Process Development and Scale Up of Advanced Electrolyte Materials

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Overview

- **Timeline**
  - Project start date: Oct. 2010
  - Project end date: Sept. 2014

- **Budget**
  - Funding received in FY11: $450K + internal funds
  - Funding received in FY12: $1.0M

- **Barriers**
  - Barriers addressed
    - Cost – Reduce cost to manufacture materials
    - Performance – Optimize process for highest purity for maximum performance

- **Partners**
  - Argonne’s EES Applied R&D Group
  - Argonne’s EES Materials Screening Group
  - US Army Research Laboratory
  - We are open to work with any ABR partner
Objectives and Relevance of this Program

- The objective of this program is to develop scalable processes for manufacturing electrolyte materials, synthesize kilogram quantities of each material and make them available for industrial evaluation:
  - Identify, rank and prioritize electrolyte materials of interest.
  - Resolve commercialization constraints by developing cost-effective and safe manufacturing processes.
  - Validate electrochemical performance of the scale-up materials.
  - Provide sufficient quantities of these materials for evaluation by industry.
  - Prepare process technology transfer packages.

- The relevance of this program to the DOE Vehicle Technologies Program is:
  - This program is a key missing link between discovery of advanced battery materials, market evaluation of these materials and high-volume manufacturing.
  - This program will provide quantities of materials with consistent quality for further validation in large format prototype cells.
  - This program will provide the basis for meeting broader industrial needs to reduce the risk associated with developing and maintaining a domestic commercially viable battery manufacturing capability.
Approach

- Identify candidate electrolyte materials from ABR program participants
  - Electrolyte solvents
  - Redox shuttle
  - Passivation additives

- Develop and maintain database of the materials
  - Source of the material
  - Chemical identity
  - Performance characteristic

- Develop rating criteria, rate and prioritize candidates for scale-up
  - Electrochemical performance
  - Manufacturing process complexity
  - Market needs
Approach

- Develop experimental work planning and control (EWPC) procedure
- Explore various chemical pathways and determine scale-up feasibility
- Proof-of-concept in a small-scale synthesis (10 g)
- First-stage scale-up and product quality verification, electrochemical performance validation (100 g)
- Second-stage scale-up and electrochemical performance validation (kilogram scale synthesis)
- Create Technology Transfer Package
- Make the material available for industrial evaluation
- Project reporting
## Approach - Milestones

### FY11
- 2-4 scale-up processes to be developed
- 4 materials completed
- 1 material considered not suitable for scale-up

### FY12
- 2-4 scale-up processed to be developed
- 3 materials completed (as of 3/22/12)
- Decommission interim laboratory, relocate to the Materials Engineering Research Facility (MERF)

### FY13
- 4-6 scale-up processes to be developed

<table>
<thead>
<tr>
<th>MILESTONE</th>
<th>DATE</th>
<th>STATUS</th>
<th>COMMENTS</th>
</tr>
</thead>
<tbody>
<tr>
<td>ANL-R52 - Scale up work completed</td>
<td>9/10/2010</td>
<td>Completed</td>
<td>Kilogram quantities available</td>
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<tr>
<td>Select CSE materials to scale</td>
<td>10/1/2010</td>
<td>Completed</td>
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<tr>
<td>Assess scalability of CSE process</td>
<td>11/1/2010</td>
<td>Completed</td>
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<tr>
<td>WP&amp;C documentation approved</td>
<td>11/1/2010</td>
<td>Completed</td>
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<tr>
<td>Develop and validate scalable process chemistry (10g bench scale)</td>
<td>12/1/2010</td>
<td>Completed</td>
<td></td>
</tr>
<tr>
<td>First process scale-up (100g bench scale)</td>
<td>12/23/2010</td>
<td>Completed</td>
<td></td>
</tr>
<tr>
<td>Second process scale-up (1000g pilot scale)</td>
<td>03/04/2011</td>
<td>Completed</td>
<td>1.576 g produced in a single batch, purity &gt; 99.9%.</td>
</tr>
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</table>

| 1NMS - Scale up work completed | 11/1/2010 | Completed | Kilogram quantities available |
| Select CSE materials to scale | 11/1/2010 | Completed |                      |
| Assess scalability of CSE process | 11/1/2010 | Completed |                      |
| WP&C documentation approved | 12/1/2010 | Completed |                      |
| Develop and validate scalable process chemistry (10g bench scale) | 02/11/2011 | Completed |                      |
| First process scale-up (100g bench scale) | 03/11/2011 | Completed | 130 g produced in a single batch, purity > 99.9%. Single fractional distillation delivered high purity product. |
| Second process scale-up (1000g pilot scale) | 05/11/2011 | Completed | 3.36 kg, produced in a single batch, purity > 99.9%. |

| 2S40 | Kilogram quantities available |
| Select CSE materials to scale | 01/07/2011 | Completed |                      |
| Assess scalability of CSE process | 01/14/2011 | Completed |                      |
| WP&C documentation approved | 02/22/2011 | Completed |                      |
| Develop and validate scalable process chemistry (10g bench scale) | 04/28/2011 | Completed |                      |
| First process scale-up (100g bench scale) | 05/26/2011 | Completed | 05/26/2011, 130 g produced in a 1L scale reaction. |
| Second process scale-up (1000g pilot scale) | 06/24/2011 | Completed | 1.477 g of material was made in a single batch. The purity of the material is > 99.9%. |

| ANL-R56 | Kilogram quantities available |
| Select CSE materials to scale | 01/07/2011 | Completed |                      |
| Assess scalability of CSE process | 01/14/2011 | Completed |                      |
| WP&C documentation approved | 03/28/2011 | Completed |                      |
| Develop and validate scalable process chemistry (10g bench scale) | 05/06/2011 | Completed |                      |
| First process scale-up (100g bench scale) | 06/03/2011 | Completed | 05/26/2011, 130 g produced in a 1L scale reaction. |
| Second process scale-up (1000g pilot scale) | 07/25/2011 | Completed | 1.815 g was produced in a single batch. The purity of the material is > 99.9%. |
Approach - Deliverables

- For each electrolyte material selected we will:
  - Develop a scalable manufacturing process.
  - Develop analytical methods and quality control procedures.
  - Prepare a “technology transfer package” which will include:
    - Summary of the original process used by discovery researchers to synthesize the material.
    - Summary of the scalable (revised) process suitable for large scale manufacturing.
    - Detailed procedure of the revised process for material synthesis.
    - Analytical data/Certificate of Analysis for the material (chemical identity and purity).
    - The material impurity profile.
    - Electrochemical performance test data.
    - Preliminary estimates of production cost.
    - MSDS for the material.
  - Make kilogram quantities of the material available for industrial evaluation.
    - The material will be fully characterized chemically and electrochemically
Technical Accomplishments and Progress Overview

- Scalable processes were developed and kilogram quantities of materials were synthesized for 5 electrolyte materials.
  - ANL-1NM3
  - ANL-2SM3
  - ANL-1S1M3
  - ARL-HFiPP
  - ANL-RS6

- Technology transfer packages were created.

- Materials were sampled for evaluation.
Technical Accomplishments and Progress
Redox Shuttle ANL-RS2

- Scale-up process developed and reported at previous AMR meeting.
- Technology Transfer Package was created.
- Process technology patent filing is in progress.
- Samples of the material were supplied for evaluation to researchers and industry.
Technical Accomplishments and Progress
Redox Shuttle ANL-RS6

- New chemistry was developed for the process.
- Multi-step synthesis was simplified to a single reaction.
- The reaction is carried out using “green” solvents.
- The material is purified to the required grade by simple crystallization.
- The amount of waste generated was reduced.
- Overall yield of the process was improved.

- The scaled-up material was validated and matched the electrochemical properties of the original material from discovery.

- Technology Transfer Package was created for the process.

- Sample of the material was provided to scientist for further research and to industry for evaluation.
Technical Accomplishments and Progress
Solvent ANL-1NM3

• All solvents were eliminated from the process (solvent-free process).
• The amount of waste generated was minimized.
• Used of a catalyst allowed lower temperature and shorter reaction time (lower cost and use of energy).
• Overall yield and purity of the material was improved.

• The scaled-up material was validated and matched the electrochemical properties of the original material from discovery lab.
• Technology Transfer Package was created for the process.
• Sample of the material was provided to scientist for further research and to industry for evaluation.
Technical Accomplishments and Progress
Solvent ANL-2SM3

• The developed process is solvent-free.
• Used of an advanced catalyst allowed better utilization of feedstock and minimized by-product formation.
• The catalyst is fully recoverable and recyclable.
• The amount of waste generated was minimized.
• Overall yield of the process was improved.

• Validation of the electrochemical performance of the material is in progress.
• Technology Transfer Package was created for the process.
• Sample of the material was provided to scientist for further research.
Technical Accomplishments and Progress
Solvent ANL-1S1M3

- Complex process (four subsequent chemical transformations).
- Chlorinated solvents were eliminated from the process.
- Highly flammable ethers were replaced with less hazardous solvents.
- Pyrophoric butyllithium was eliminated from the process.
- Purification of intermediate materials were eliminated.
- The amount of waste generated was reduces.

- Validation of the electrochemical performance of the material is in progress.

- Technology Transfer Package compilation is in progress.
Technical Accomplishments and Progress
Passivation Additive ARL-HFiPP

• Scale-up process developed in cooperation with ARL
• Optimizing the reagents ratio and reaction condition resulted in improved yield and quality of the product.
• The material is purified to the required grade by simple, one-step distillation (originally double distillation followed vacuum sublimation).
• Hazardous diethyl ether was replaced with a less flammable solvent.
• Overall yield of the process was improved.

• Validation of the electrochemical performance of the material is in progress.
• Technology Transfer Package was created for the process.
• Sample of the material was provided to scientist for further research.
Collaborations

- Argonne’s Electrochemical Energy Storage, Applied R&D group
  - Electrolyte solvents and redox shuttle were scaled

- US Army Research Laboratory
  - Passivation additive was scaled

- Argonne’s Electrochemical Energy Storage, Material Screening group
  - Conducted electrochemical validation of scaled materials

- We invite all ABR program participants to submit new candidates to our advanced electrolyte materials process R&D program.
Activities for Next Fiscal Year

- Manage electrolyte materials database and populate with new candidates from other institutions in the ABR program.
  - Rank and prioritize the materials.

- Develop scalable process for manufacturing 4-6 electrolyte materials from discovery to bulk quantities.
  - Develop scalable process, analytical methods and quality control procedures.
  - Validate the manufacturing process, quality of the materials and their electrochemical properties.
  - Create Technology Transfer Package.
  - Supply material samples to researchers and industry for further evaluation.
Activities for Next Fiscal Year
The Materials Engineering Research Facility (MERF)

- 10,000 sq. ft. facility consists of “Group-H occupancy” pilot labs and high bay spaces.
- All interim labs will be relocated to the MERF, starting in April.
- 50% DOE – 50% DoD funded
Summary

- The procedures used to make small, research sample of materials are not suitable for large scale production. Manufacturing processes for the newly discovered advanced materials must be scalable to facilitate the transition from basic research to commercial application.

- This program has been developed to provide a systematic approach to process R&D and scale-up, and to provide sufficient quantities of advanced electrolyte materials for industrial evaluation.

- Argonne’s process R&D program enables industry to carry out large-scale testing of new electrolyte materials.

- Integration of materials discovery with process R&D will expedite the time needed to commercial deployment.

- Processes for 5 electrolyte materials were successfully developed. The materials were manufactured at the kilogram scale.

- Samples of the material were provided to various entities for industrial evaluation.
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