DEVELOPING SUSTAINABLE SYNTHETIC ROUTES TO LITHIUM-ION BATTERY CATHODES

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INTRODUCTION

The increasing demand for energy and the threat from Global Warming make electrical energy storage a worldwide priority. Rechargeable batteries are modular and scalable and as such can meet the demands of a wide range of applications, like electric vehicles. While concerns about the sustainability and cost of lithium-based batteries has encouraged the development of battery chemistries relying on more abundant elements, the lithium-ion technology still dominates the market.

Figure 1. *Tesla Model X*

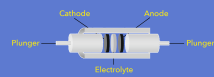

Figure 2. *Swagelok Cell Diagram*
Picture by ResearchGate

NMC-type cathodes have replaced lithium cobalt oxide in electric vehicle batteries, owing to their higher energy density and lower cost and toxicity due to the reduced cobalt content. Yet, the synthesis of these materials is commonly achieved via a solid-state route which generally involves two high-temperature (~1000°C) calcination steps of about 15 hours each.

Microwave (MW) synthesis is an emerging technique to replace the conventional solid-state route. What makes MW optimal is its homogeneous heating ability, compared to the conventional surface-to-core heating. This significantly decreases the time it takes for synthesis from 30 hours to 15 minutes. This also reduces energy consumption from 6 kW/h to 0.25 kW/h. These different heating processes are expected to result in different microstructures and therefore properties of the battery materials of interest to this project.

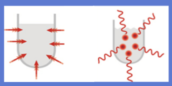

Figure 3. *Surface-to-core vs. homogeneous heating*

OBJECTIVE

Figure 4. *Tube Furnace*

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Figure 5. *Microwave*

The objective of this research study was to determine whether differences in synthesis methods (solid-state vs. microwave) will have an effect on NMC-type cathodes in rechargeable batteries. The purpose of this is to identify cheaper ways of synthesizing the active electrode material, which makes up ≥ 70 wt.% of the electrode.

CHARACTERIZATION TECHNIQUES

X-Ray Diffraction (XRD)
- Gives information on structure and composition of the material

Atomic Emission Spectroscopy (ICP-AES)
- Quantifies material composition more precisely

Nuclear Magnetic Resonance (NMR)
- Shows accurate material structure

Battery Cycler
- Determines electrochemical properties of material

MATERIALS AND METHODS

1. Masses of Li_2CO_3, MnO_2, Ni acetate, and Co oxalate were weighed.
2. The reagents were ground in mortar and pestle for 20 minutes.
3. The compound was pressed into a 6mm pellet with 1.2 tons of pressure.
4. The compound was placed heated in a microwave for 10-20 minutes.
5. The material was ground with carbon and binder inside and Argon glove box to create a cathode.
6. A coin cell battery was assembled using the cathode made.
7. The active battery cell was connected to a potentiostat to be cycled at a range of 1 V to 4.2 V for two weeks.
8. The X-ray Diffraction and cycling data of solid-state and microwave methods were compared.

XRD

KEY COMPONENTS

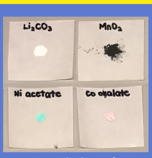

Figure 6. *Clockwise from top-left: Li_2CO_3, MnO_2, Ni acetate, Co oxalate*

Figure 7. *Microwave*

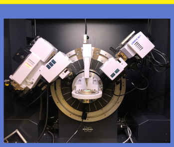

Figure 8. *X-ray diffraction machine*

Figure 9. *Coin cell battery*

Figure 10. *Three coin cell batteries being cycled*

X-RAY DIFFRACTION RESULTS

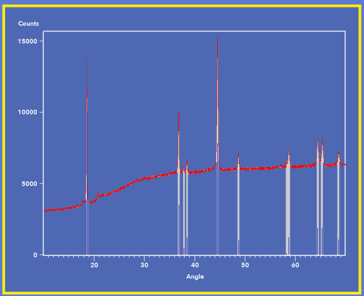

Figure 11. *Solid-state synthesis XRD result*

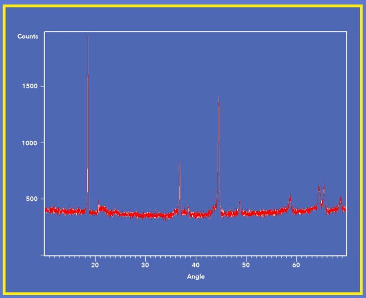

Figure 12. *Microwave synthesis XRD result*

The solid-state synthesis XRD graph of the NMC-type cathode material shows phase purity as indicated by the pattern (gray lines) and its correspondence to the peaks. On the other hand, the microwave synthesis result shows impurities at angle 43°. Futher optimization of microwave conditions must be conducted to achieve phase purity. The high count of peaks in solid-state synthesis also shows more prominent crystalization compared to microwave synthesis.

CONCLUSION

Figure 13. *Tube Furnace*

From the preliminary XRD data, it can be concluded that microwave synthesis can yield the same product as solid-state synthesis. If battery performance also deliver similar results, a significant amount of energy and time can be saved in the synthesis of battery cathodes for electric vehicles, and possibly beyond, using microwave techniques.

FUTURE WORK

To attain a more extensive understanding of the differences between solid-state and microwave synthesis and their effects on cathode-making, other characterization techniques must be conducted. As mentioned, ICP-AES and NMR will be the next steps. Furthermore, Scanning Electron Microscopy (SEM) must be used to test particle size.

Figure 14. *Atomic Emission Spectroscopy Machine*

Figure 15. Nuclear Magnetic Spectroscopy Machine

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