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VG-2022-288

Efficient Process for the Production of High Conductivity, Carbon-rich Materials from Coal

DE-SC0018837 (PSI-8040)

Presentation for the

2022 Resource Sustainability Annual Project Review Meeting

October 25-27, 2022

DoE Program Manager – Dr. Brett Hakey

PI – Dr. Dorin V. Preda

Acknowledgment: This material is based upon work supported by the U.S. Department of Energy, Office of Science, Office of Chicago Ops, under Award Number DE-SC0018837.

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Programmatic Information

- **Topic: # 18. ADVANCED MANUFACTURING & MATERIALS FOR FOSSIL ENERGY TECHNOLOGIES**
  - **Subtopic c. High Value Products from Coal**

• **Period of Performance:**	Phase IIA: 8/26/2021 – 8/26/2023
• **Funding:**	$1.1M
• **Technical Monitor:**	Brett Hakey (current), Charles Miller (retired)
• **PI:**	Dorin Preda
• **Team Members:**	Sharon Song, Nathan Shipley, Caitlin Bien, Gabrielle Aversa, Rachel Vozikis
• **Technical Reviewer:**	Christopher Lang
• **Consultants:**	Professor Larry Scott (BC, Emeritus)
	Dr. Joe DiCarlo

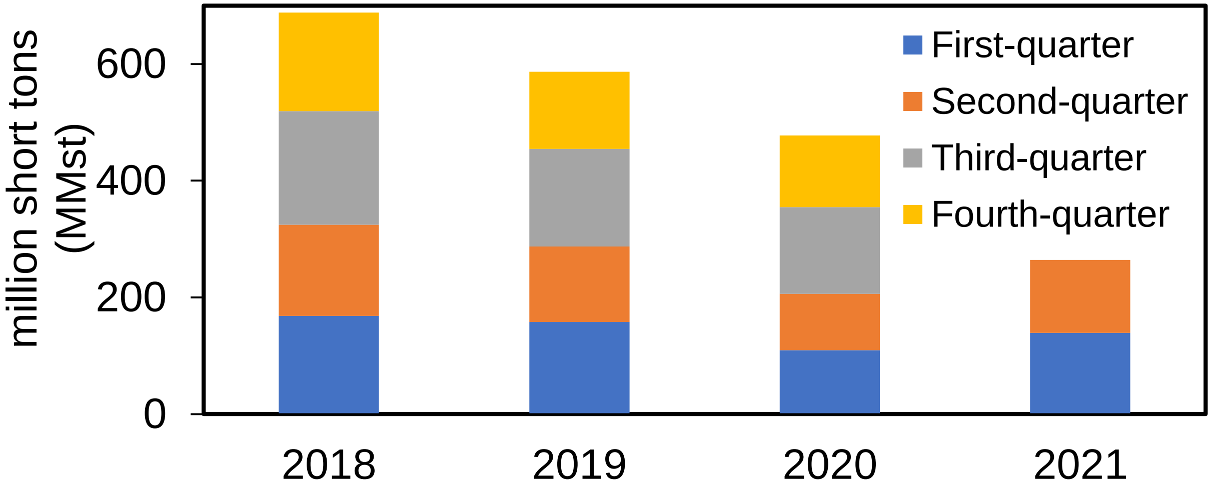
DOE Solicitation

- **Domestic coal can be used to manufacture solid carbon products** including carbon fiber, **battery/electrode materials**, carbon-based photovoltaics, carbon materials for 3D printing applications, carbon-based construction materials, and carbon additives for manufacturing composites. **The market value of these high-performance materials often exceeds the fuel and heat value of coal, which illustrates there are sustainable market forces for manufacturing carbon materials from coal.**

- Proposals are sought that focus on using domestic coal and closely related by-products (pitch, char) as a manufacturing feedstock to produce carbon materials and composites. Utilizing coal for producing carbon materials creates new business opportunities by integrating coal into the value-chain of industries that typically do not use coal in their manufacturing processes. Coal-based carbon fiber and carbon fiber reinforced polymers offer opportunities for producing new forms of lightweight structural materials and composites which have utility in automotive and aerospace applications. Coal-based carbon nanomaterials, such as graphene and carbon quantum dots, offer opportunities to bring down the costs of these materials so they can be used in electronic display screens, pigments/dyes/coatings, enhanced textiles, and structural composites. Inexpensive carbon nanomaterials can also be used in 3D printing fluids/plastics to enhance the electrical/thermal/optical properties of the final printed material. Coal-based coke, pitch, and carbon nanomaterials can be used to produce **electrode materials** for a range of applications such as aluminum production, batteries/energy storage, and supercapacitors.

Background: Coal Consumption

- Coal can be used to manufacture high value carbon products for multiple applications. The market value of these high performance materials often exceeds the fuel and heat value of coal, illustrating sustainable market forces for manufacturing carbon materials from coal.
- Current processes to produce high performance carbon materials from coal requires significant chemical modifications of the native coal structures (e.g., high temperatures and corrosive reagents).
- U.S. coal consumption totaled **124.6 million short tons** (MMst) in second-quarter of 2021. This is 29.1% higher than the 96.5 MMst reported in second-quarter of 2020.
- Electric power sector accounted for about **91.3%** of the total U.S. coal consumption in the second-quarter of 2021.



https://www.eia.gov/coal/production/quarterly/

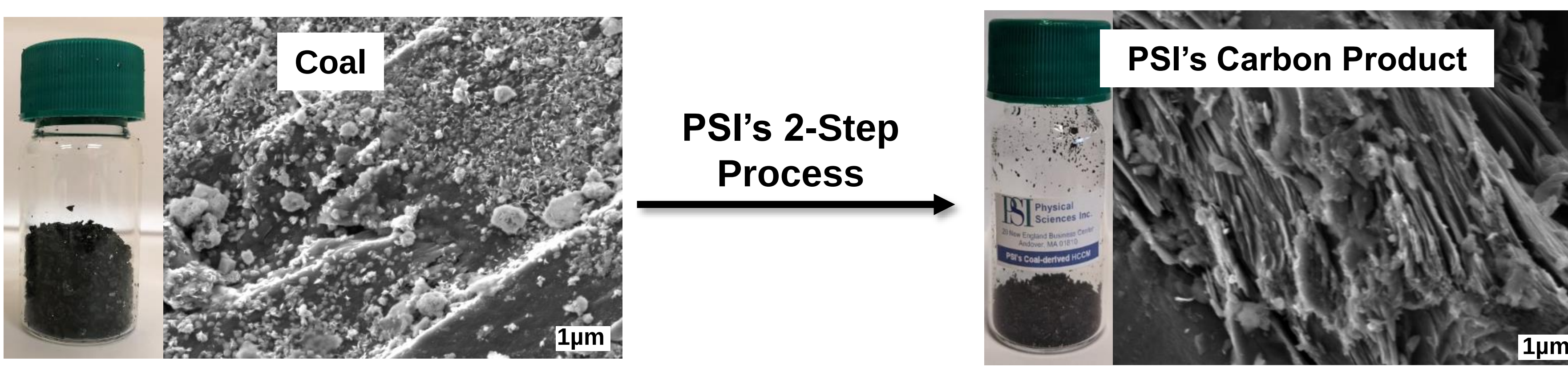
Need to increase the usage of domestic coal to manufacture value added carbon-based products

Executive Summary

- PSI successfully developed an innovative technology for the production of high value, high performance carbon materials from coal feedstocks for electrochemical applications.

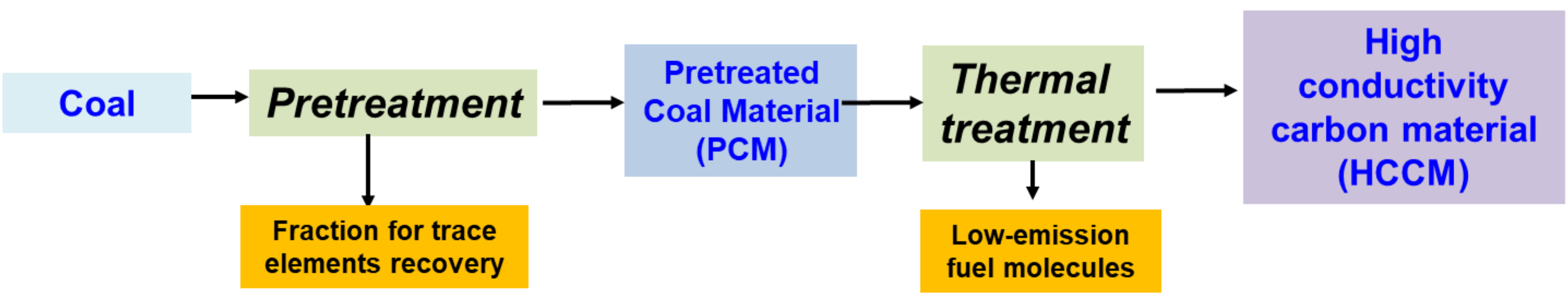
- During the Phase I and initial Phase II Program, PSI demonstrated: (1) feasibility and scalability of the process; (2) key elements of the scale-up processes; (3) initial market competitiveness of PSI's carbon product against commercial carbon products for battery electrode applications and (4) initial economic viability of commercialization.

- **PSI is currently executing on the Phase IIA program to complete and mature the technology developed in Phase I and the initial Phase II programs.**



Technology Overview

- PSI's technology is a novel approach that produces high value, high performance carbon materials from coal for electrochemical applications under mild conditions.
- 1st step: Pretreatment that produces the intermediate PCM.
  - Minimal reagent usage, efficient recycling process, and amenable to multiple coal feedstocks.
  - Valuable byproduct (mineral fraction).
- 2nd step: Thermal treatment that produces the target HCCM.
  - Carbon product for electrochemical applications.
  - Valuable byproduct (low emission gaseous fuels).



Capability to be Provided

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Objective/Description: Develop, optimize, and scale-up innovative process for producing high value, carbon-based products (HCCMs) from coal feedstocks for electrochemical applications.

- **Phase I:** PSI successfully demonstrated the feasibility of the proposed approach. Domestic coal feedstocks were screened and selected, scalable processes were demonstrated to produce the high conductivity material, and high performance battery electrodes were produced with the material. A techno-economic analysis was performed to outline pathways for scale-up and further development in Phase II.
- **Phase II:** PSI successfully demonstrated scale-up production of the high conductivity carbon material, demonstrated the performance in battery electrode formulations, and developed a transition and scale-up plan for profitable conversion of coal to high conductivity carbon on the multi-ton scale.
- **Phase IIA (Current):** PSI will demonstrate: (1) continuation of the initial Phase II work to further demonstrate the HCCM/PCM scale-up processes and battery electrode applications, (2) enhanced objectives with respect to the scale-up of low cost processes for transition to Phase III, and (3) work with transition partner to develop a robust strategy for technology transition into commercialization. **The overall goal of the Phase IIA program is to complete and mature the technology developed in the initial Phase II program (demonstrated key elements of the scaled-up processes and techno-economic model).**

Phase I Summary

Under the Phase I program, PSI successfully demonstrated the capability to produce HCCM from coal using an economical, simple, two-step process and initial electrochemical applications.

- Anthracitic and bituminous coals were suitable as feedstocks for the PSI process.
- Coal pretreatment process with high yields resulting in a pretreated coal material (PCM) suitable for the production of HCCMs.
- Robust process conditions to produce HCCMs.
  - HCCMs with the required properties for use in electrochemical applications: (1) Low mineral content: Fe < 100 ppm; (2) High surface area: > 50 m^2/g
- Production of gaseous by-products that can be used as low emission fuels.
- Processes for the production of battery electrode formulations with HCCM.
  - High performance electrodes could be produced with the performance necessary for integration in state of the art batteries.
- The design of an economically viable scale-up production process.

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Phase II Summary

Under the Phase II program, PSI scaled-up the capability to produce HCCMs from domestic coal sources. These results provide the basis for the Phase IIA program to complete the work of the Phase II program with enhanced objectives relative to the scale-up parameters of the production of HCCM and increased fidelity of the techno-economic model. During the Phase II program, PSI demonstrated:

- Scalable PCM production processes using:
  - Soxhlet Reactor at 0.15 kg/batch
  - Micro-pilot reactor at 1 kg/batch
- Scalable HCCM production process with a capacity of 0.5 kg/batch.
- Suitable HCCM properties for electrochemical applications including:
  - Layered, sheet-platelet-like morphology (SEM)
  - High purity (99+% on metals basis, ICP-OES)
  - Tailorable surface area (10 – 300 m^2/g, BET)
- High electrochemical performance in battery pouch cells.
- Economic viability through initial techno-economic analysis (e.g., sensitivity analysis)

Phase II Key Results : Scalable PCM Production

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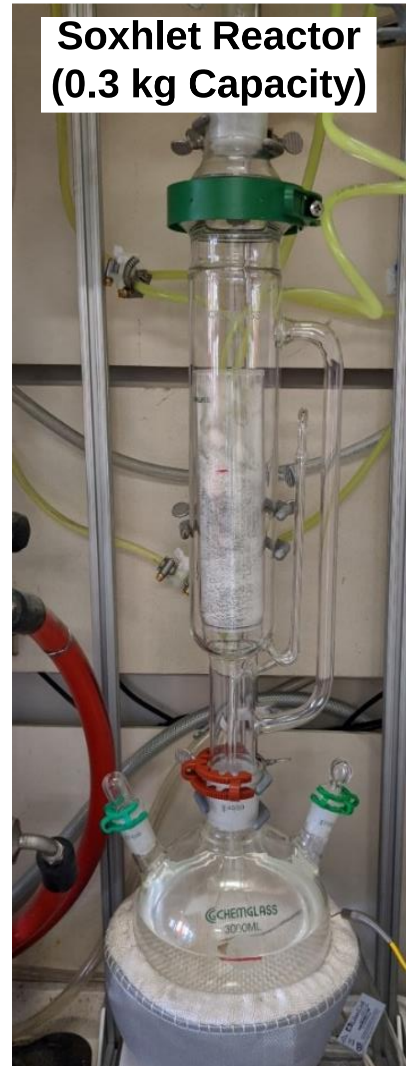
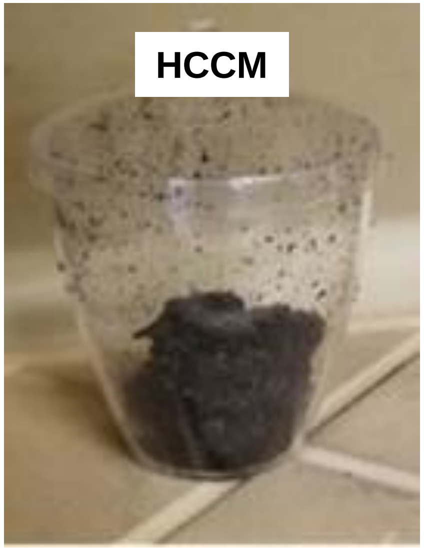
- **Demonstrated significant increase in the coal pretreatment process scale.**
  - Soxhlet Reactor at 0.15 kg/batch
  - Micro-pilot (MP) Reactor at 1 kg/batch
- Validated the Soxhlet and MP Reactors for scalable PCM production processes.
- Demonstrated high yields using the Soxhlet reactor (>75% wt.).

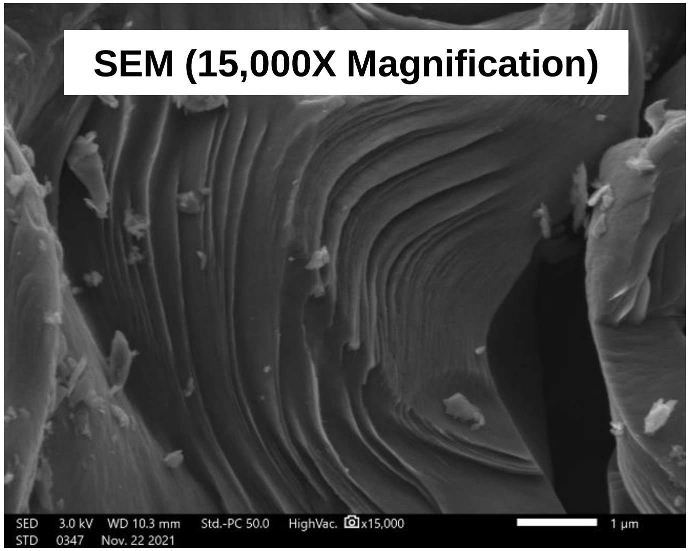
PSI's Coal-derived PCM (1 kg)



Phase II Key Results : Scalable HCCM Production

- **Demonstrated multiple HCCM products with suitable properties for electrochemical applications:**
  - SEM: Layered, sheet-platelet morphology (SEM)
  - ICP-OES: High purity (99%+ metal basis)
  - Gas Adsorption: Tailorable surface area (10 – 300 m^2/g)
- **Demonstrated scalable process conditions to produce HCCM (0.25 kg).**
  - Optimized configurations in a muffle furnace (0.5 kg capacity)
  - Similar characteristics as that of HCCMs produced in the Phase I program.





Phase II Key Results : Electrochemical Testing

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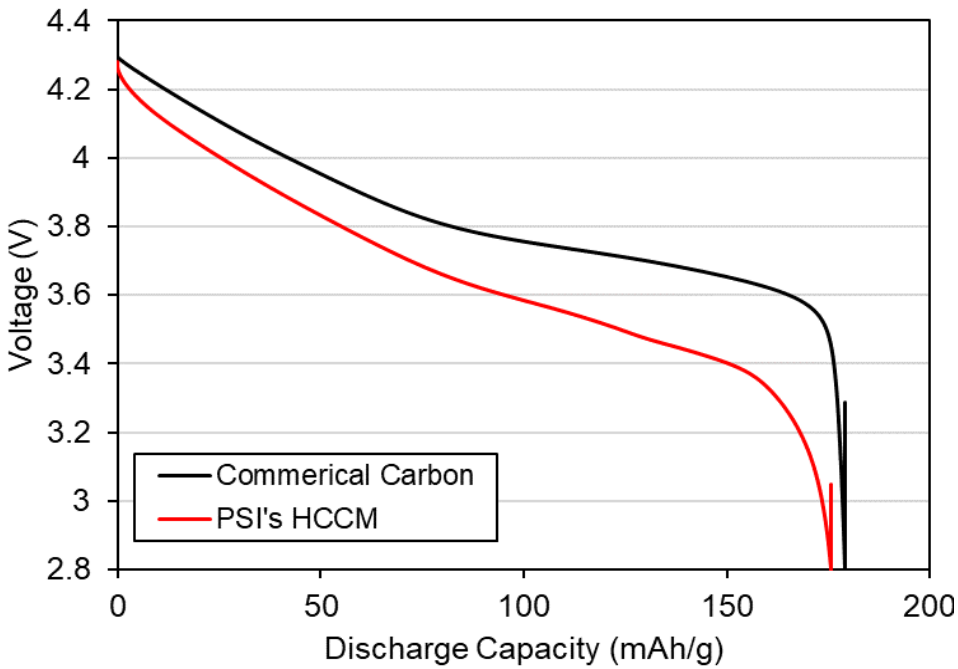
- **Results indicated potential market competitiveness of PSI’s carbon product against benchmark carbon products for battery electrode applications.**
- Successfully demonstrated HCCM formulation into battery pouch cells using industry established procedures.
- Results indicated high electrochemical performance – comparable to that of benchmark carbon products.



Phase II Key Results : Techno-economic Analysis

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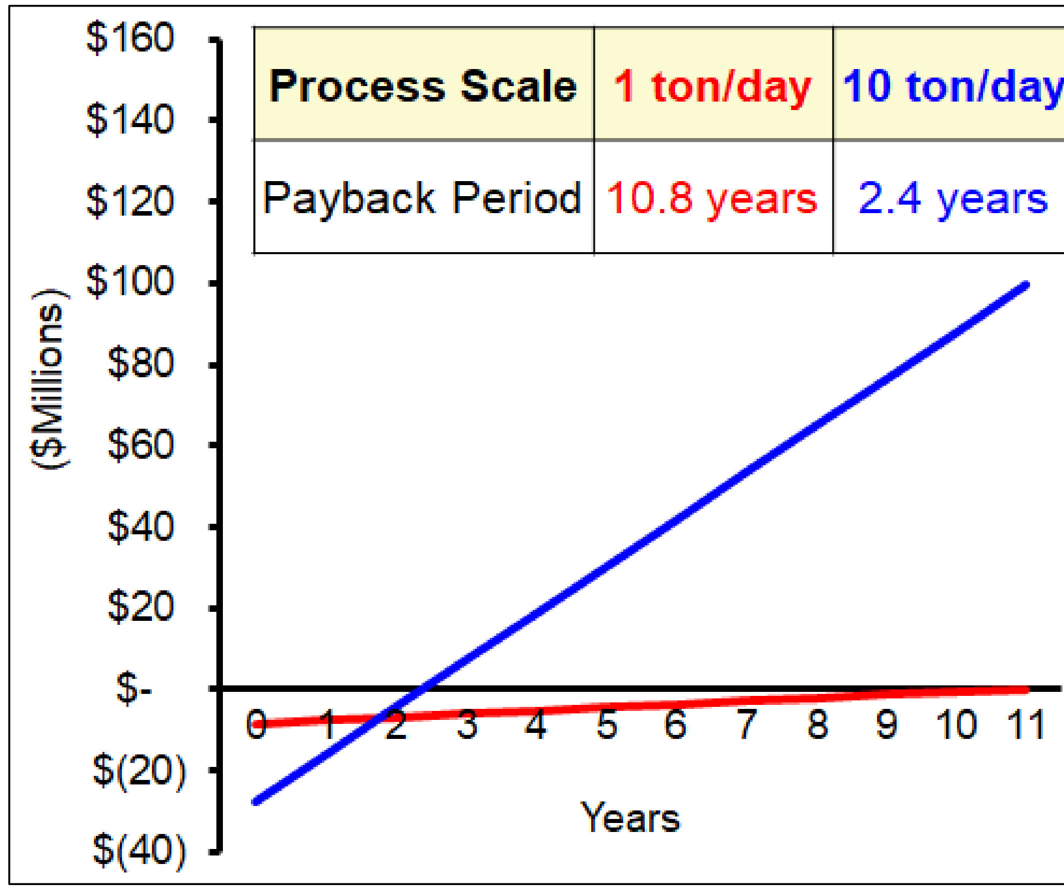
- PSI team performed a preliminary techno-economic analysis and results indicated **profitable conversion of coal to HCCM** and **economic viability of the scale-up commercial operation**.
- Preliminary cost analysis indicated that PSI's HCCM is:
  - **2X lower** in cost compared to a benchmark product for battery application.
  - **>20X higher** in value than coal.
- Sensitivity analysis results indicated the important of the process scale for profitable commercialization.

Sensitivity Analysis : Process Scale

Process Scale	1 ton/day	10 ton/day
Payback Period	10.8 years	2.4 years



Phase IIA Technical Objectives

The overall goal of the Phase IIA program is to complete and mature the technology developed in the initial Phase II Base program, which demonstrated key elements of the scaled-up processes and techno-economic model.

1. Demonstrate scaled-up coal pretreatment processes that produce >2kg per week of pretreated coal materials (PCMs) with high yields (>90% g/g basis) and high purity (99.9+% on metals basis).
2. Demonstrate incremental scale-up conversion processes (kg to tens-of-kg per batch) to produce HCCM products (>10% yield).
3. Demonstrate equivalent or higher electrochemical performance of the HCCM products compared to the products generated in the initial Phase II.
4. Demonstrate blends of PCM with commercial precursors that produce carbon products with suitable electrochemical and structural properties for electrochemical and other applications.
5. Further develop and refine the transition and scale-up plans from the initial Phase II for profitable conversion of coal to HCCM on the multi-ton scale.

Phase IIA – Results to Date and Next Steps

Phase IIA Work Plan: Task Overview

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- **Task 1 – Scale-up PCM Production:** Further optimize the scalable coal pretreatment processes initially developed in the Phase II Base program to produce PCM for the process in Task 2.
- **Task 2 – Scale-up HCCM Production:** Further optimize the scalable process initially developed in the Phase II Base program to produce high purity HCCM products from PCM (Task 1).
- **Task 3 – Electrochemical Performance Validation:** Evaluate the HCCM products (Task 2) for electrochemical applications using established electrochemical testing refined in the Phase II Base program, in addition to scaled-up evaluation.
- **Task 4 – Mixed Precursor Evaluation:** Produce a mixed blend of commercial precursor product and PCM (Task 1). The blend will be evaluated for electrochemical performance (Task 3) and structural properties (Task 2).
- **Task 5 – Techno-economic Analysis for Phase III and Commercialization:** Perform a detailed techno-economic analysis and develop process scale-up/manufacturing plans for the transition into Phase III and commercialization.
- **Task 6 – Management and Reporting**

Task 1 – Scale-up of PCM Production

- **Goal:** Demonstrate scale-up coal pretreatment processes that produce >2 kg of PCM per week with high yields (>90% g/g basis) and high purity (99.9+% metal basis) using Soxhlet and Micro-pilot reactors.

- **Status:**
  - Initiated the synergistic operations of the Soxhlet and the MP reactors for enhanced scale-up PCM production (Next Slide).
    - On-going optimization and refinement of the scale-up processes
  - Production and validation of PCMs using scale-up processes.
    - Successfully demonstrated scale-up efforts on a 2 kg process scale
    - Validated the scale-up processes and corresponding PCM/HCCM materials

- **Next Steps:**
  - Demonstrate high yielding (>90% wt.) PCM production using a combination of Soxhlet and MP reactors.
  - Validate the scaled-up PCM products and demonstrate suitability for HCCM production.

PSI's PCM Scale-up Facility: Initial Design

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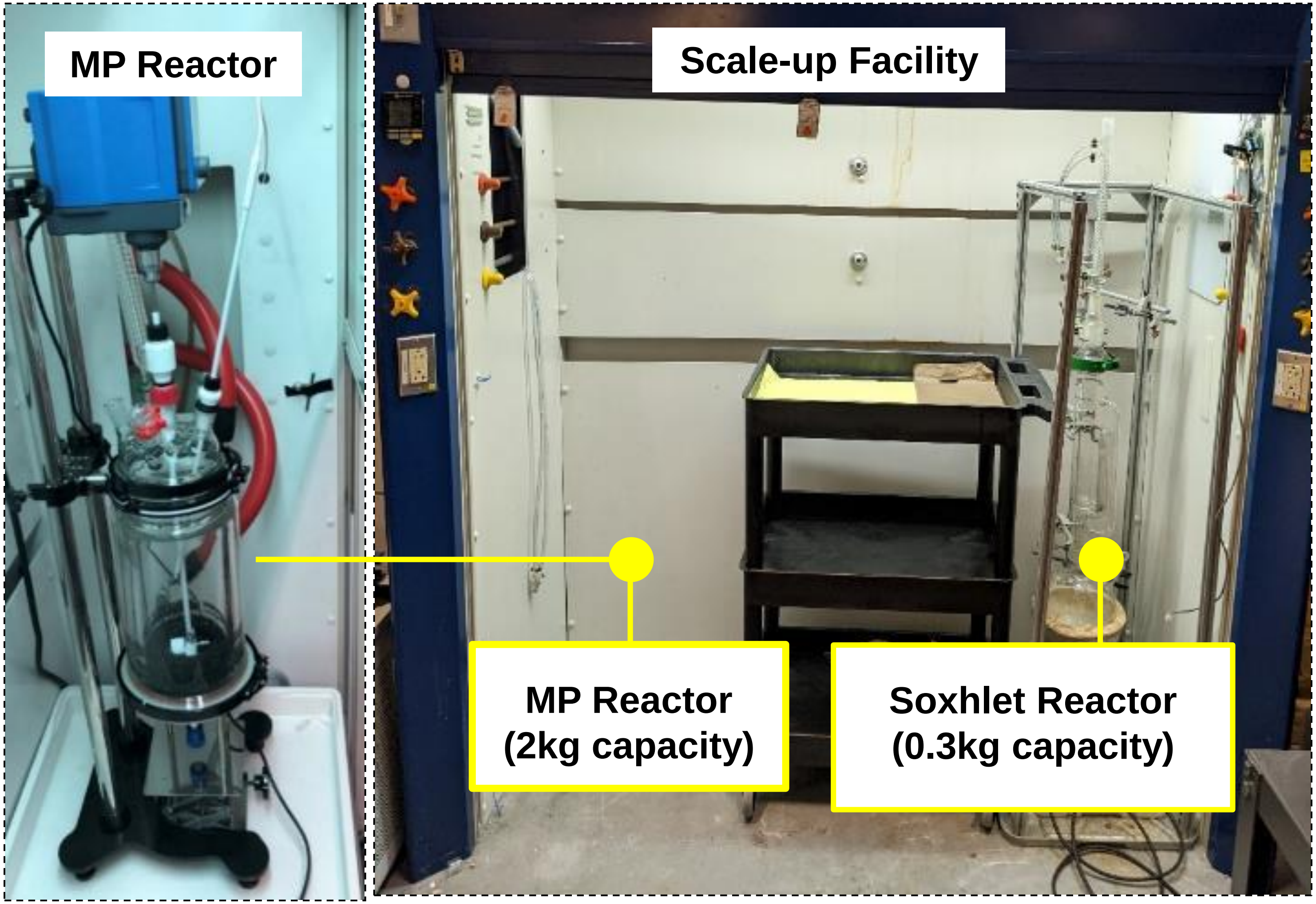

Provides the capability to simultaneously operate the MP and the Soxhlet Reactors

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PSI's PCM Scale-up Facility: Progression of the Scale-up Efforts

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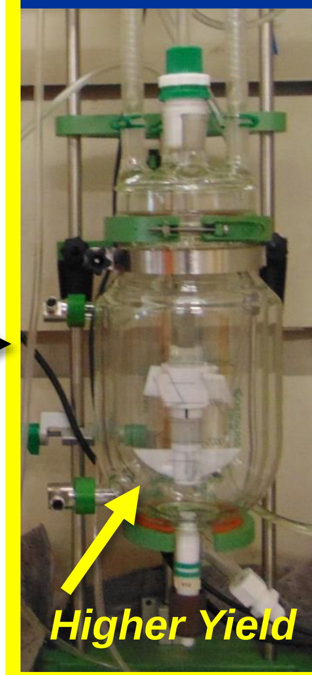
- **PSI successfully demonstrated production of PCM on kilogram process scale.**
  - Phase I: Small scale (1 – 50 g per batch) using benchtop set-up, RBF
  - Phase II: Intermediate scale (0.15 – 1kg per batch) using Soxhlet and MP Reactors
  - Phase IIA: Large scale (1 – 2kg per batch) using Micro-pilot reactor

Phase I : 1 – 50g

Phase II : 0.15kg

Phase II/A: 1kg

Phase IIA:→2 kg



Successful sequential increase of the process scale from grams to kilograms

Task 2 – Scale-up of HCCM Production

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- **Goal:** Use the scalable process initially developed in the Phase I program to produce HCCM products suitable for electrochemical application from Task 1 materials.

- **Status:** Refining the process conditions to produce HCCMs with high yields (>10% wt.) and high purity (>99+%).
  - Demonstrated a pathway towards high yields via TGA studies
  - Extensive characterization using PSI's enhanced analytical capabilities (Next Slide).

- **Next Steps:**
  - Finalize the refinement of the process parameters.
  - Transition refined process parameters to a industry partner for the scale-up HCCM production efforts (kg to tens-of-kg).
  - Perform comprehensive characterization of scaled-up HCCM product(s) to demonstrate suitability for electrochemical application.

PSI's In-house Analytical Capabilities

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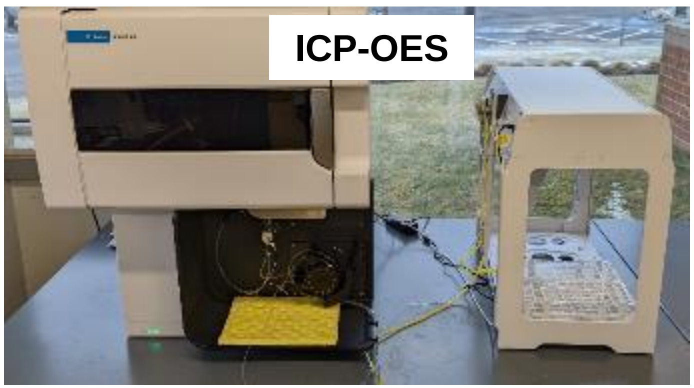
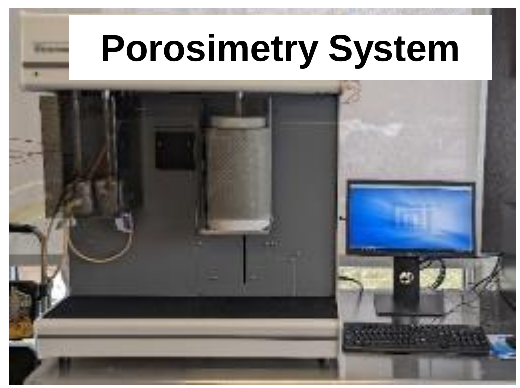
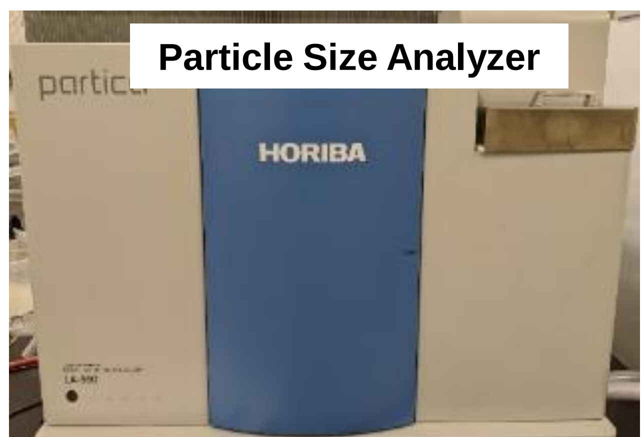
- **PSI recently procured multiple instruments that provide enhanced analytical capabilities including:** (a) Inductively coupled plasma atomic emission spectroscopy – ICP-OES, (b) Surface area/porosimetry system, (c) Scanning electron microscopy – energy dispersive X-ray spectroscopy – SEM-EDS, (d) Particle size analyzer, and (e) Thermogravimetric analysis (TGA) system.





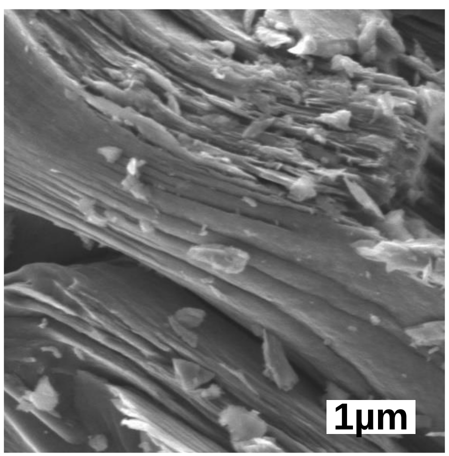
PSI's analytical lab also houses the following instruments:

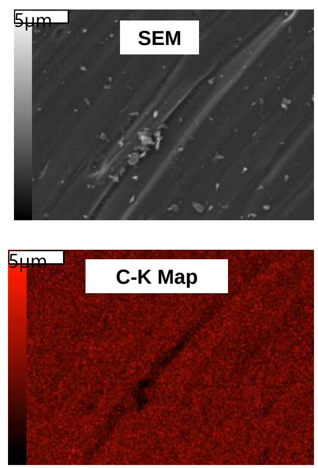
- NMR spectrometer
- Fluorometer
- UV-Vis Spectrometer

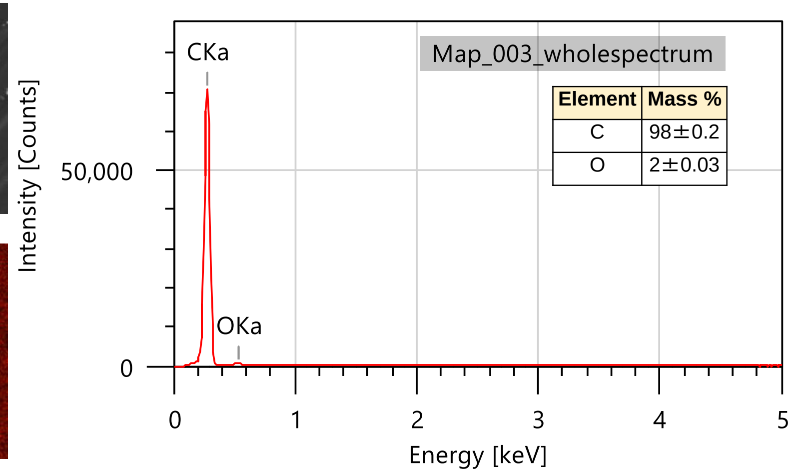


HCCM Characterization: Initial Runs

- PSI performed a comprehensive characterization of the HCCM produced from the 0.25kg scale-up process conditions. Combined results indicate suitable properties for electrochemical applications.
  - SEM: Platelet, sheet-like morphology.
  - EDS: High surface carbon content (>98%).
  - ICP-OES: Low trace metal content (>99.7%).
    - Developed pathways to reduce the trace metal content.
  - BET Surface Area: Low surface area (4.1 m^2/g).

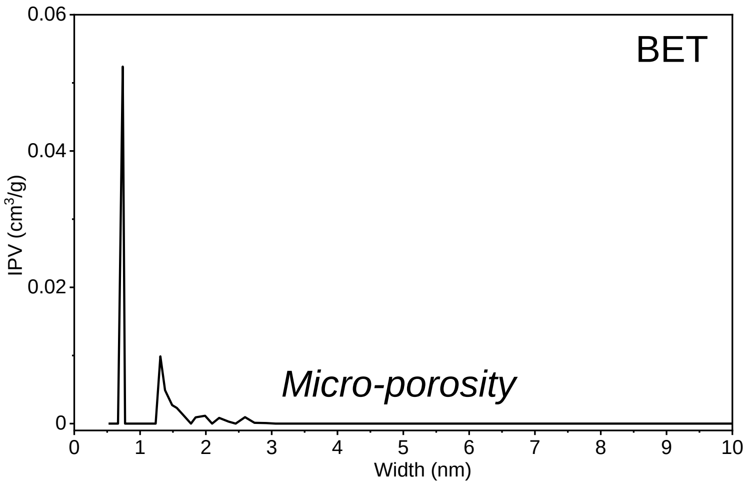


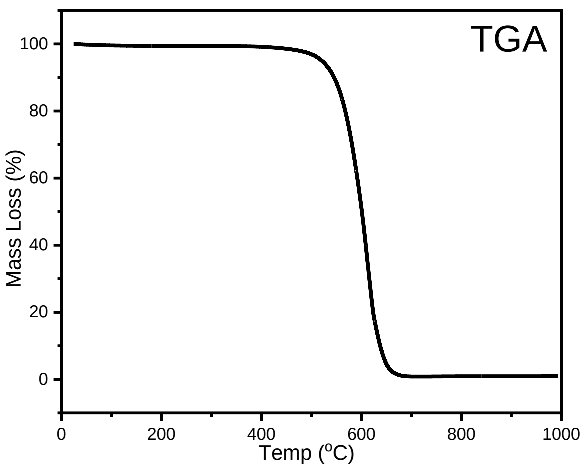


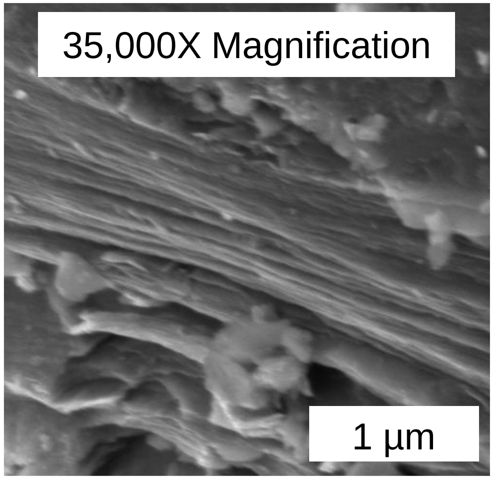


HCCM Characterization: Recent Results

- HCCMs analysis indicate similar properties to those of initial materials: process accommodates variation in the PCM feedstock and production methods
  - SEM: Platelet, sheet-like, layered morphology
  - EDS: High surface carbon content (as high as 98%)
  - ICP-OES: Low trace metal content (as low as 2,700 ppmw)
  - TGA: Low ash content (as low as 0.2%)
  - BET Surface Area: Tailorable (10 - 300 m^2/g)







HCCMs showed suitable properties for electrochemical applications (next slides)

Task 3 – Electrochemical Performance Validation

- **Goals:** Demonstrate equivalent or higher electrochemical performance of the scale-up HCCM (Task 2) compared to the products generated in the Phase II Base and demonstrate electrochemical application on a commercially relevant size.

- **On-going work:**
  - Perform pouch-cell testing of scaled-up HCCM (from Task 2) using the optimized Phase II electrochemical testing conditions.
  - Produce commercially relevant sized pouch cells.

PSI's 13,000 mAh Commercial Pouch Cell

Task 3 – Electrochemical Performance Validation: Preliminary Results

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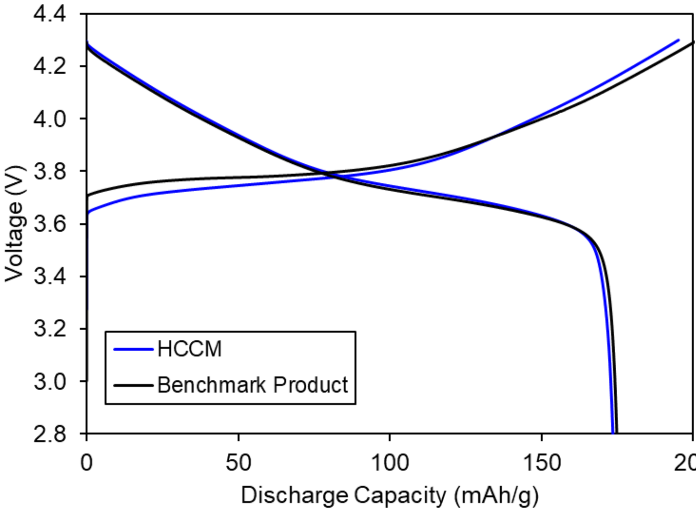
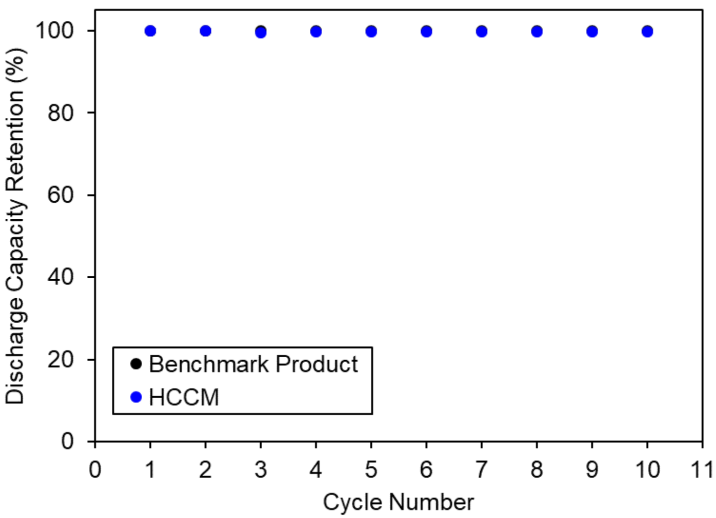
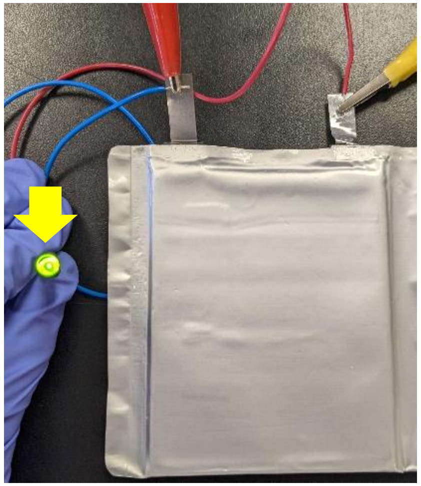
- Successfully produced 8,000 mAh sized pouch cells (30 Wh, 32 layers) with scaled-up HCCM using industry established protocols.

- Results showed repeatable, high electrochemical performance that is comparable to a benchmark carbon with specific level performance of 175 mAh/g.
  - TO Achieved: 99% of the SOA/Phase I/II results
  - Consistent cell to cell performance (St. dev <0.5%, n=4)
  - High capacity retention (>99.7%) over initial 10 cycles





Functional end-user application demonstration



Successful demonstration of the HCCM performance for battery applications

Task 4 – Mixed Precursor Evaluation

- **Goal:** Demonstrate capability to insert the PCM into commercial products to produce lower cost carbons for electrochemical and other applications with equivalent or higher electrochemical performance and structural properties, respectively.

- **On-going Work:**
  - Down-select most relevant precursors for electrochemical and other applications.
  - Produce mixed blends of commercial precursor products and materials from Task 1 on a >100 g/batch process scale.
  - Perform comprehensive evaluation of the mixed blend products.

Task 5 – Techno-economic Analysis for Phase III and Commercialization

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- **Goals:** Demonstrate economic viability of HCCM/PCM commercialization which will provide the basis for technology transition and develop a robust transition plan.

- **On-going Work:**
  - Perform comprehensive techno-economic analysis based on the technical results of the scale-up PCM and HCCM production (Tasks 1 – 3).
  - Develop a transition plan for commercial production of HCCM on a multi-ton scale.
  - Work with commercial partners to produce HCCM using a patent & licensing strategy.

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Conclusions

- Domestic coal can be used to produce high value carbon products that exceeds the fuel and heat value of coal, illustrating sustainable market forces for manufacturing carbon materials from coal.
- PSI’s innovative technology provides a robust pathway to manufacture high value, high performance carbon materials (HCCMs) from coal with suitable properties for electrochemical applications.
- In the initial Phase II Program, PSI demonstrated key elements of the scale-up processes (hundreds-of-grams to kg) and initial economic feasibility for commercialization.
- In the current Phase IIA program, PSI is focusing on demonstrating enhanced scaled-up material production capability and suitable properties for electrochemical applications.

Acknowledgements

- Dr. Brett Hakey (DOE PM)
- Mr. Charles Miller
  (DOE PM - retired)
- Professor Lawrence T. Scott
  (Consultant, BC, Emeritus)
- Dr. Joe DiCarlo
- Industry partners
- PSI team

Physical Sciences Inc. (PSI) – Andover, MA

Appendix

Organization Chart

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- **Project Team, Organization, and Participants**
  - PSI:
    - Dorin Preda (PI/PM)
    - Sharon Song, Nathan Shipley, and Caitlin Bien (Material production and evaluation, Process optimization)
    - Chris Lang and Rachel Vozikis (Electrochemistry)
    - Professor Lawrence T. Scott (Consultant, BC, Emeritus)
    - Dr. Joe DiCarlo (Consultant)
    - Industry partners
  - DOE:
    - Dr. Brett Hakey (DOE PM)
    - Mr. Charles Miller (DOE PM - Retired)

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Gantt Chart

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