A Novel Synthesis of Lithium Sulfide Nanocrystals for Advanced Rechargeable Batteries
Miranda McCeig, a,b Yangzhi Zhao, a Xuemin Li, a Colin Wolden, a,b Yongan Yang, a
a Colorado School of Mines
b Department of Materials Science and Engineering, Rutgers University

Energy Storage Demands
Intrinsic material properties of the current lithium ion batteries (LIBs) are limiting the performance of in-demand electronic devices and vehicles.

Lithium sulfur batteries are a promising improvement.
Li2S batteries have a higher specific capacity, energy density, and cost less to produce, but dangerous lithium dendrites can grow within the battery and degradation can occur during lithiation.

Li2S nanoparticles offer a solution.
Li2S is pre-lithiated and thermally stable. However, current synthesis methods are high cost, high energy, and not producing nanostructures.[1]

Project Objective: To synthesize pure Li2S nanocrystals using recyclable reagents and solvents at ambient conditions while remediating toxic H2S and recovering valuable H2.

Synthesis Techniques

<table>
<thead>
<tr>
<th>Reaction</th>
<th>Equations</th>
</tr>
</thead>
<tbody>
<tr>
<td>Metalorganic (ROM)</td>
<td>[ \text{H}_2\text{S} + \text{M}_2\text{R}_2 \xrightarrow{\text{RT}, 1 \text{atm}} \text{M}_2\text{S} + 2\text{ROH} ]</td>
</tr>
<tr>
<td>Recycling</td>
<td>[ \text{ROH} \xrightarrow{\text{RT}, 1 \text{atm}} \text{M}_2\text{S} ]</td>
</tr>
</tbody>
</table>

- A metalorganic solution of lithium is first prepared in an argon environment.
- The solution is transferred into a Parr reactor and reacted with H2S to produce Li2S nanoparticles.
- The power is separated via centrifugation and fully dried.

Solvents: Dimethoxylethane (DME), Dibutyl Ether (DBE), Dioxane, Toluene, and 2-Methyltetrahydrofuran (MeTHF)

Reactions

Several combinations of reagents and solvents were tested.

Analysis:
- H2S was fully reacted with all solutions as shown via QMS.
- Pure Li2S could not be synthesized for most combinations.
- The time of solvent addition and drying process were studied further for the promising ethanol-DBE system.

Lithium Ethoxide in Dibutyl Ether

Fig. 3 XRD comparison of different synthesis treatments.
- (A) Overnight solvent addition, 80°C drying.
- (B) Overnight solvent addition, 250°C annealing.
- (C) 5 hours solvent exposure, 80°C drying.
- (D) 5 hours solvent exposure, 130°C drying.
- (E) Solvent addition immediately before reaction, 80°C drying.
- (F) Solvent addition immediately before reaction, 130°C drying.

SEM Images of Overnight Solvent Addition, 80°C Drying

- Pure Li2S was obtained from the Li-ethanol-DBE system.
- Solvent exposure time affects both nanocrystal purity.
- The drying process affects the nanocrystal morphology, but has little affect on purity.

Conclusion

Future Work
- Electrochemical performance of pure Li2S as a cathode material will be tested.
- Morphology of nanocrystals will be tuned.
- Production yield will be optimized.
- One pot technique combining synthesis and electrode fabrication will be developed.
- Scalability will be investigated.

References and Acknowledgements


I would like to acknowledge the funding and support of the National Science Foundation award DMR-1461275, REU Site: Research Experiences for Undergraduates in Renewable Energy and the Colorado Office of Economic Development and International Trade (COEDIT).