Mizzou
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An Integrated Flame Spray Process for Low Cost Production of Battery Materials

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Overview

Timeline

- Start: January 1, 2016
- End: December 31, 2018
- Work completed: 1st quarter

Budget

- DOE: $2,215,556
- Cost Share: $310,694

Partners

- EaglePicher Technologies
- Other partners (in talks with National Labs and relevant Industries).

Technical Barriers

- High cost in cathode materials manufacturing
- Multiple manufacturing processes
- Pollution prone manufacturing (NOx, SOx, etc. in gas phase)
- Water intensive manufacturing (aqueous solution processing)

Project Objectives

An integrated manufacturing technology for battery materials production.

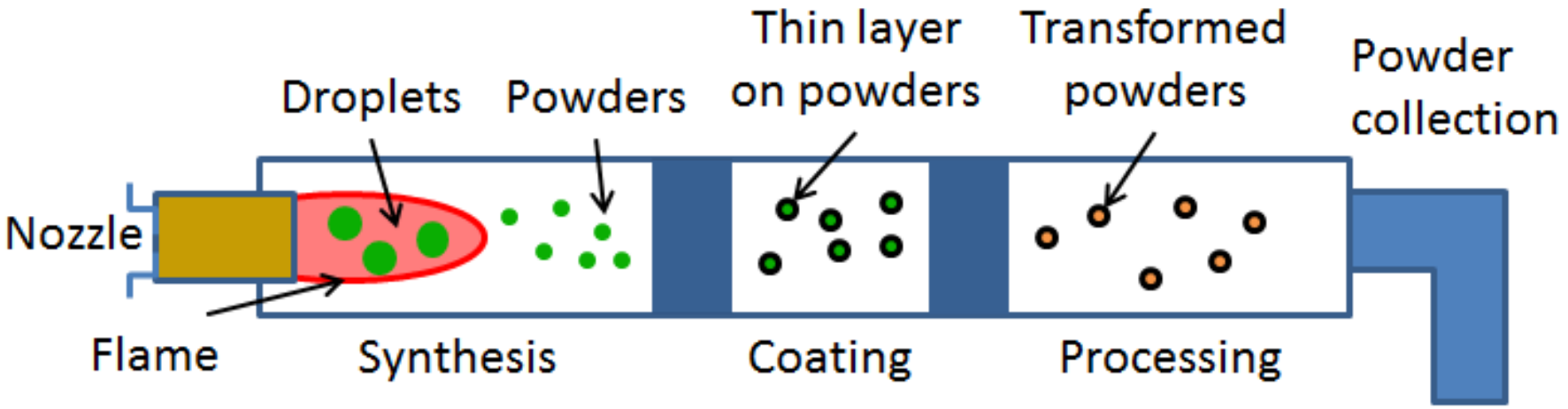
- To develop green manufacturing processes.
- To reduce materials cost by 25% (vs. baseline).
- To develop cells with 250 Wh/kg.
- To develop a pilot scale production line.

Technical Approaches

The proposed manufacturing technology will integrate multiple processes into one to reduce cost.

- Development of new chemical precursors, deep eutectic solvents (DESs), in which biomass glycerol is used as solvent in stead of water.
- Development of a new flame spray process to spray DESs and convert their droplets into battery material powders.
- Development of an integrated technology combining materials synthesis and processing into one process.
- Development of high performance cells using the produced cathode materials and existing anode materials.

Illustration of the Integrated Process



The battery powder production is aimed at integrating synthesis, coating, and processing into one process. The materials produced are ready for use in battery cathode fabrications without further processing.

Task - Spray Precursor Formulation, Year 1

Formulation of metal containing Deep Eutectic Solvent (DES) precursors

1 - Develop formulations of DESs for making NMC materials.
2 - Develop formulations of DESs for making NCA materials.
3 - Investigate DESs for low viscosity solvents: the formulations of DESs obtained will be studied to obtain their viscosities.
4 - Investigate compositions for making stoichiometric materials.

Task - Spray Nozzle Development, Year 1

Spray nozzle development for DES spraying

1 - Design and fabricate spray nozzles and flame reactor. This subtask will redesign a flame spray nozzle as well as the flame reactor.

2 - Investigate spray parameters to produce uniform spray droplets. In conjunction with nozzle development, the droplets from sprays will be characterized.

3 - Study conditions to control powder sizes. Active material powders, depending on applications, can be large (e.g., ~10 microns) or small (e.g., ~0.5 microns). The flame spray technology will be studied to produce a range of powder sizes.

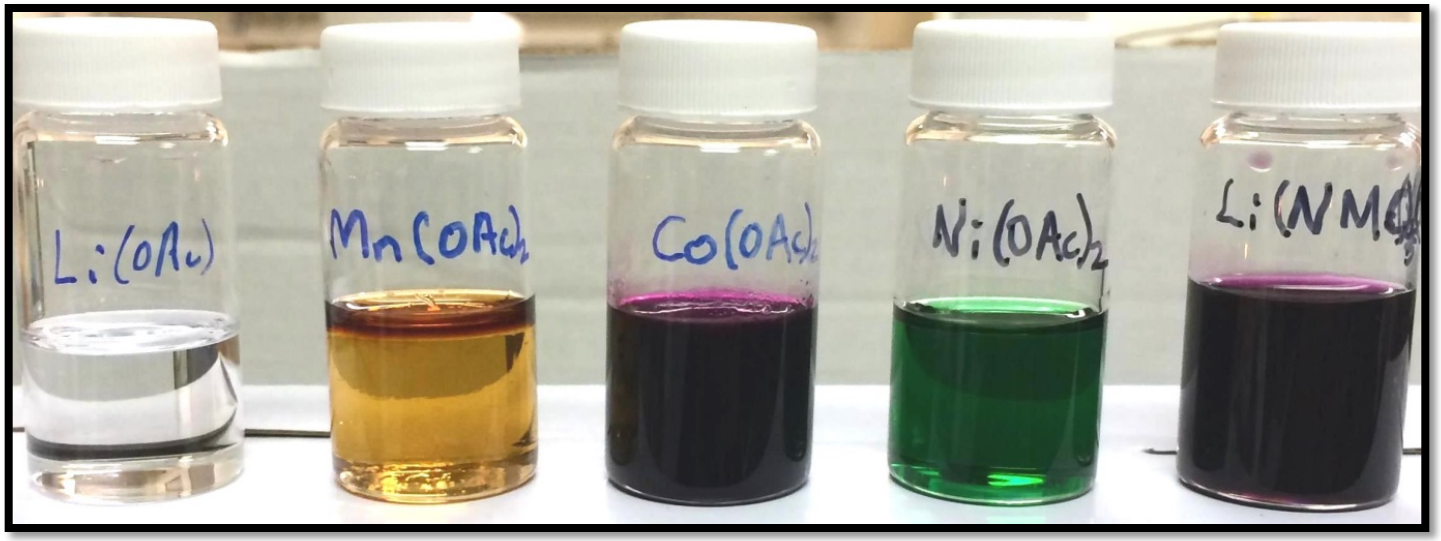
Task - Battery Cell Development, Year 1

Battery cells based on manufactured materials

1. Full characterization of the produced cathode powders, and evaluation with battery cells in consideration. Initial cathode powder evaluation will be performed in the half (coin) cell format.
2. Cathode fabrications will use similar formulations to those developed in previous programs that delivered high energy cells but with the materials produced from the spray process.
3. Performance data will be used to improve materials formulations and morphologies, and cell design and optimization in subsequent subtasks.

Q1 Results 1 – DES Formulations

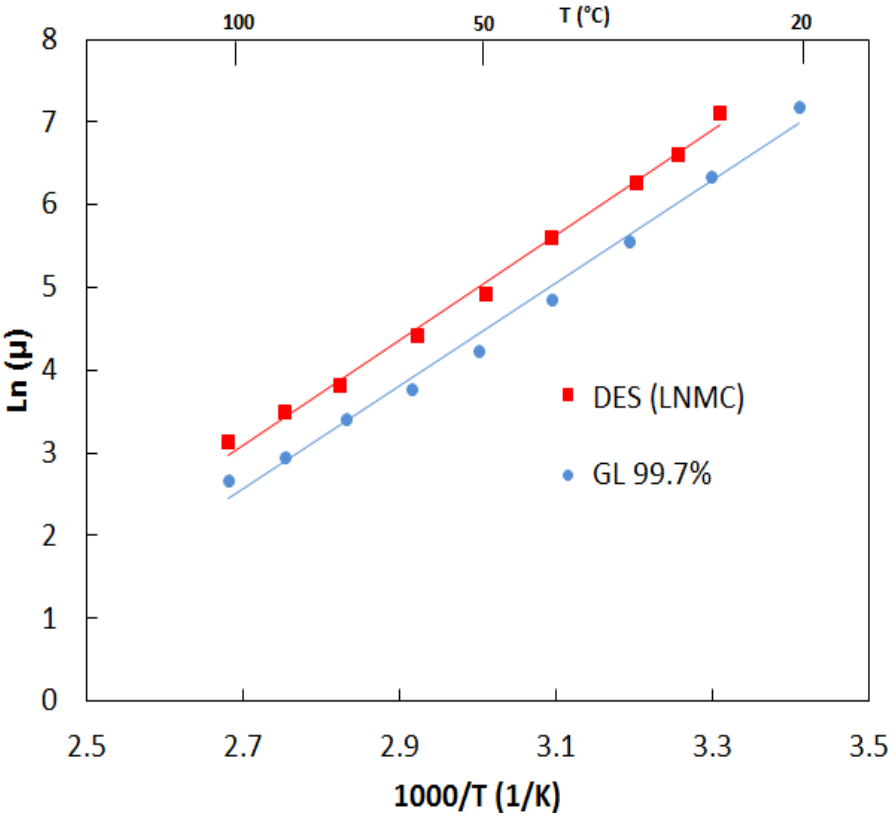
DESs have been under study in their formulations and properties.

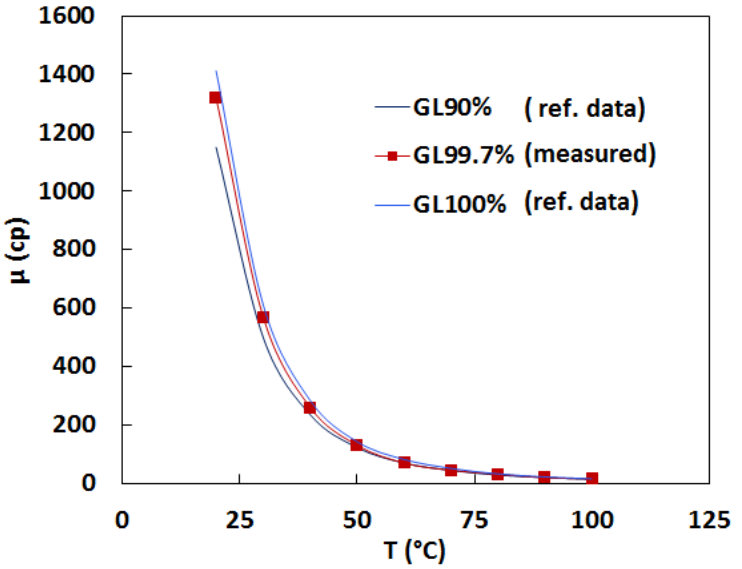


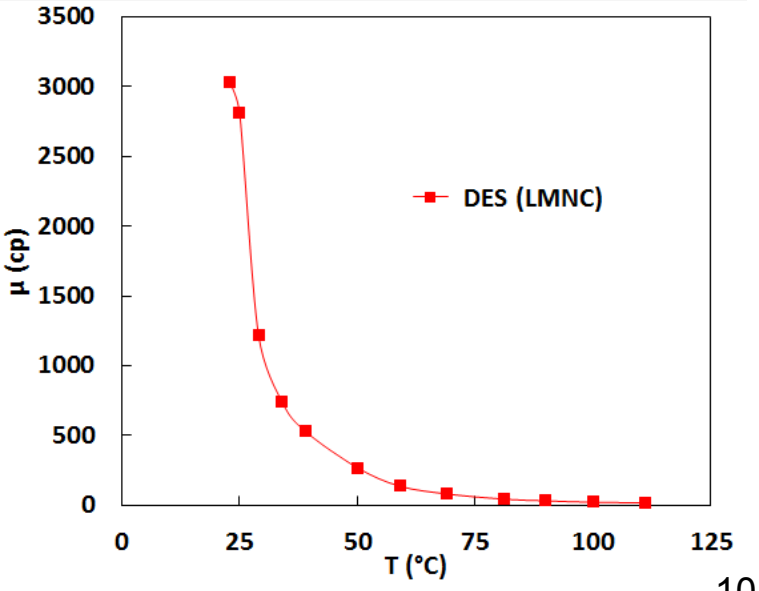
Room temperature DESs of different metals for battery materials. The DESs show clear solutions of the metal acetates dissolved in glycerol (shown are with 1:5 molar ratio).

Q1 Results 2 – DES Viscosity

DES has a viscosity of a Newtonian fluid at temperatures above room temperatures (25 ºC), but larger than glycerol (GL) at same low T.

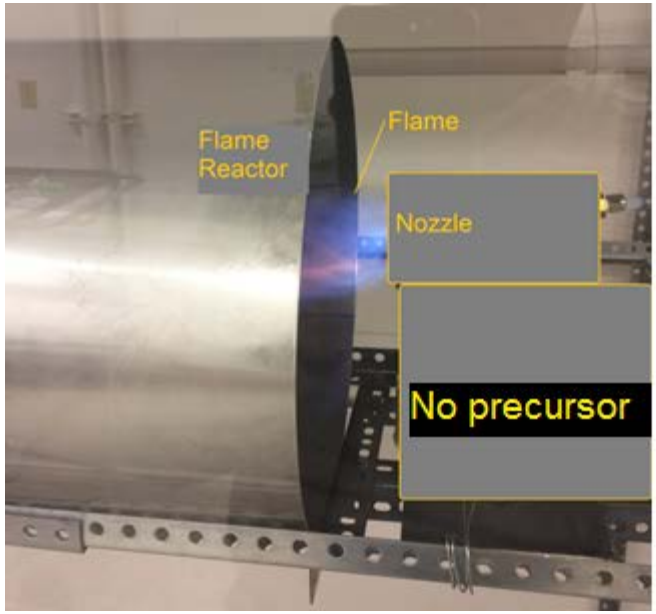


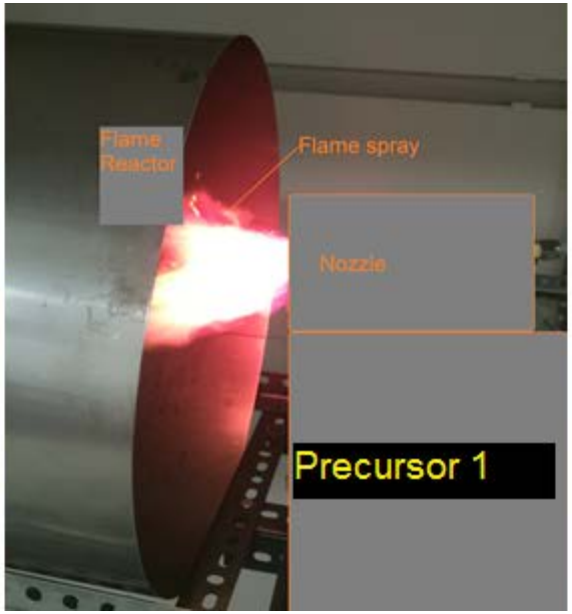


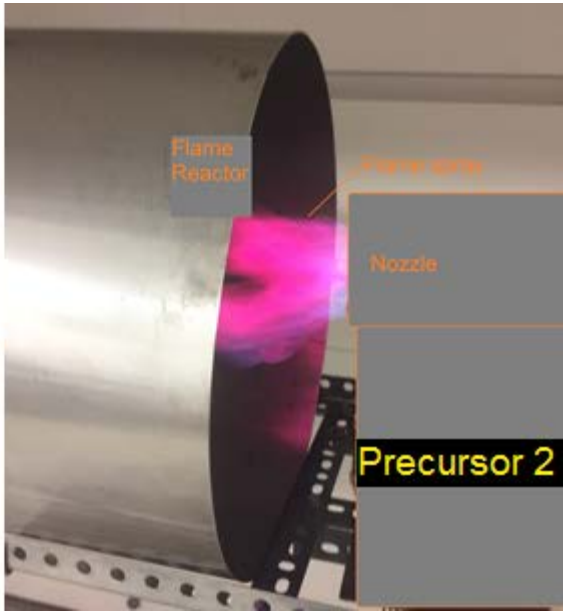


Q1 Results 3 – Process Development

- **Spray nozzle is being developed and under test.**
- **Flame reactor is being developed and under test.**



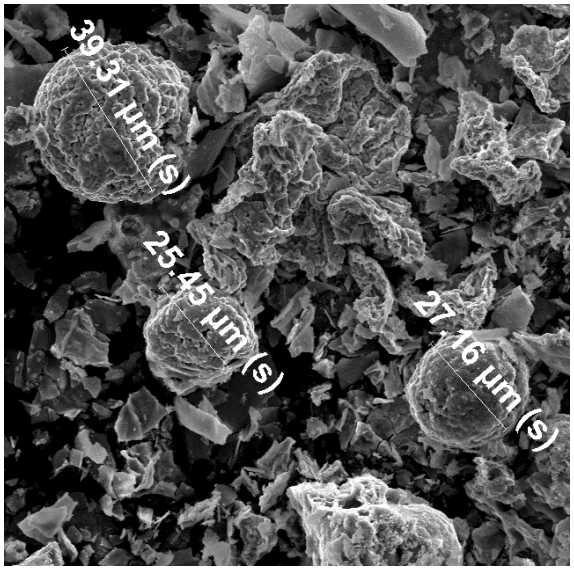


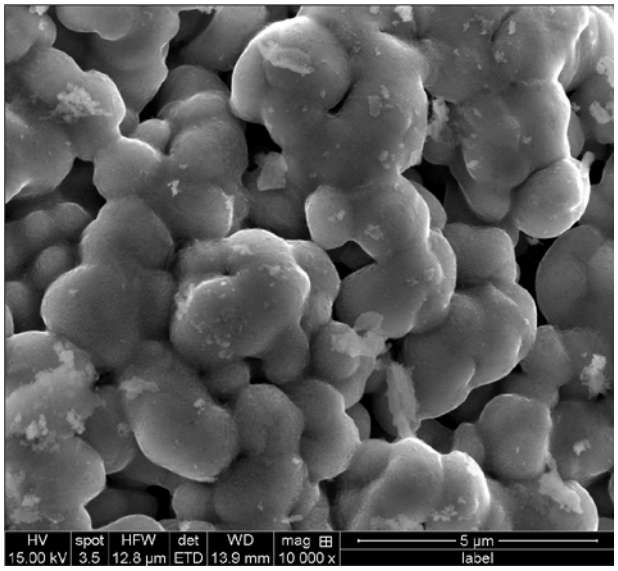


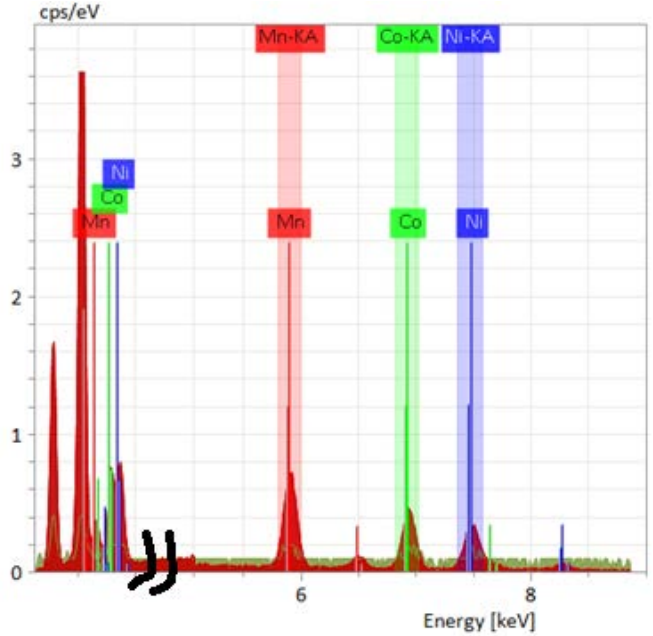
Photos for demonstration purpose only, not necessarily reflecting full experimental set-up.

Q1 Results 4 – Powder Morphology

$Li(Ni_{1/3}Mn_{1/3}Co_{1/3})O_2$ has been produced in the flame spray process using DES of the corresponding precursors. NMC are detected in the material from energy dispersive spectroscopy (EDS).



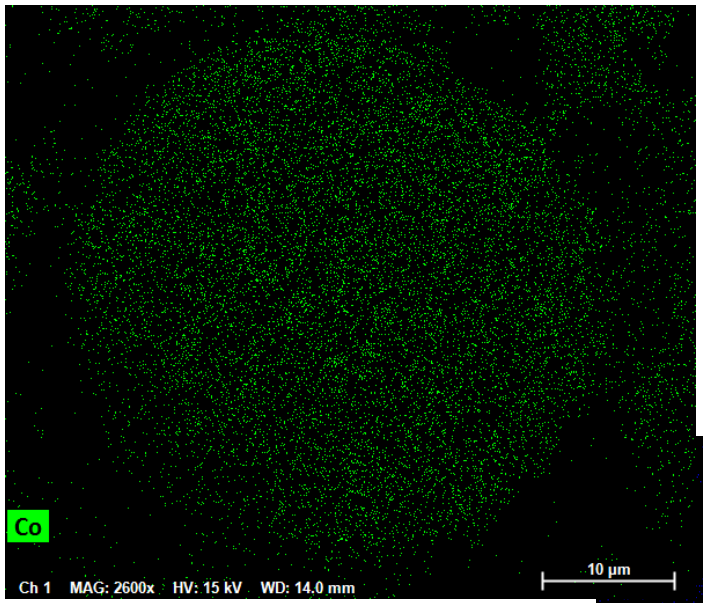


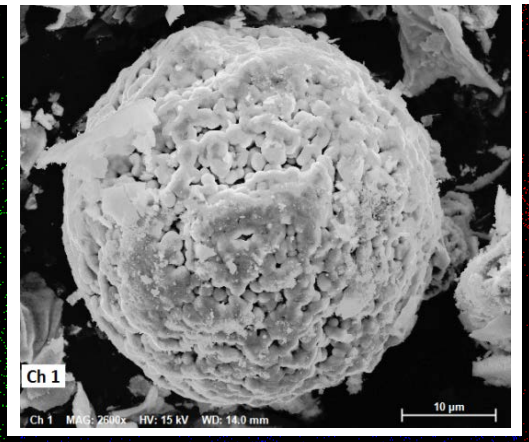


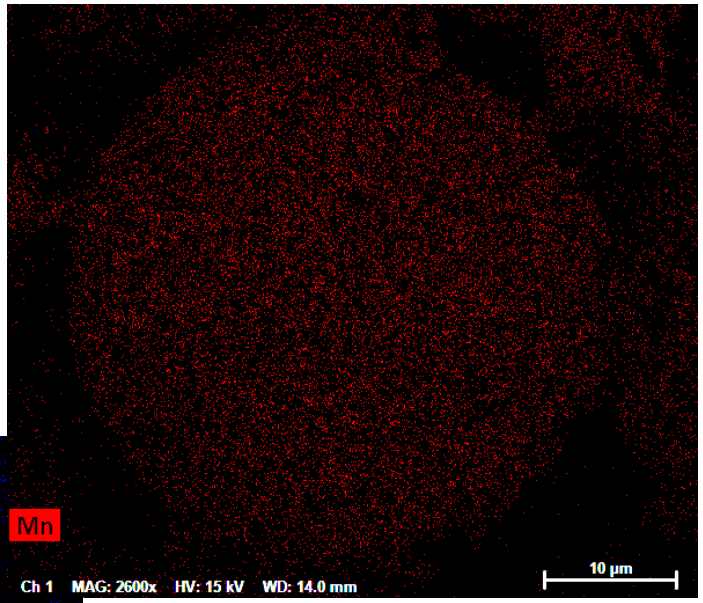
The NMC powders show a wide range of morphologies, including spheres consisting of secondary particles.

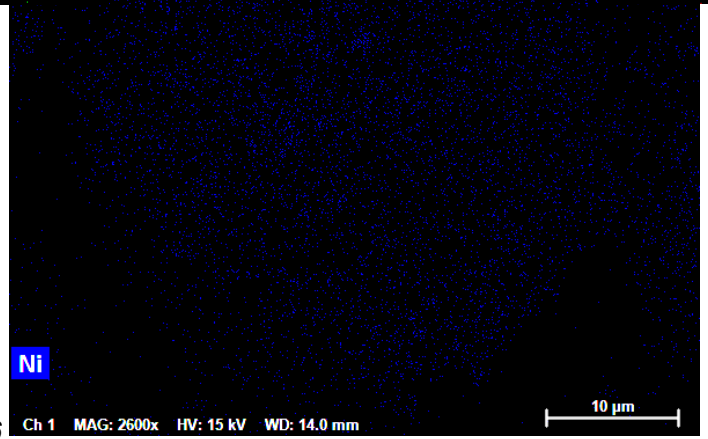
Q1 Results 5 – Elemental Distribution

Elemental distributions in the powder have been detected and found uniform throughout the powder.









Technical Challenges and Barriers

1. **Since this is a new project, the work undergoing in Q1 is in a "test" mode. As we proceed further into the work, we may encounter some technical challenges.**
2. **The current challenge is to make powders with controllable size with desired morphologies (e.g., spheres).**

Future Work (year 1)

1. Continue on obtaining optimal DES precursors for making stoichiometric compositions of the NMC powders.
2. Continue on improving nozzle design and operation conditions for spraying the DESs.
3. Continue on improving flame reactor design.
4. Continue on studying the factors affecting the powder morphologies.
5. Start on cell testing and design using the produced powders.

Collaborations

1. EaglePicher Technologies as a partner on the project will begin work from the 3rd quarter (starting July 2016).
2. In talks with Argonne National Lab and other industries in terms of future collaborations.

Summary

1. This review presented an overview of work and the work completed in the first quarter.
2. During this first quarter we have completed initial studies in DES precursor formulation and measured their properties (e.g., viscosity).
3. We have had an initial design of our flame spray process, and made a few tests on it.
4. Preliminary results showed that we can produce NMC powders from the DES precursors. It was shown that Ni, Mn and Co distribute uniformly within the powders without segregation.
5. We had initial contacts with other potential partners.

End of Presentation

Thank You!