

Engineering Sulfur Cathode Materials for Lithium-Sulfur Batteries



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Introduction

With the rise in demand for energy storage, Li-Ion batteries are critical devices[1,2]. For this project, three materials were attempted to be synthesized as Li-Ion battery cathodes, iron(II) sulfide-sulfur composite, lithium sulfide, and lithium disulfide.

Cathode Materials

- $\text{FeS}_2 + x\text{S}$ derived from FeE_3 ($E = -\text{S}_2\text{C}_5\text{H}_{10}\text{N}$)
 - FeE_3 synthesized according to $\text{FeCl}_3 \cdot 6\text{H}_2\text{O} + \text{Na}(\text{S}_2\text{C}_5\text{H}_{10}\text{N}) \cdot 3\text{H}_2\text{O} \rightarrow \text{Fe}(\text{S}_2\text{C}_5\text{H}_{10}\text{N}) + \text{NaCl}_3 + 15\text{H}_2\text{O}$
- Li_2S
 - Commercial (Alfa Aesar)
 - With PVP, Carbon Black
- Li_2S_x – synthesized
 - Solid state, precipitate, crystallization

FeS + S Cathode

Electrospinning

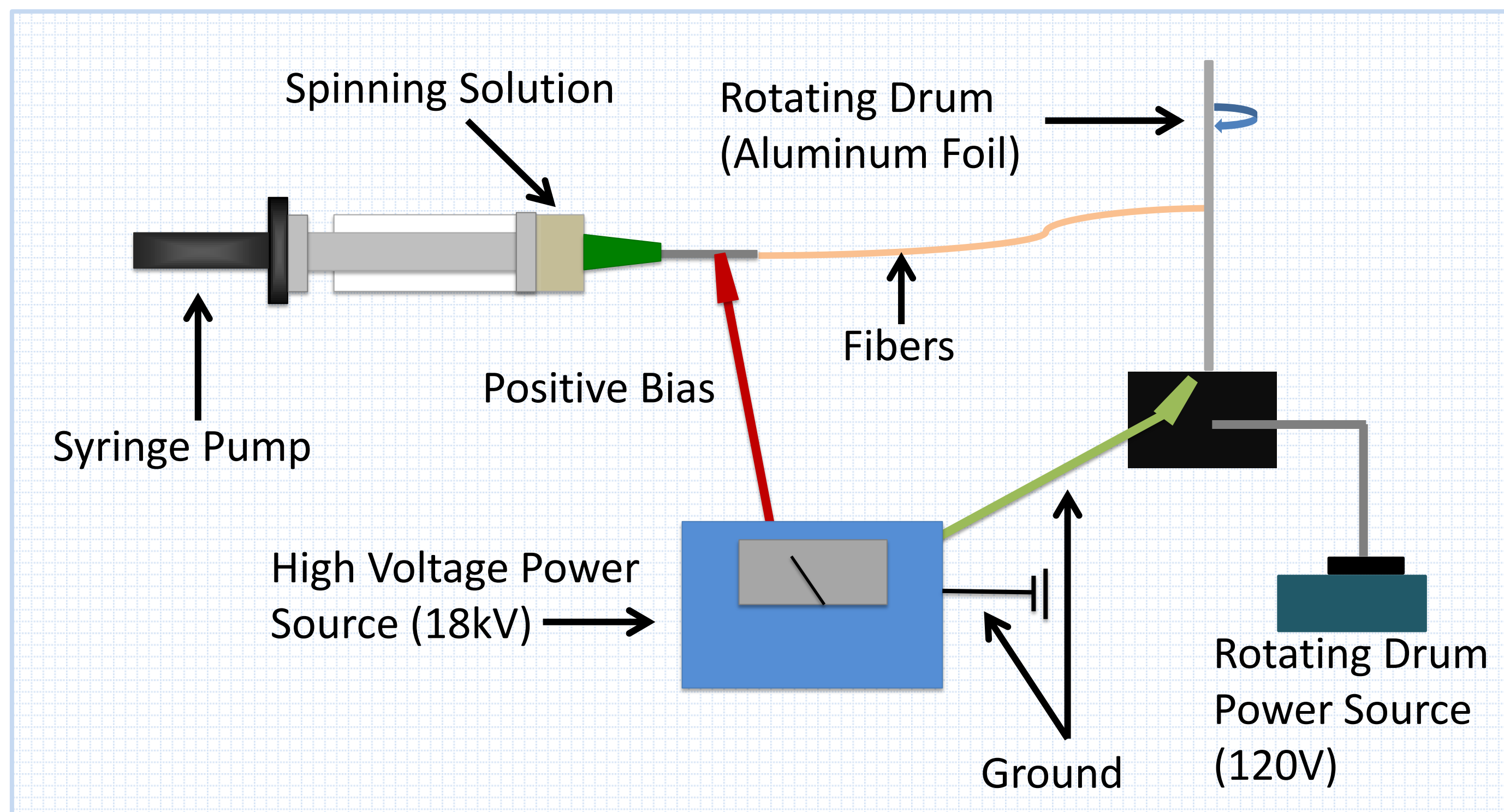


Figure 1- Schematic of the electrospinning set up used for this project. All power sources were separated from the spinning process via an insulated box.

Carbonization

- Free standing membrane obtained
- Annealing procedures : 250-600°C for 1-12 hours
- Poor capacitance



Figure 2- (A,B) Free-standing membranes of PAN/Graphene/FeE₃. (C) Annealed intact membrane (500C for 3 hours)

Li₂S Cathode

- Alfa Aesar Li₂S-PVP-Carbon Black/EtOH Slurry on Carbon paper
- Li₂S dissolved in EtOH before additives
- Batteries relatively stable, good capacitance

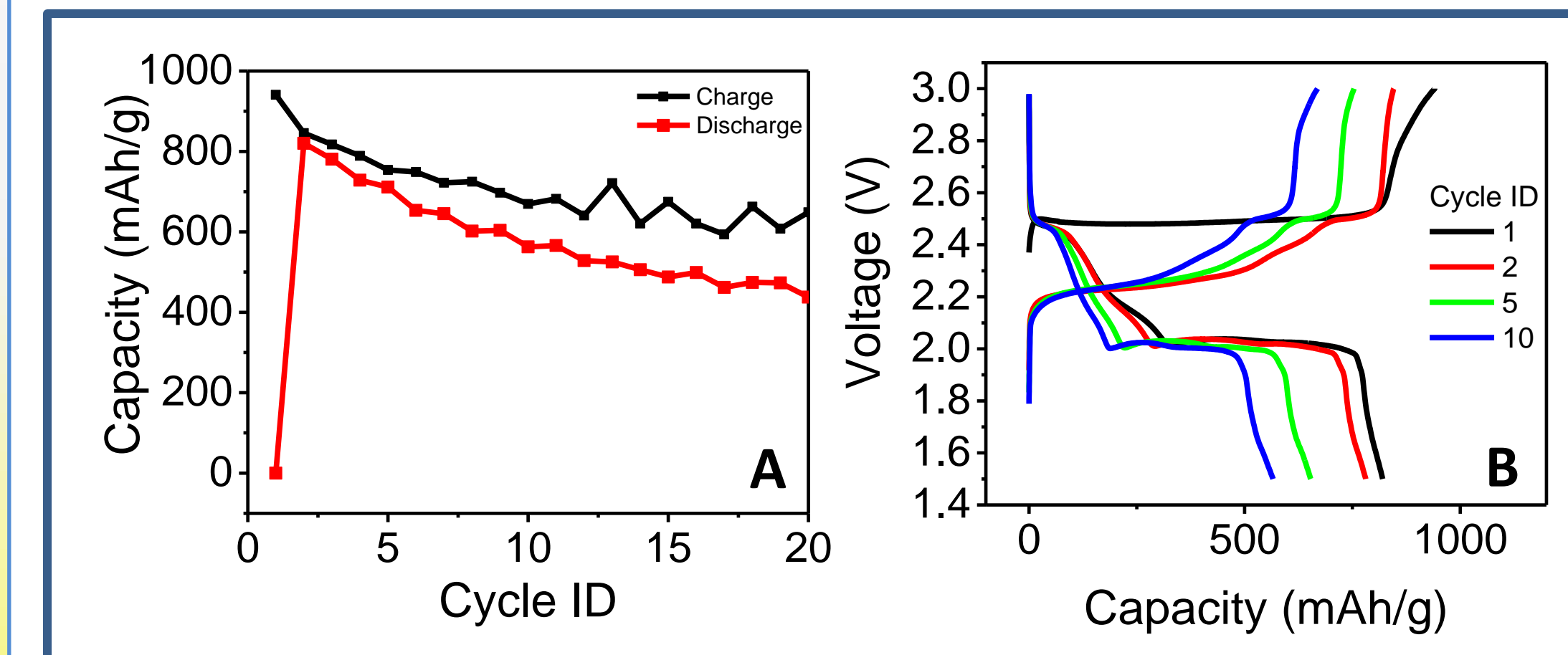


Figure 3- Battery Analysis: (A) Cycling performance. (B) Voltage profile against capacity.

Figure 4- (A)

Cyclic voltammograms plot. (B) Plot of dQ/dV vs. Voltage. Corresponding peaks between plots are labeled 1-5.

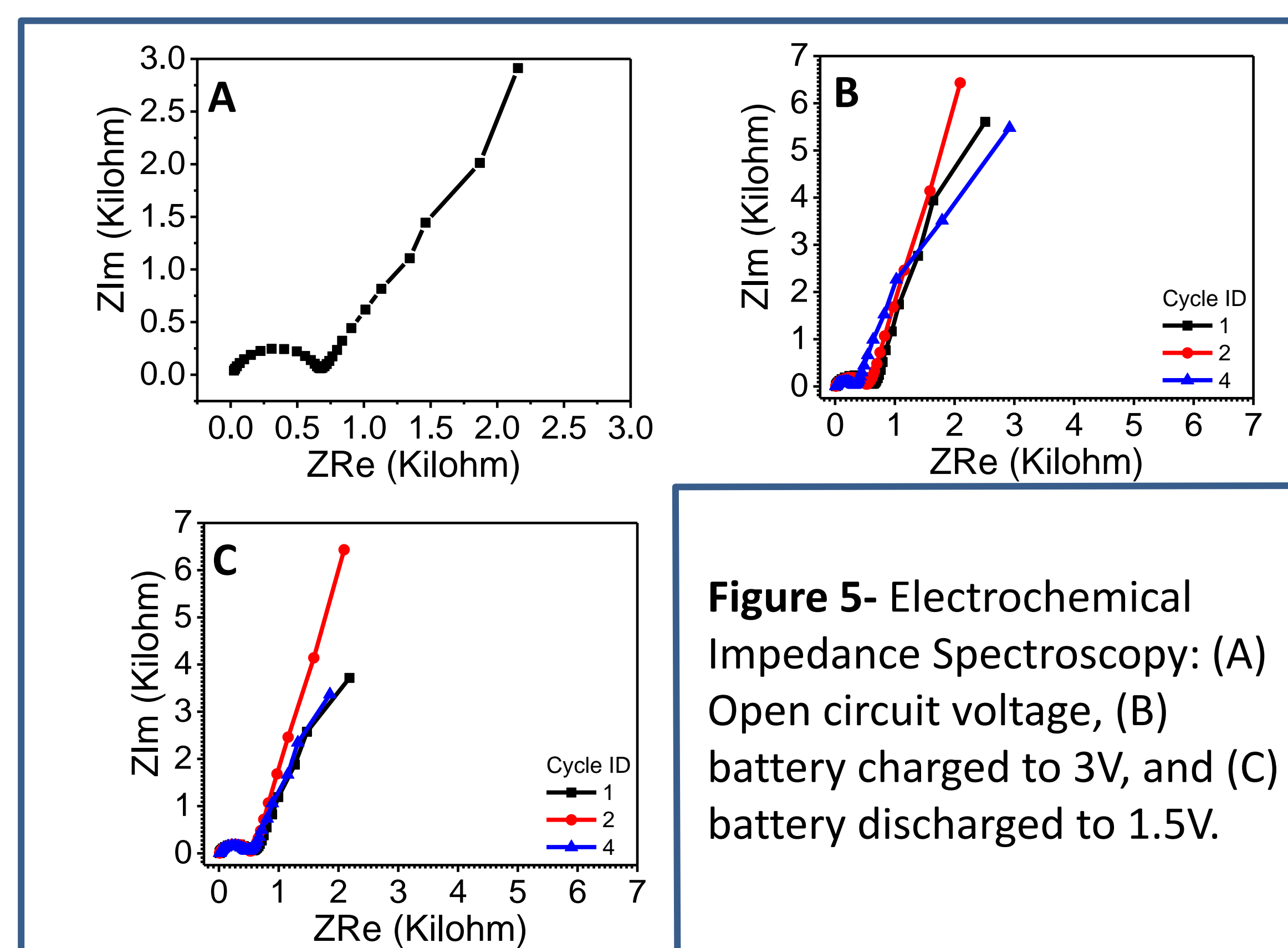
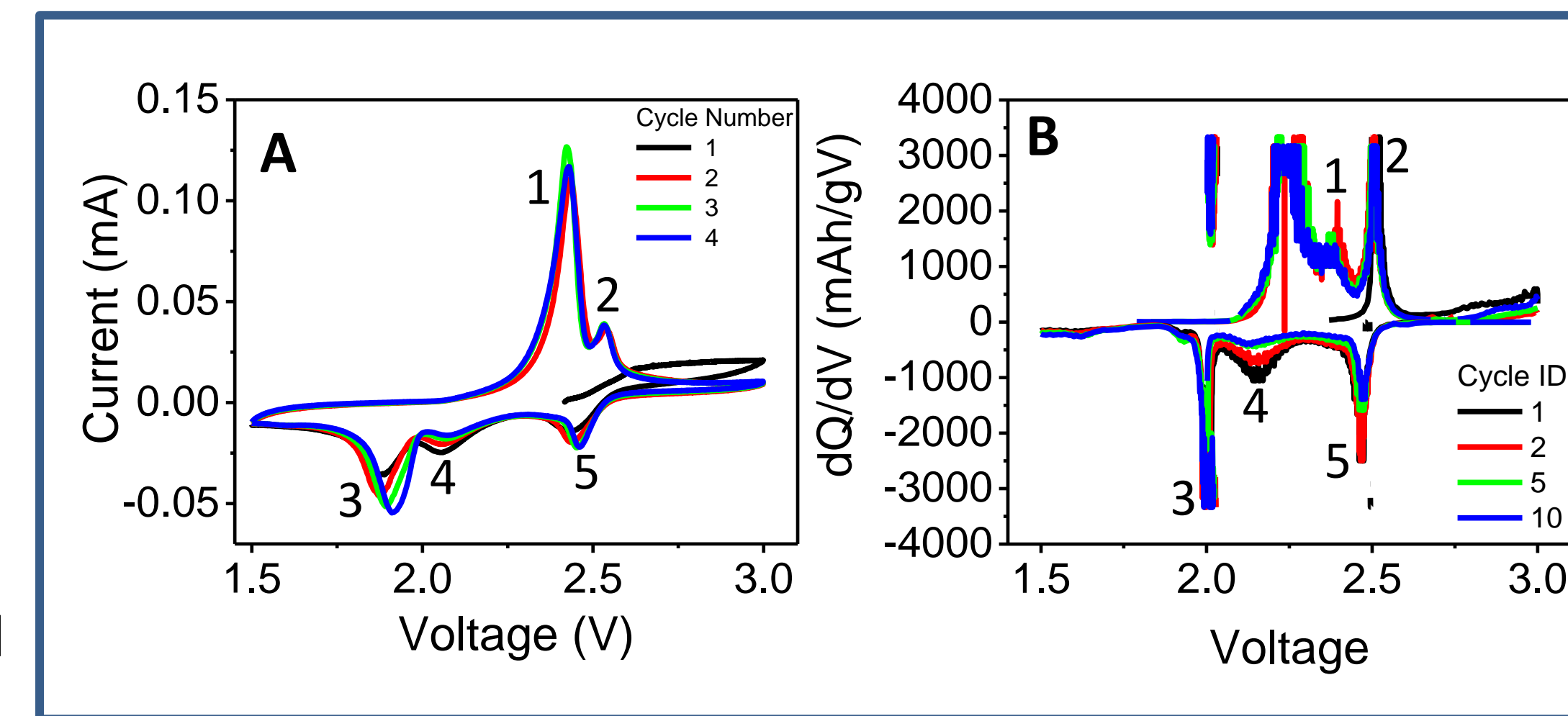


Figure 5- Electrochemical Impedance Spectroscopy: (A) Open circuit voltage, (B) battery charged to 3V, and (C) battery discharged to 1.5V.

Li₂S_x Synthesis and Characterization

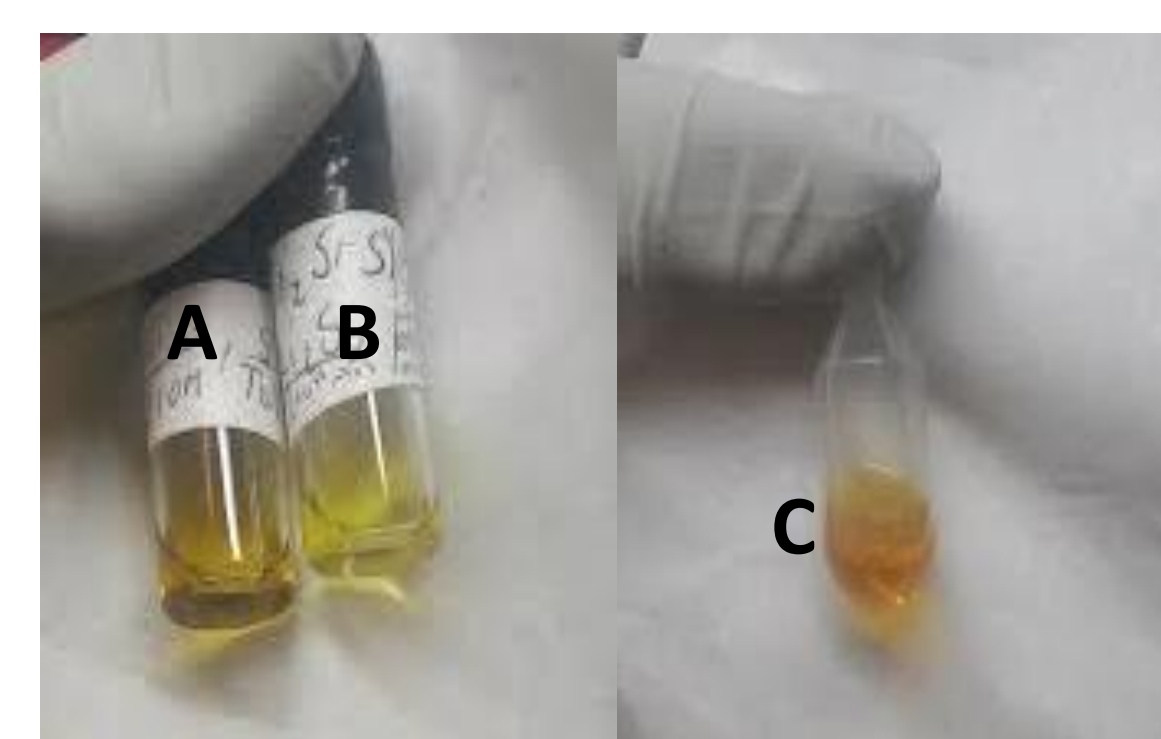


Figure 6- Li₂S_x synthesis attempts using commercial powder. (A) Li₂S/EtOH + S/Tol (1:1), (B) Li₂S/Mix + S/Mix (Mix is EtOH-Tol 50/50). (C) Several solutions were made in attempts of crystallizing Li₂S₂, Li₂S₃, and Li₂S₄ (Li₂S₃ shown). All solutions were colorless before combination.

Ultraviolet-Visible Spectroscopy of Li₂S₂

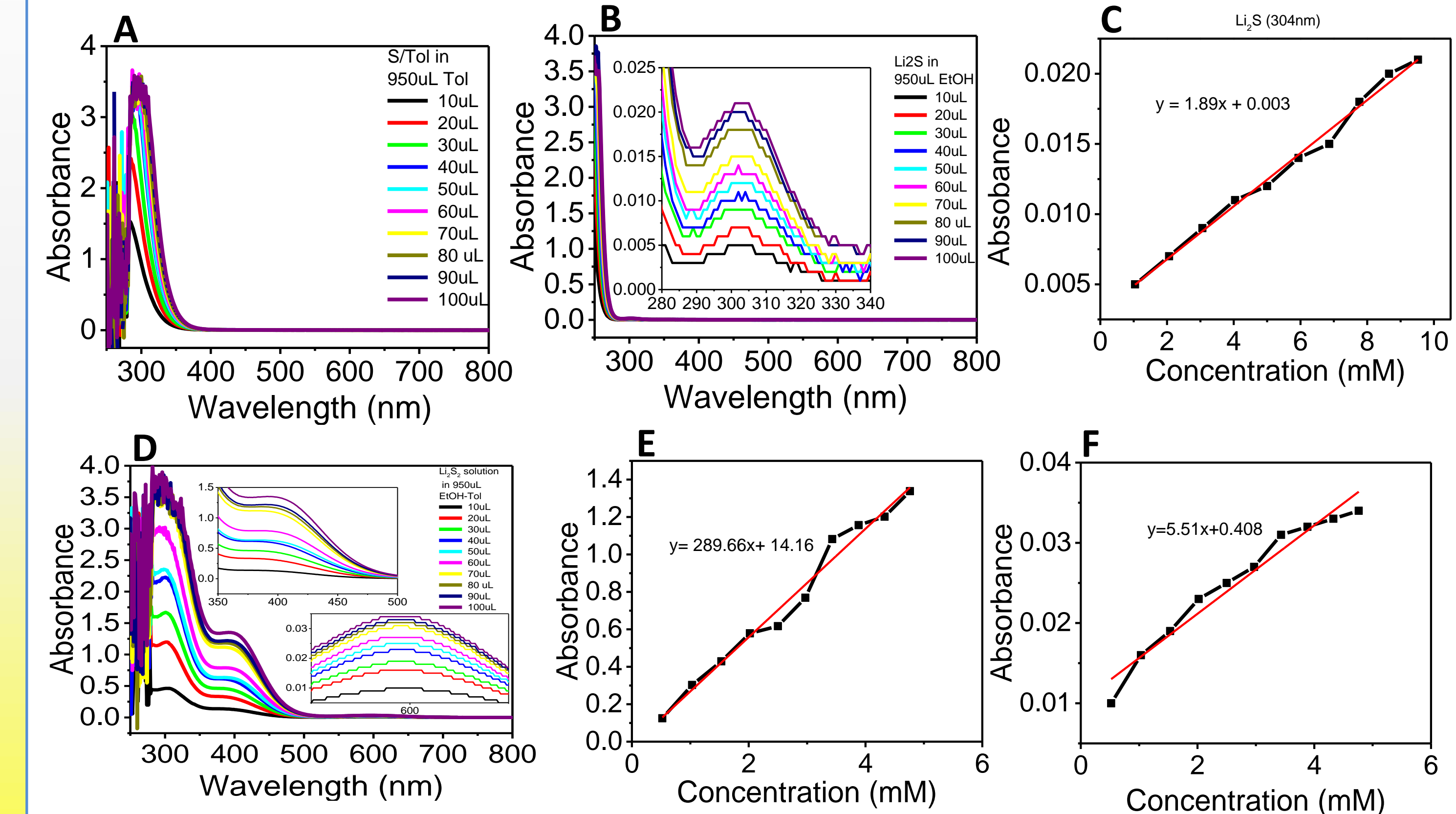


Figure 7- UV-Vis of (A) S/Tol, (B) Li₂S/EtOH, & (D) Li₂S₂/Tol-EtOH. Absorbance vs. concentration of (E) Li₂S at 304nm and (F,G) Li₂S₂ at 400nm and 600nm respectively.

Other Characteristics of Li₂S₂

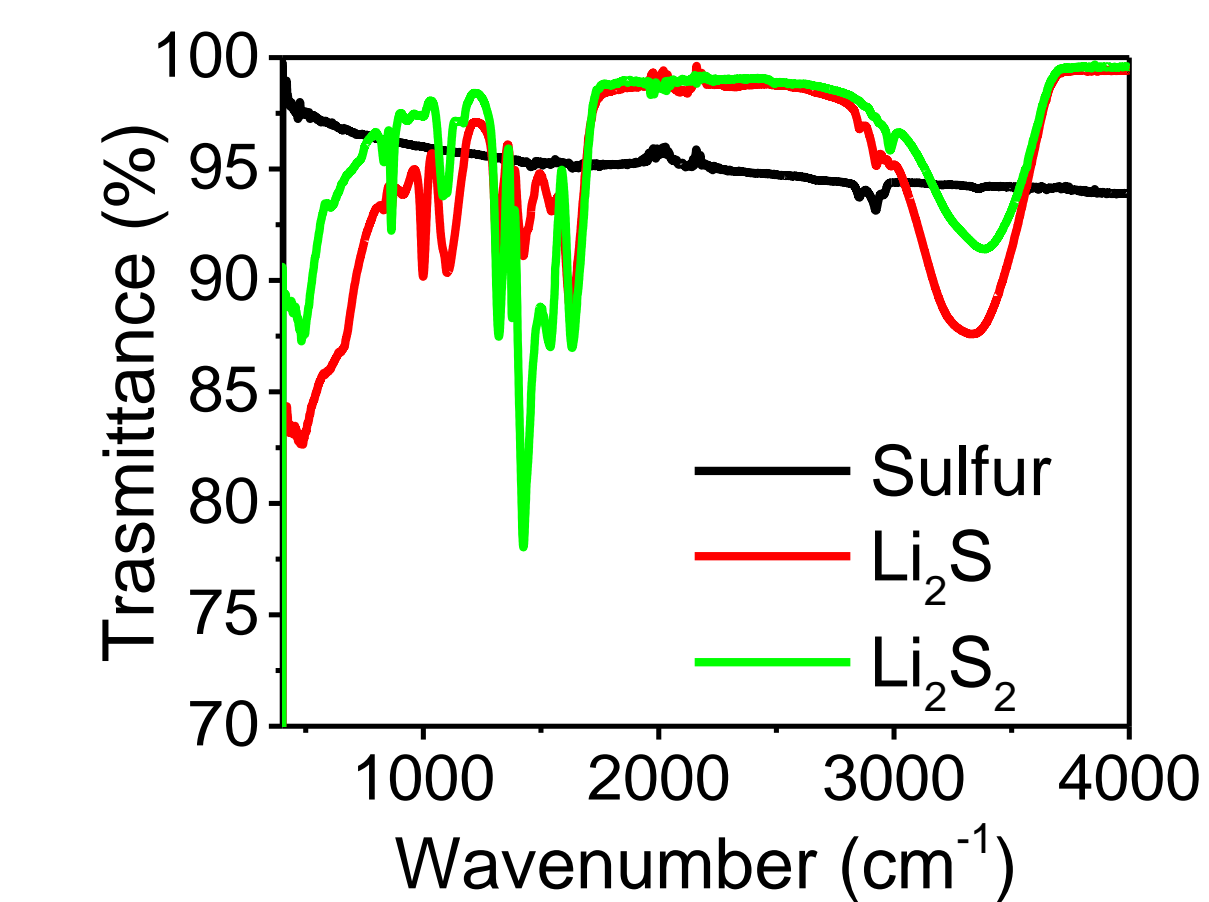


Figure 8- FTIR of S, Li₂S, and Li₂S₂.

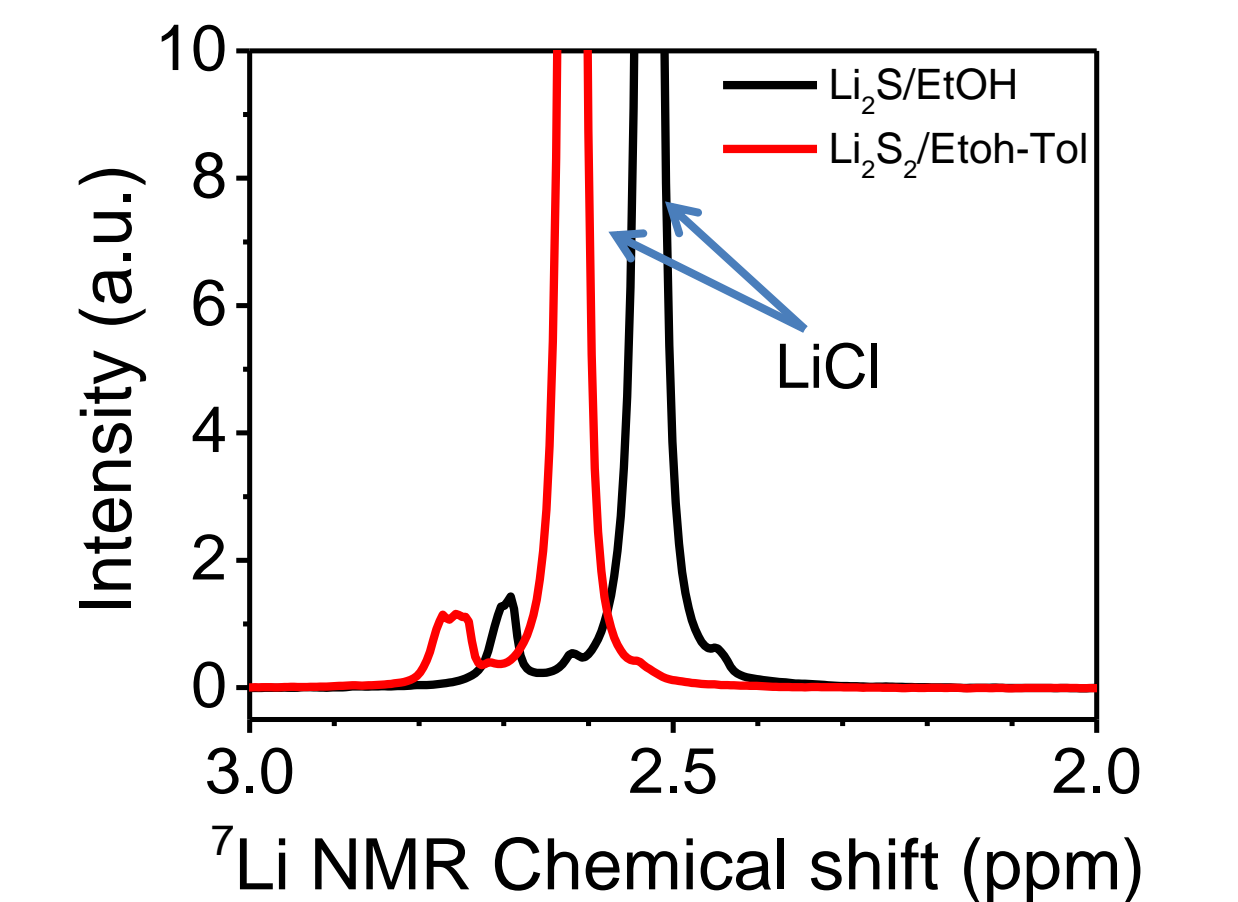


Figure 9- NMR peaks of Li₂S & Li₂S₂ with referent LiCl peak.

Conclusions

- The synthesis of FeS-S composite from FeE₃ is challenging and needs optimization of the thermal treatment.
- Li₂S cathodes present good capacity and stability.
- New compound Li₂S_x may have been successfully synthesized.

Acknowledgements

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References

- [1] Yin, Y.; Xin, S.; Guo, Y.G.; Wan, L.J. "Lithium-Sulfur Batteries: Electrochemistry, Materials, and Prospects," *Angewandte Chemie-International Edition*, 2013, 52, 13186-13200.
- [2] Song, M.K.; Cairns, E. J.; Zhang, Y.G. "Lithium/sulfur batteries with high specific energy: old challenges and new opportunities," *Nanoscale*, 2013, 5, 2186-2204.