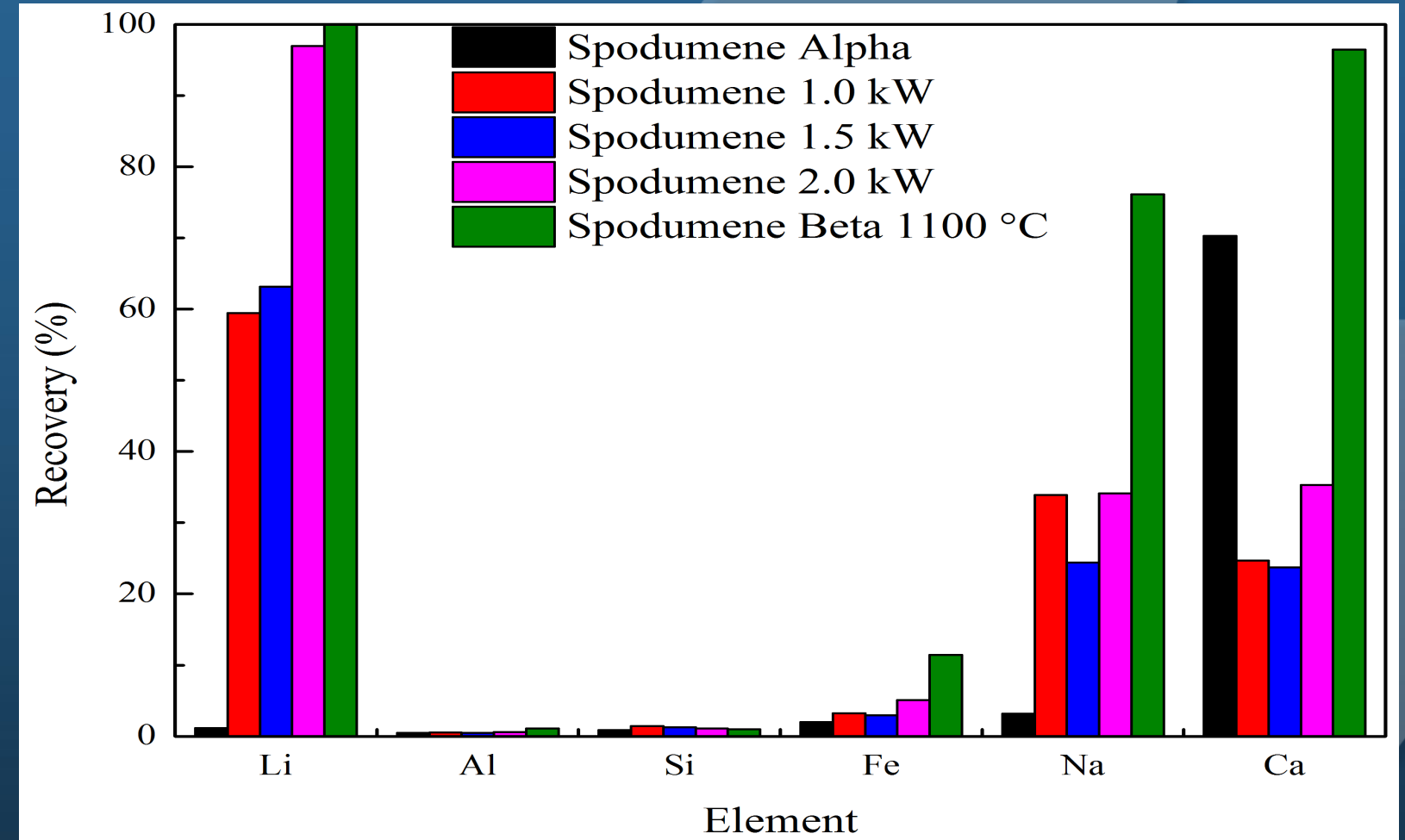
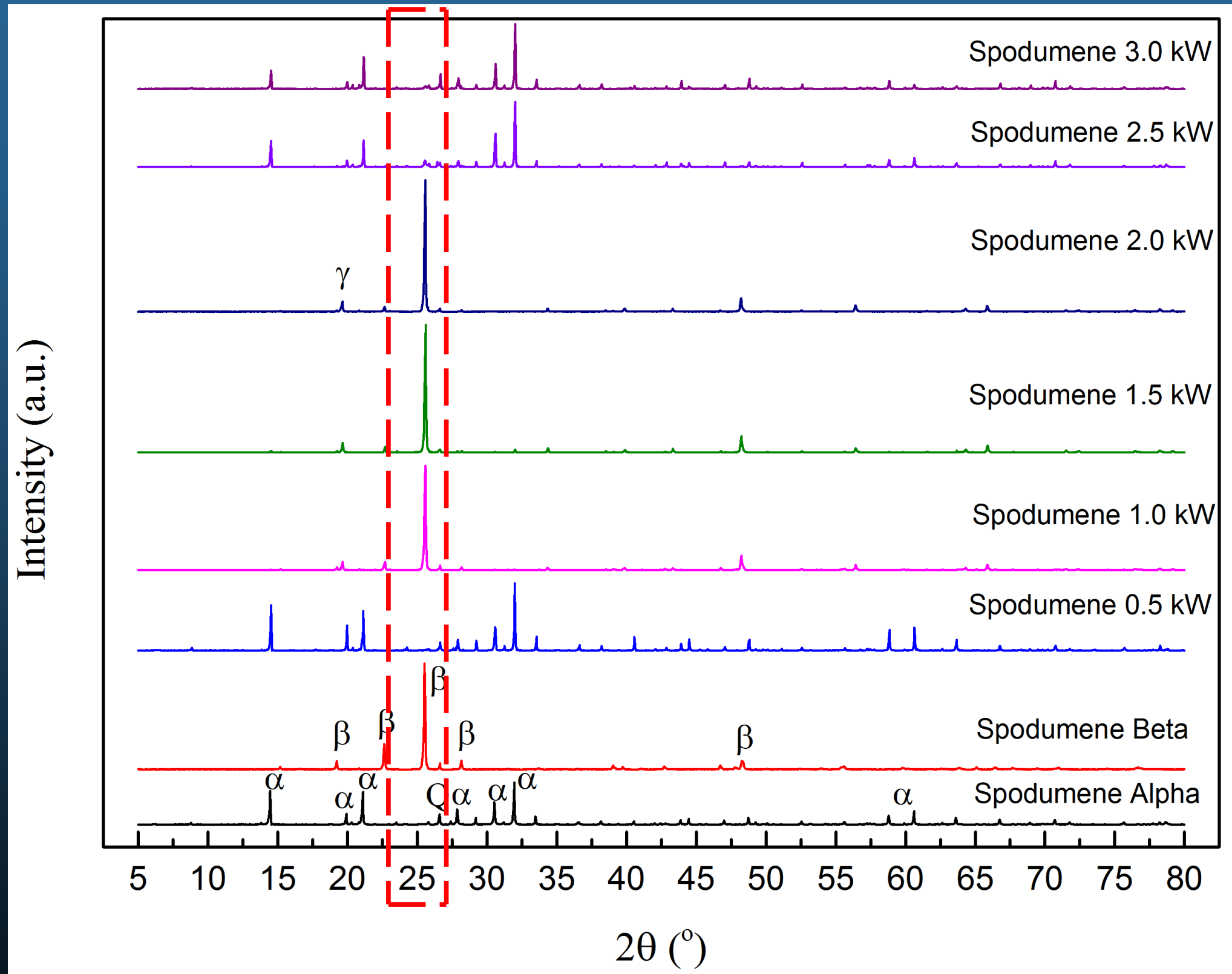
Critical temperature 570 °C



APPENDIX V - ELEMENTAL RECOVERY

- Conversion to β -spodumene

- 2kW: 97% Li
- Lower concentrations of impurities



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Microwave-assisted calcination of spodumene for efficient, low-cost and environmentally friendly extraction of lithium

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