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

NATIONAL ENERGY TECHNOLOGY LABORATORY

Dorin V. Preda (PI), Min K. Song, Jake T. Herb, David P. Gamliel, Nathan R. Shipley, Gabrielle Aversa, Christopher M. Lang, Zach D. Whitermore, Jeffrey Y. Yee
Physical Sciences Inc. (PSI), DOE Contract Number: DE-SC0018837

Acknowledgment/Disclaimer

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

Project Description: DoE's Need

- Domestic coal can be used to manufacture high value carbon products for multiple applications.

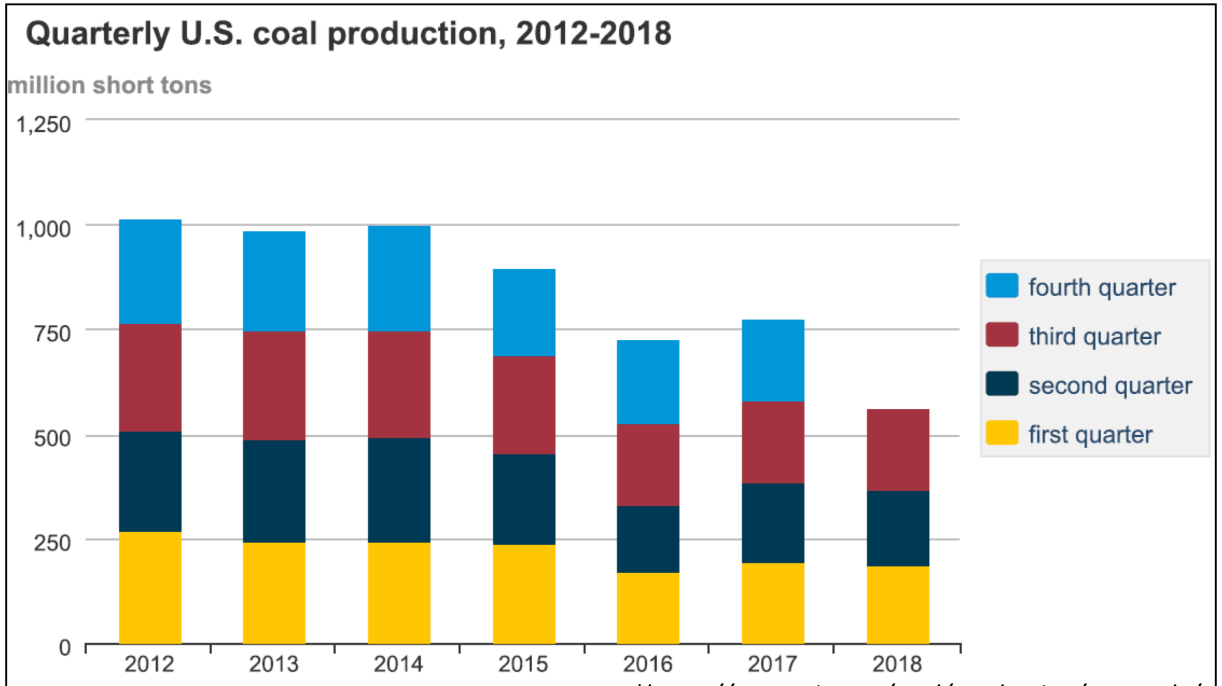
- The market value of these high performance materials often exceeds the fuel and heat value of coal, which illustrates there are sustainable market forces for manufacturing carbon materials from coal.

- Current processes to produce high performance carbon materials from coal pose significant challenges associated with substantial chemical modifications of the native coal structures that require high temperatures and corrosive reagents.

Coal Consumption Overview

NATIONAL ENERGY TECHNOLOGY LABORATORY

- U.S. coal consumption totaled 194.1 million short tons in the third-quarter of 2018
  - 23.9% higher than the 15.6 million short tons reported in second-quarter 2018.
  - 4.6% lower than the 203.5 million short tons reported in third-quarter 2017.
- The electric power sector accounted for about **93.6%** of the total U.S. coal consumption in the third-quarter of 2018.



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

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

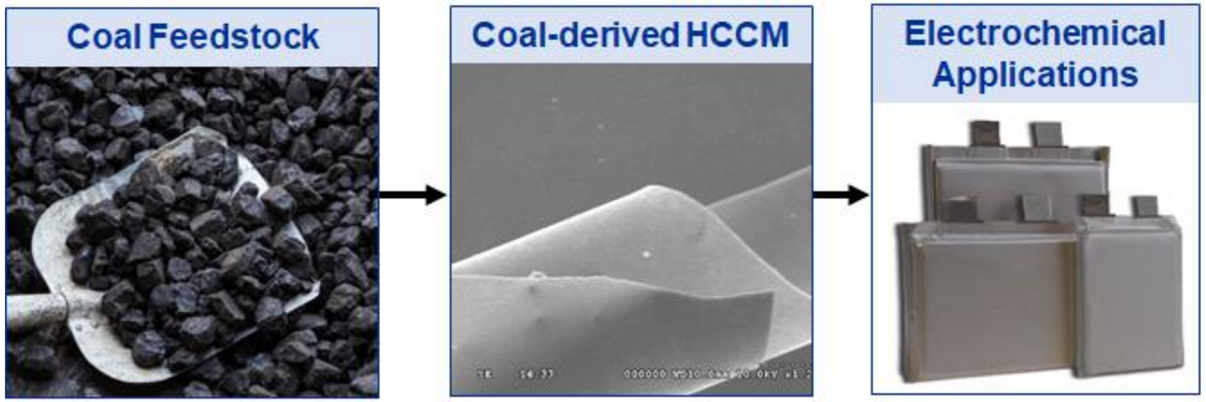
Executive Summary

NATIONAL ENERGY TECHNOLOGY LABORATORY

- **Physical Sciences Inc. (PSI) developed and demonstrated an innovative and scalable approach to produce high value, carbon-based products from coal feedstocks.**
- The innovation is a two-step process that generates both a high conductivity carbon material (**HCCM**) with high surface area (>50 m^2/g) and low mineral content (Fe <100ppm) as well as valuable byproducts (mineral fractions for trace elements recovery and low emission fuels).
  - Processes are scalable and amenable to multiple coal feedstocks
- The coal-derived HCCM was demonstrated in battery electrode formulations and showed comparable electrochemical performance to that of the benchmark commercial product.
- Preliminary techno-economic analysis demonstrated economic feasibility of scale-up plan and commercialization.
- Collaboration with EnerG2 for product scale-up and commercialization pathways.

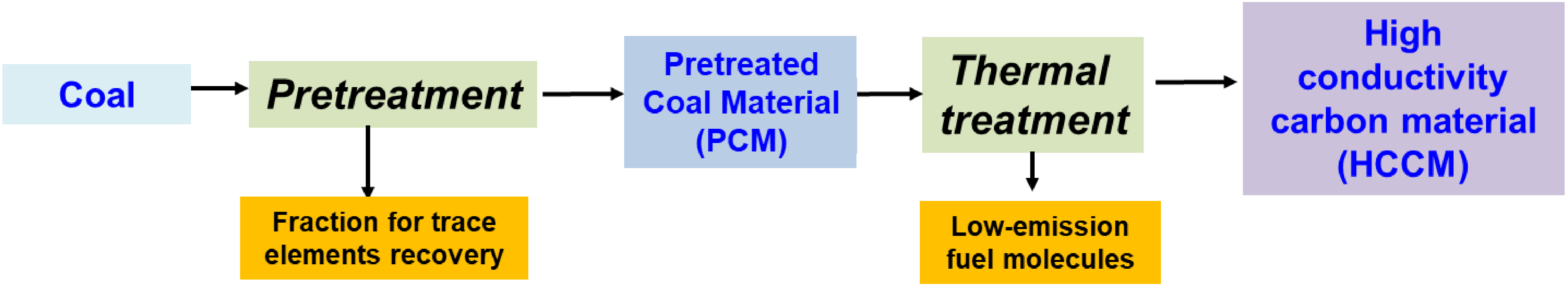
Innovative Approach

- PSI's technology builds upon pre-existing coal structures to create high conductivity features under mild conditions using innovative 2-step process.
- Coal pre-treatment processes → high yields, minimal reagent usage, and efficient recycle.
- Robust process to produce HCCMs from pre-treated coal → Valuable byproducts
  - (e.g., minerals, low-emission gas fuels)
- Processes for production of battery electrode formulations with HCCM.
- Pathways for process economic viability.
- Robust transition plans.



Process Steps

- Robust two step process to produce HCCM.
  - High value products and byproducts.
- 1st Step: Pretreatment that produces the intermediate PCM.
  - Valuable mineral fraction byproduct → recovery of trace elements.
- 2nd Step: Thermal treatment that produces the target HCCM.
  - Byproduct comprises low-emission fuel molecules → additional revenue stream.



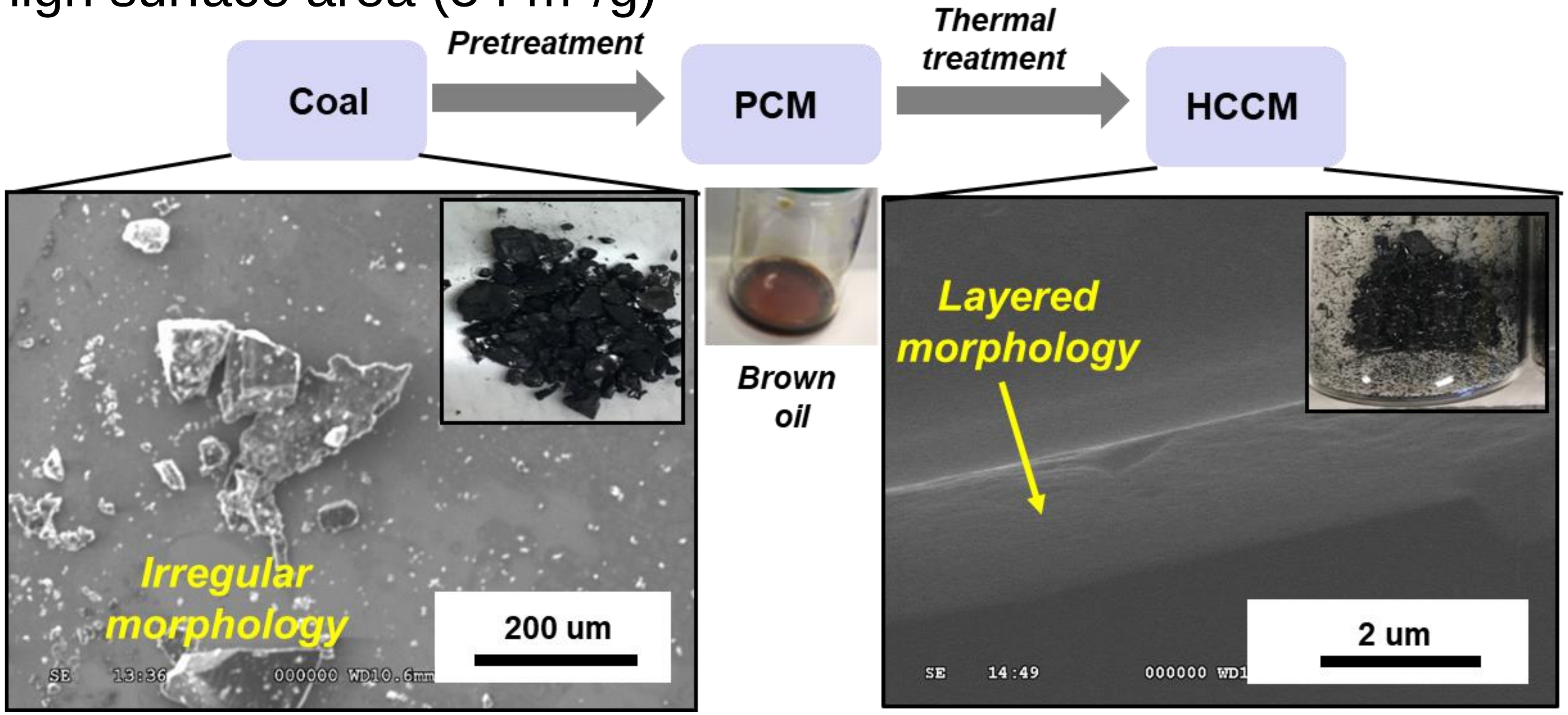
Phase I Results: Summary

NATIONAL ENERGY TECHNOLOGY LABORATORY

- Anthracitic and bituminous coals were suitable as feedstocks for the PSI process.
- Coal pretreatment with high yields resulting in a pretreated coal material (PCM) suitable for the production of HCCMs.
- Robust pyrolytic processes to produce high conductivity carbon materials (HCCMs) from PCM.
  - HCCMs with the required properties for use in electrochemical applications: (1) Low mineral content: Fe < 100 ppm; (2) High surface area: 54 m^2/g.
- The production of gaseous byproducts 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.

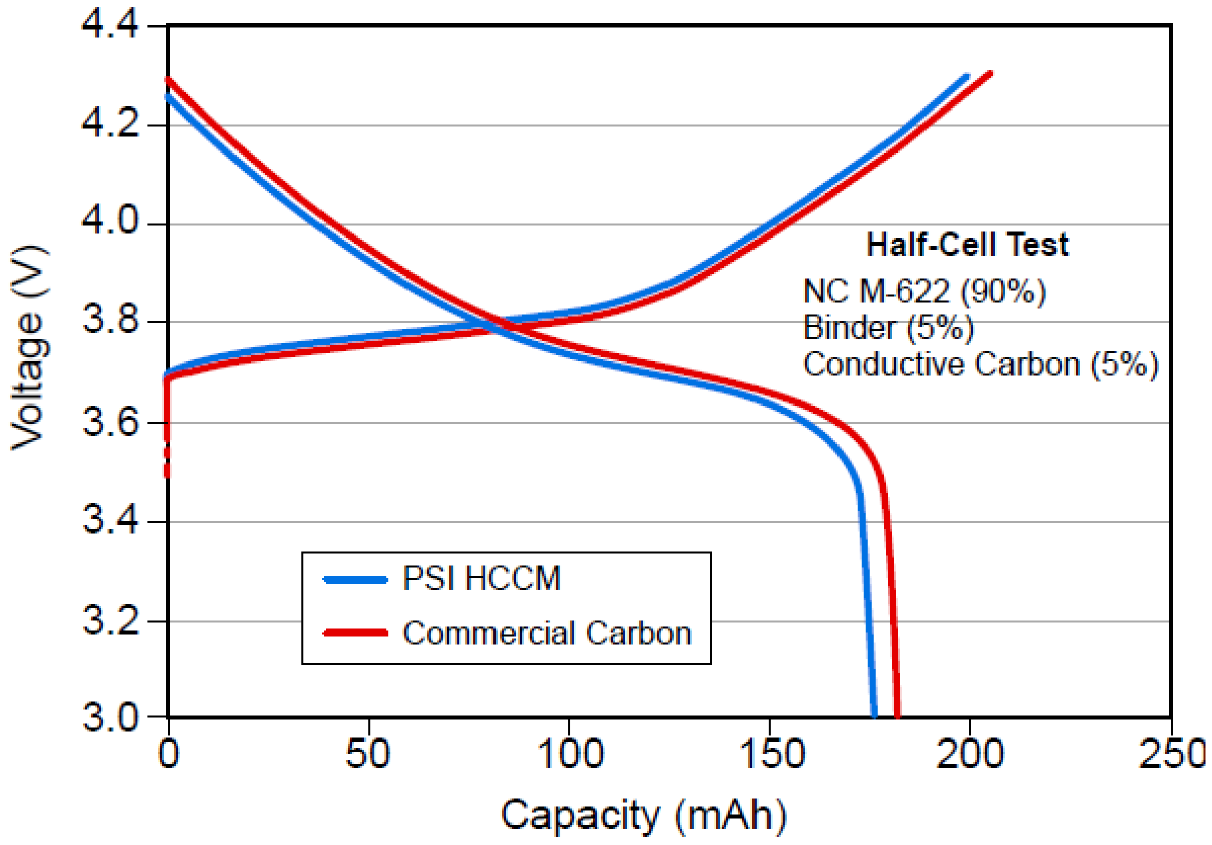
Phase I: HCCM Characterization

- **HCCM product with suitable properties for electrochemical applications including:**
  - Layered morphology
  - Low mineral content (Fe < 100 ppm)
  - Relatively high surface area (54 m^2/g)



Electrochemical Evaluation – Cathode

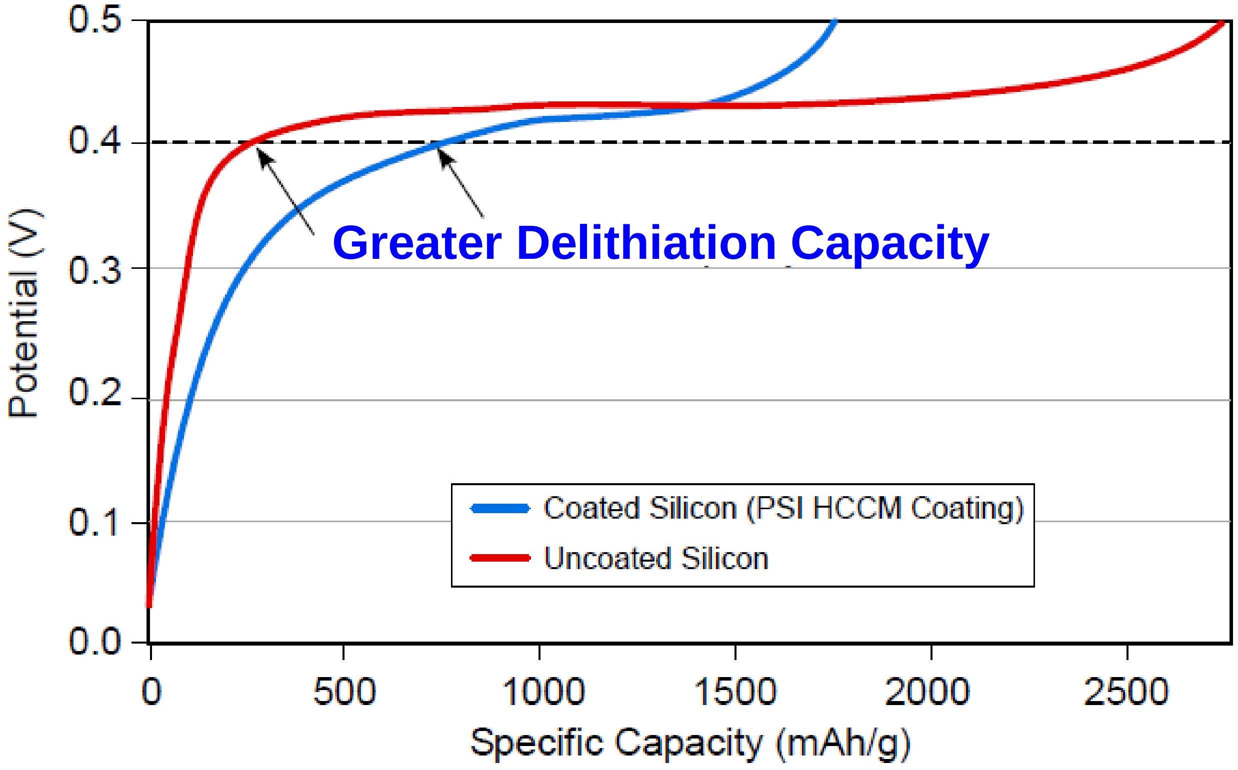
- Battery electrodes were produced with HCCM using industry established protocols.
- NCM 622 cathode formulations showed comparable performance to formulations that use commercial conductive carbon.
- **Performance analysis of cathode HCCM formulations demonstrated the feasibility of using HCCMs in state-of-the art batteries.**



Electrochemical Evaluation – Anode

NATIONAL ENERGY TECHNOLOGY LABORATORY

- In silicon composite anode formulations, the PSI HCCM silicon exhibited an improved voltage profile upon delithiation compared to an uncoated control.
- Greater delithiation capacity in the coated silicon material at a given voltage compared to uncoated silicon materials.
- **Performance analysis of anode HCCM formulations demonstrated the feasibility of using HCCMs in state-of-the art batteries.**



Phase II Technical Objectives

- Demonstrate scaled-up coal pretreatment processes that produce >1 kg/batch of pretreated coal materials (PCMs) with high yield (>75% g/g basis) and high purity (99+% on metals basis).
- Demonstrate that the PCM produced via the scale-up process can be processed using the Phase I process to produce HCCM material.
- Demonstrate that the HCCM product satisfying TO#2 can be formulated into electrodes with the equivalent electrochemical performance as that measured in Phase I.
- Demonstrate large scale (>0.5 kg/batch) production of the HCCM from PCM.
- Demonstrate blends of PCM with commercial precursors that produce conductive carbon products with key properties within ±2% of those obtained with pure precursors.
- Develop a transition and scale-up plan for profitable conversion of coal to HCCM on the multi-ton scale.

Phase II Work Plan

NATIONAL ENERGY TECHNOLOGY LABORATORY

- **Task 1 – Coal pretreatment scale-up**
  - Develop and optimize a scalable process that will produce PCM for the HCCM production process
- **Task 2 – HCCM production validation**
  - Evaluate HCCM products (Task 1) for electrochemical applications
- **Task 3 – Electrochemical performance validation**
  - Evaluate the materials produced in Task 2 in lithium ion battery cathode formulations
- **Task 4 – HCCM production scale-up**
  - Develop a scaled-up HCCM production process that will produce high purity HCCM products from materials produced in Task 1
- **Task 5 – Mixed precursor evaluation**
  - Produce a mixed blend of commercial precursors and PCMs from Task 1
  - Evaluate the blends to produce HCCMs that will be evaluated for electrochemical performance (Task 3)
- **Task 6 – Techno-economic model for commercialization**
  - Evaluate multiple economic drivers for the commercialization and process scale up
- **Task 7 – Management and reporting**

Phase II Results

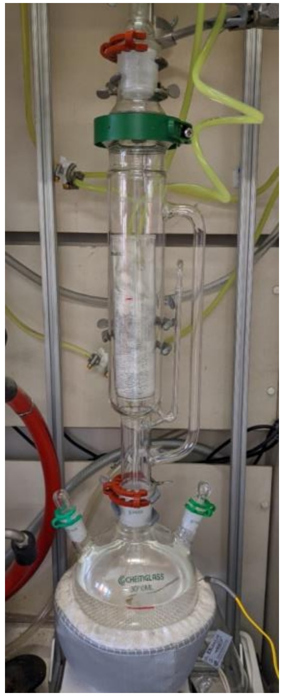
NATIONAL ENERGY TECHNOLOGY LABORATORY

- **Procured anthracitic coal (kgs) for scale-up efforts.**
    - Validated procured coal for the pre-coal treatment and thermal treatment processes compared to that of the Phase I results.
- **Developed and optimized scalable (up to 1 kg/batch) pre-coal treatment process to produce PCM using the Soxhlet and micro-pilot scale reactor.**
- **Robust, scalable (up to 0.2 kg/batch) pyrolytic processes to produce high conductivity carbon materials (HCCMs) from PCM.**
    - HCCMs with similar properties as that of the Phase I results: (1) high surface area: 291 m^2/g, (2) sheet-like morphology, and (3) Low mineral content: Fe < 42 ppm.
- **Validated the Phase I process for the production of battery electrode formulations with HCCM derived from scaled-up PCM.**
    - High performance electrode could be produced with the performance necessary for integration in state of the art batteries.
    - Improved electrochemical performance compared to the Phase I results.

1st Step: Production of PCM

- **PSI has demonstrated the capability to produce PCM on various scale.**
  - (Phase I) Small Scale: up to 50 g per batch using batch method.
  - (Phase II) Large Scale: 0.2-2 Kg using Soxhlet and micro-pilot reactors.

Small Scale
(20-50g per batch)



Large Scale
(~0.2 Kg per batch)

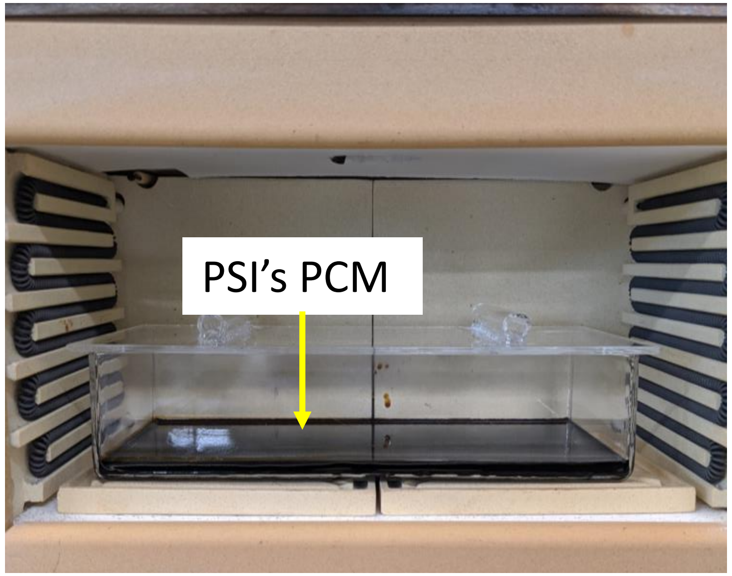
Large Scale
(1-2 Kg per batch)

PCM
(200g)

2nd Step: Production of HCCM

- **PSI has demonstrated the capability to produce HCCMs on various scale.**
  - (Phase I) Small Scale: <50g per batch
  - (Phase II) Large Scale: 0.1 – 0.25 kg per batch

Small Scale
(<50g per batch)



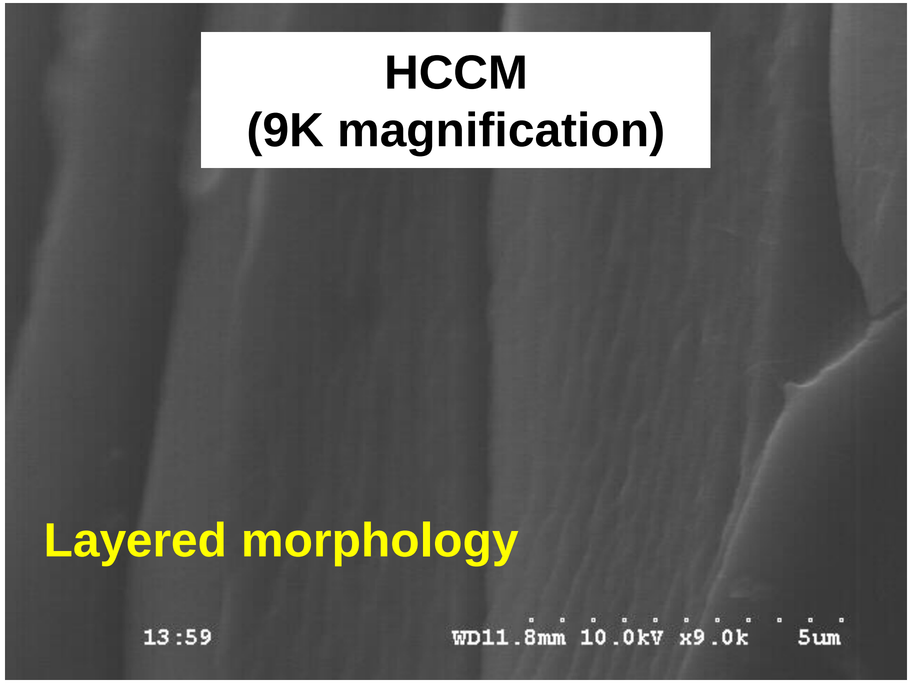
Large Scale
(0.1 - 0.25 kg per batch)

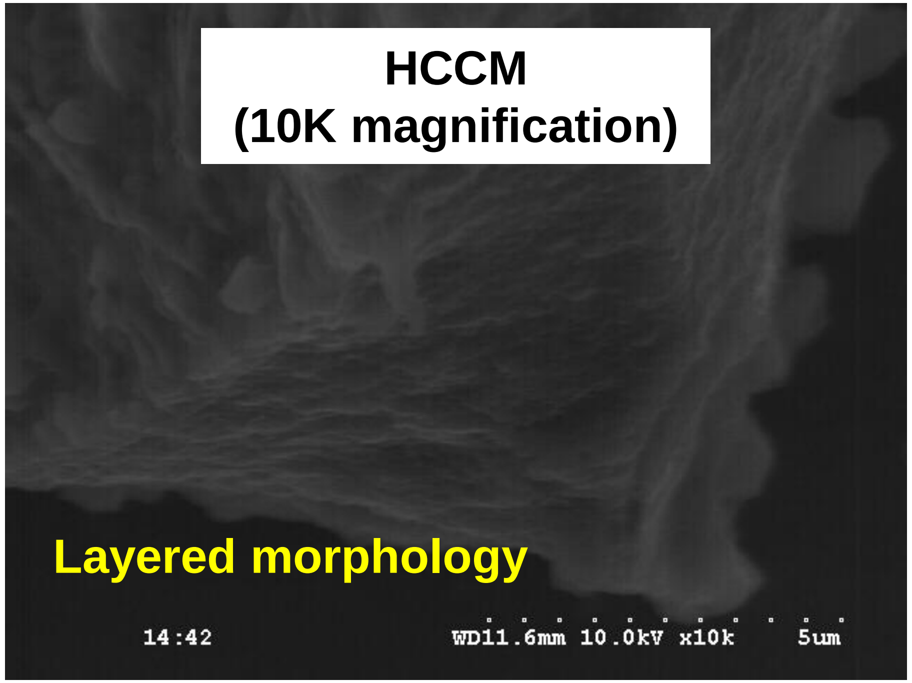
PSI's HCCM

Phase II: HCCM Characterization

NATIONAL ENERGY TECHNOLOGY LABORATORY

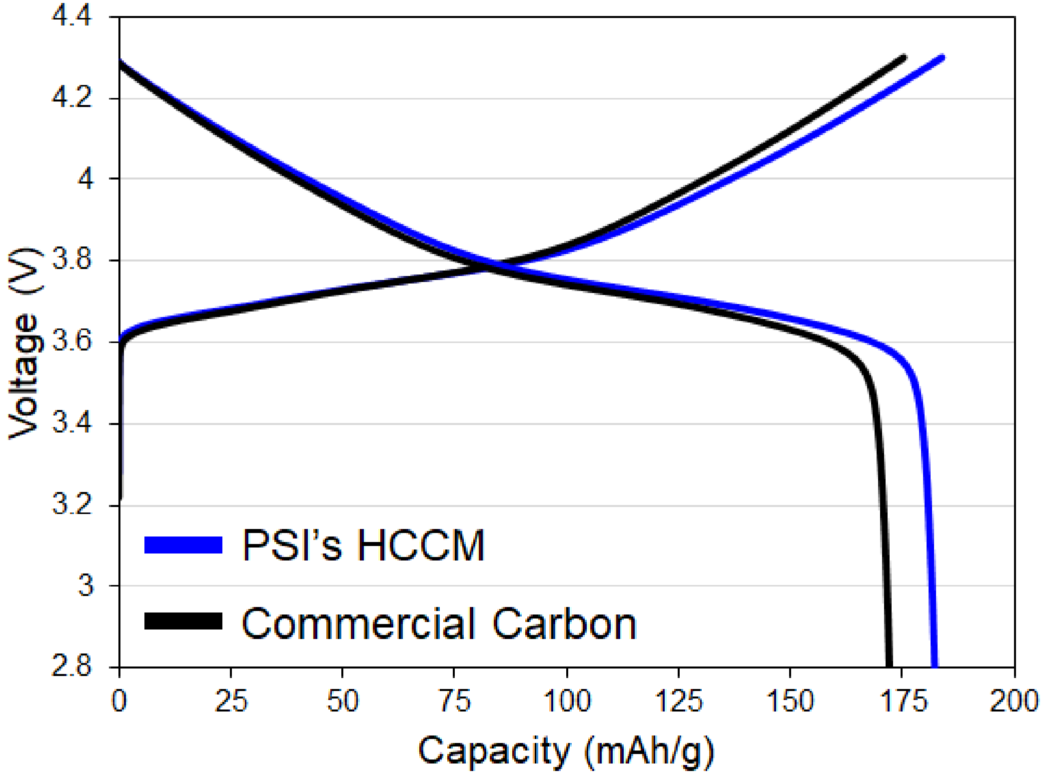
- **HCCM product with suitable properties for electrochemical applications including:**
  - Layered morphology
  - High surface area (as high as 290 m^2/g)
  - Low mineral content (e.g., Fe ~40 ppm)





Phase II: Electrochemical Evaluation

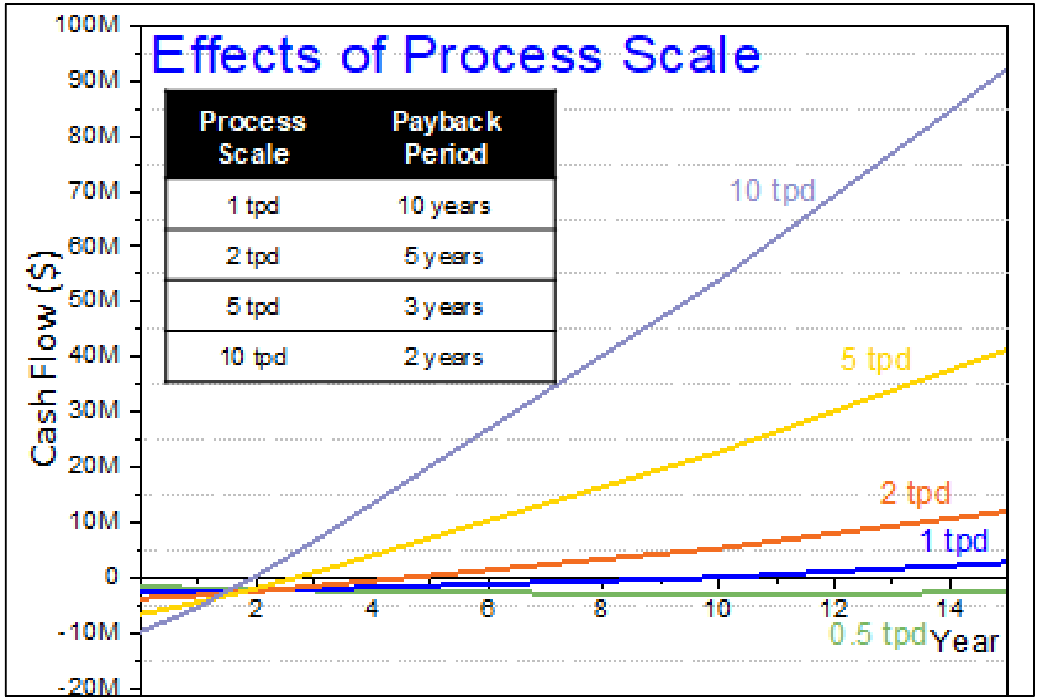
- Battery electrodes were produced with HCCM using industry established protocols.
- NCM 622 cathode formulations showed comparable performance to formulations that use commercial conductive carbon.
- **Performance analysis of cathode HCCM formulations demonstrated the feasibility of using HCCMs in state-of-the art batteries.**
    - **Improved electrochemical performance compared to that of the material produced in Phase I**



Techno-economic Analysis

NATIONAL ENERGY TECHNOLOGY LABORATORY

- The technical results were used to perform a preliminary techno-economic analysis.
- Aspen simulations indicated that process scale is a main contributor to the process economics.
- 5X reduction in the payback period upon an increase of the capacity from 1 tonne-per-day (tpd) to 10 tpd.
- **The developed processes can result in an economically viable commercial operation on scale-up.**



Process Scale	Payback Period
1 tpd	10 years
2 tpd	5 years
5 tpd	3 years
10 tpd	2 years

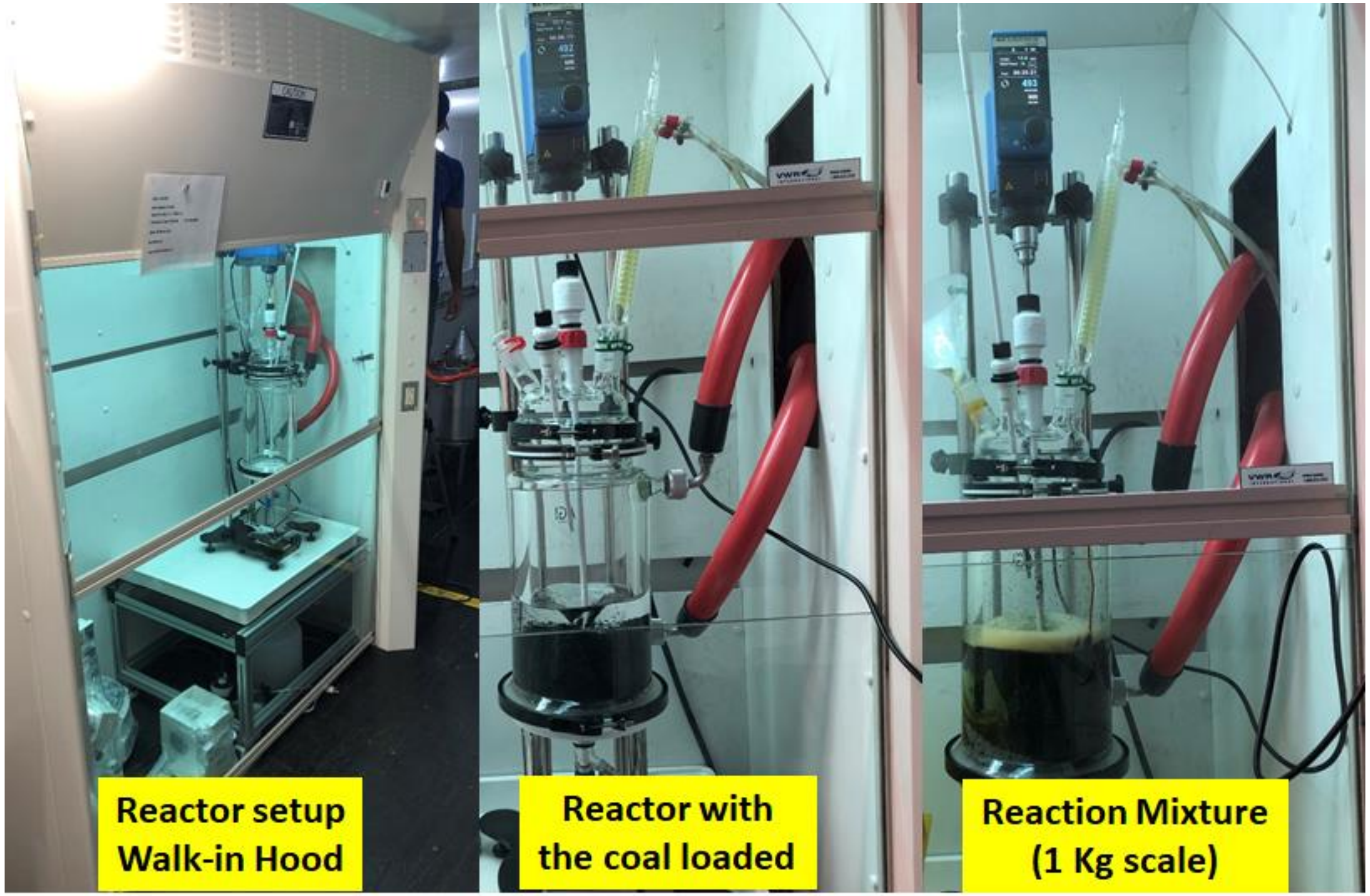
Conclusions

- Domestic coal can be used to produce high value carbon products for multiple applications including electrochemistry.
    - Market Value of these high value carbon exceeds the fuel and heat value of coal, illustrating a sustainable market forces for manufacturing carbon materials from coal.

- PSI’s innovative technology provides a mechanism to manufacture high value, high performance carbon materials (HCCMs) from domestic coal through the 2-step process.
    - Economically viable process to produce high value, conductive carbon from coal.
    - Economically feasible for scale-up and commercialization.
    - High conductivity carbon products with >20X higher value than coal.
    - Valuable byproducts (low-emission energy and trace element recovery).

Future Work

- Process optimization and scale up (>1kg/batch).
- Further demonstration of electrochemical and other potential applications.
- Work with industrial partners such as EnerG2 to assess the market opportunity and applications and develop commercialization strategies.



Acknowledgements

- Mr. Charles Miller (PM - DOE/NETL)
- Professional Lawrence T. Scott (BC, Emeritus)
- EnerG2 (Industry Partner)
  - Chris Hohman
  - Ben Kron
- PSI team