DOE Bioenergy Technologies Office (BETO)
2019 Project Peer Review

Pilot-Scale Biochemical and Hydrothermal Integrated Biorefinery (IBR) for Cost-Effective Production of Fuels and Value added Products

Date: March 4-8, 2019
Technology Session Area Review

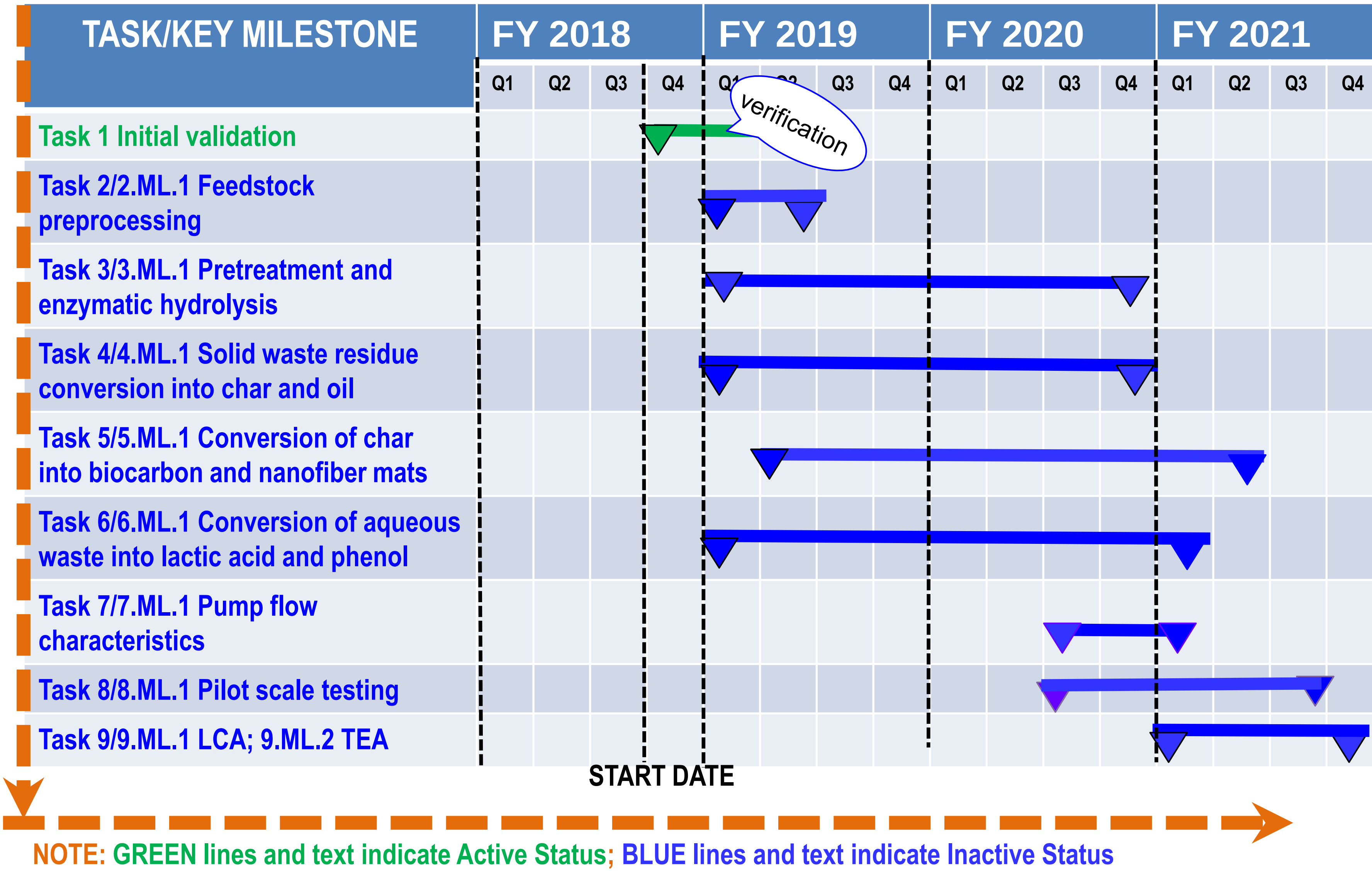
Principal Investigator: Rajesh Shende
Organization: South Dakota School of Mines & Technology

Goal Statement

- Demonstrate production of high value products from waste streams, as given below, generated during conventional biochemical processing at a pilot scale level with 1 tpd throughput.
  - Aqueous waste stream (I) from alkaline pretreatment of corn stover
  - Solid waste residue (I), unhydrolyzed solids (UHS)
  - Aqueous waste stream (II) from hydrothermal processing
  - Biochar waste (II)

- **Outcomes**: i) Four products from solid waste and aqueous waste, which include biocarbon, carbon nanofibers, lactic acid/PLA and phenols and ii) inclusion of revenues derived from these products into TEA and LCA demonstrating BETO’s 2022 cost target of $3/gge with >50% reduction in GHGs emission.

Tasks and Key Milestones



U.S. DEPARTMENT OF ENERGY | Energy Efficiency & Renewable Energy

Project Budget Table

	Original Project Cost (Estimated)			Project Spending and Balance		Final Project Costs
Budget Periods	**DOE Funding**	**Project Team Cost Shared Funding**	**Contingency**	**Spending to Date**	**Remaining Balance**	**What funding is needed to complete the project.**
BP1 – (July 1, 2018- May 31, 2019)	$122,200	$15,869	----	$40,422	$97,647	
- SDSMT Verification	$90,077	$11,632		$40,422	$61,287	
- ODU Verification	$21,623	$3,126		$0	$24,749	
- VCU Verification	$10,000	$1,111		$0	$11,111	
SwRI	$500	$0		$0	$500	
BP – 2 (Not yet started)	$1,803,960	$215,966	----	$0	$2,019,926	
*FFRDC $ / % BP-1 $1000 / 1% *FFRDC $ / % BP-2 $159,000 / 7%						

Quad Chart Overview

Timeline

- Project start date: 07/01/2018
- Original project end date: 06/30/2021
- Percent complete: Still under validation

Budget

	Total Costs Pre FY 17	FY 17 Costs	FY 18 Costs	Total Planned Funding (FY 19-Project End Date
DOE Funded	$0	$0	$6,425	$1,919,735
Project Cost Share	$0	$0	$0	$231,835
SDSMT	$0	$0	$6,425	$1,223,280
ODU	$0	$0	$0	$250,000
VCU	$0	$0	$0	$280,000
INL	$0	$0	$0	$0
SwRI	$0	$0	$0	$166,455

Barriers

- Barriers addressed
  - Ct-J: Identification and Evaluation of Potential Bioproducts,
  - Ct-K: Developing Methods for Bioproduct Production, and
  - ADO-A: Process Integration

Partners

- Tyler Westover, Idaho National Laboratory (INL)
- Ram Gupta, Virginia Commonwealth University (VCU)
- Sandeep Kumar, Old Dominion University (ODU)
- Hao Fong, South Dakota School of Mines & Technology (SDSMT)
- Southwest Research Institute

1 – Project Overview

Common problems

- Lignin/solid residue and acids land up in waste stream during biochemical processing
- Phenols and organics acids also found in waste stream of thermochemical processing
- Valuable carbon resource is wasted in solids and aqueous wastes
- Recovery and treatment of wastes do not contribute to offset the fuel costs

Proposed solution

- If wastes are converted into high value products, it will offset the fuel cost
- *Novel* approach of integrated technologies for solid waste conversion into biocarbon and carbon nanofibers, and aqueous waste conversion into lactic acid and phenols
- Reduce solid waste, utilize aqueous phase carbon, generates products that will reduce fuel cost

1 – Project Overview

This project provides-

Data/results for i) technology development for the conversion of solid waste (UHS) derived from corn stover into biocarbon and carbon nanofibers and enrichment of aqueous waste with lactic acid and phenols, ii) pilot scale testing at 1 tpd throughput, and iii) TEA and LCA

Application of products in other industries

Batteries, EMI shielding, heat management, conductors, capacitors, packaging, agriculture, transport, electronics, textiles etc

Market price of the products (2016)

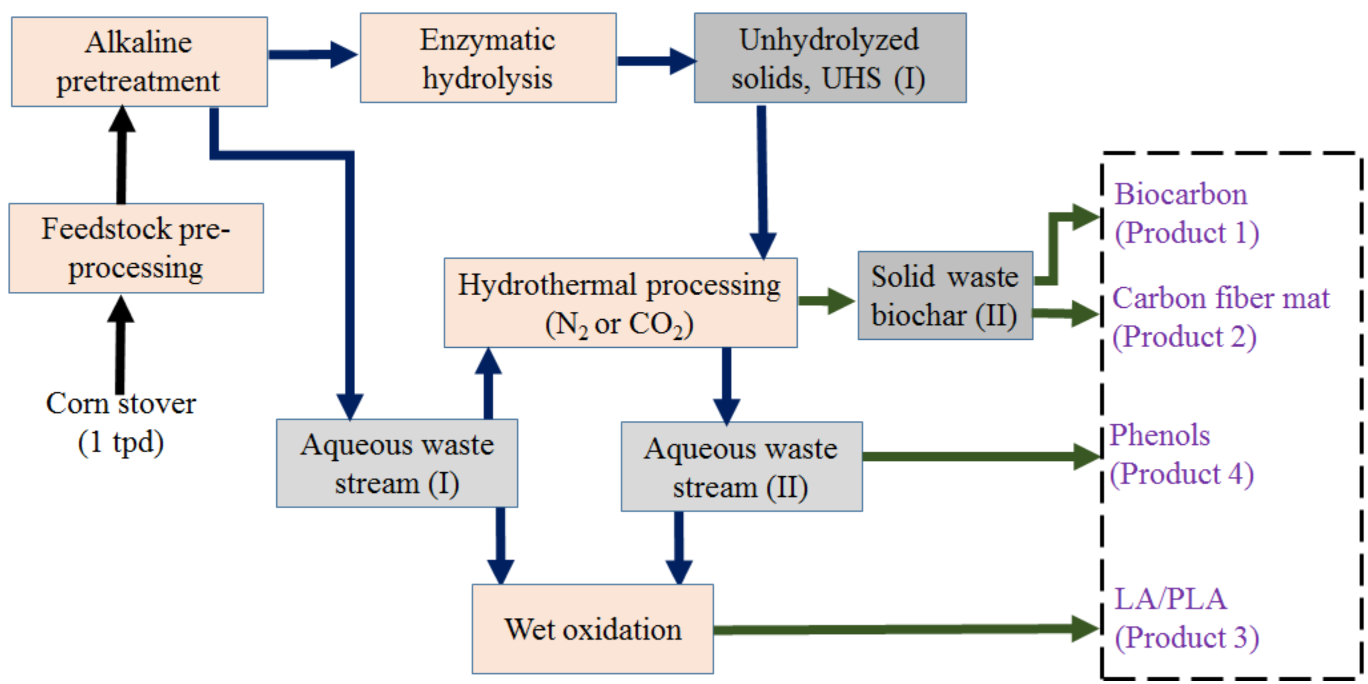
- Biocarbon > $3/kg
- Carbon nanofibers ~ $1200/kg
- Polylactide (PLA) ~$4/kg
- Phenol ~ $1/kg

2 – Approach Technical: Summary

1. Develop integrated technology platform to effectively process wastes derived from corn stover processing into solid and liquid products
2. Characterize the products yield and quality
3. Perform TEA and LCA and estimate fuel costs in terms of $/gge

Bench scale
- Physical tests
- Characterization
- Mass/energy balance

Pilot scale
- 1 tpd throughput
- Characterization
- Mass/energy balance
- TEA and LCA



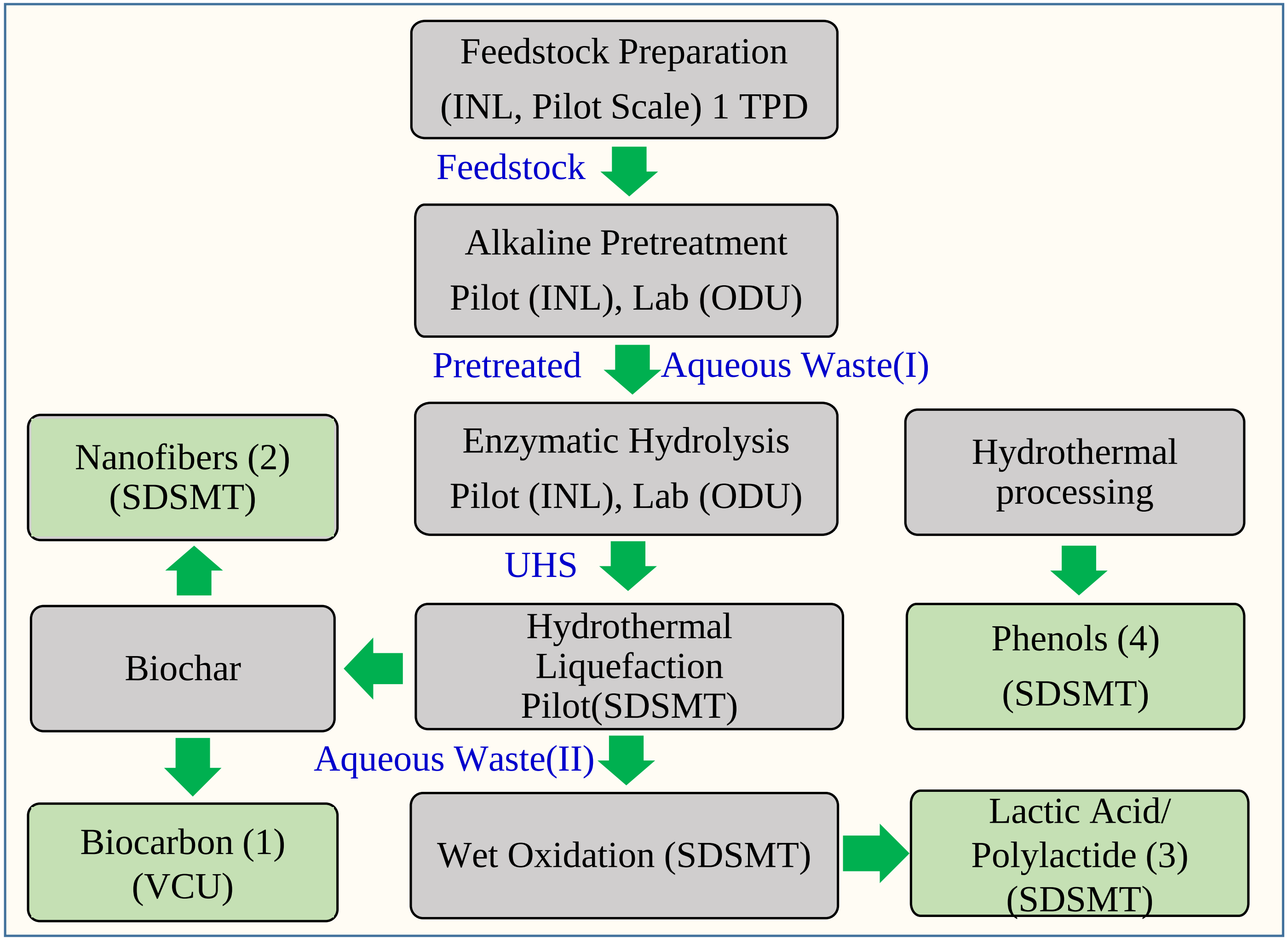
2 – Approach (Technical)

Challenges

- Products yield and quality at a pilot scale
- Suitability of char produced at a pilot scale for biocarbon and carbon nanofibers
- Effectiveness of wet oxidation on liquid side co-products
- Transport of UHS and aqueous waste

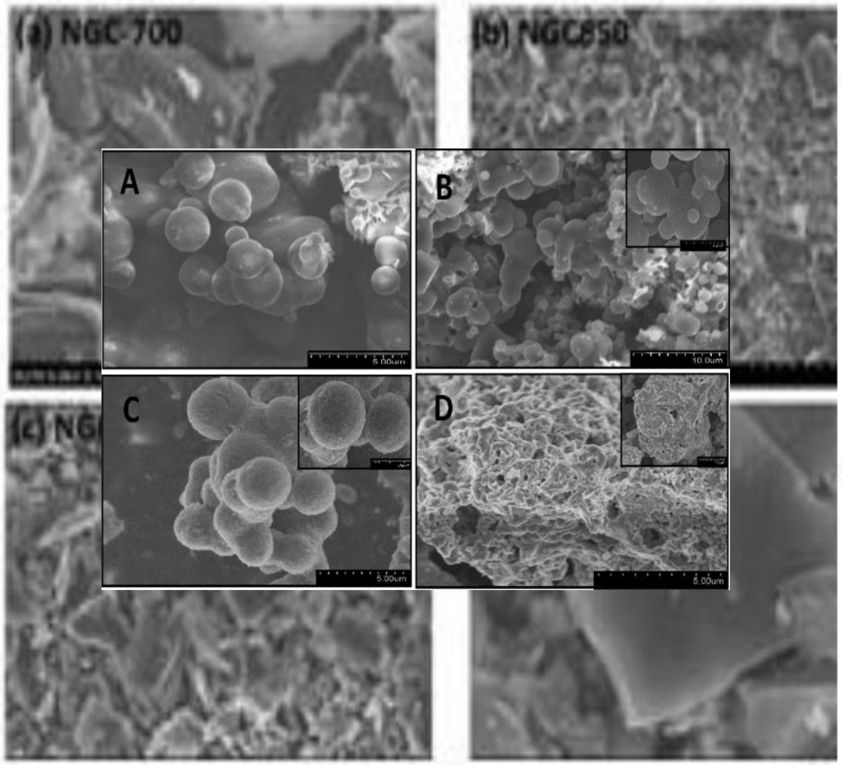
Key success factors

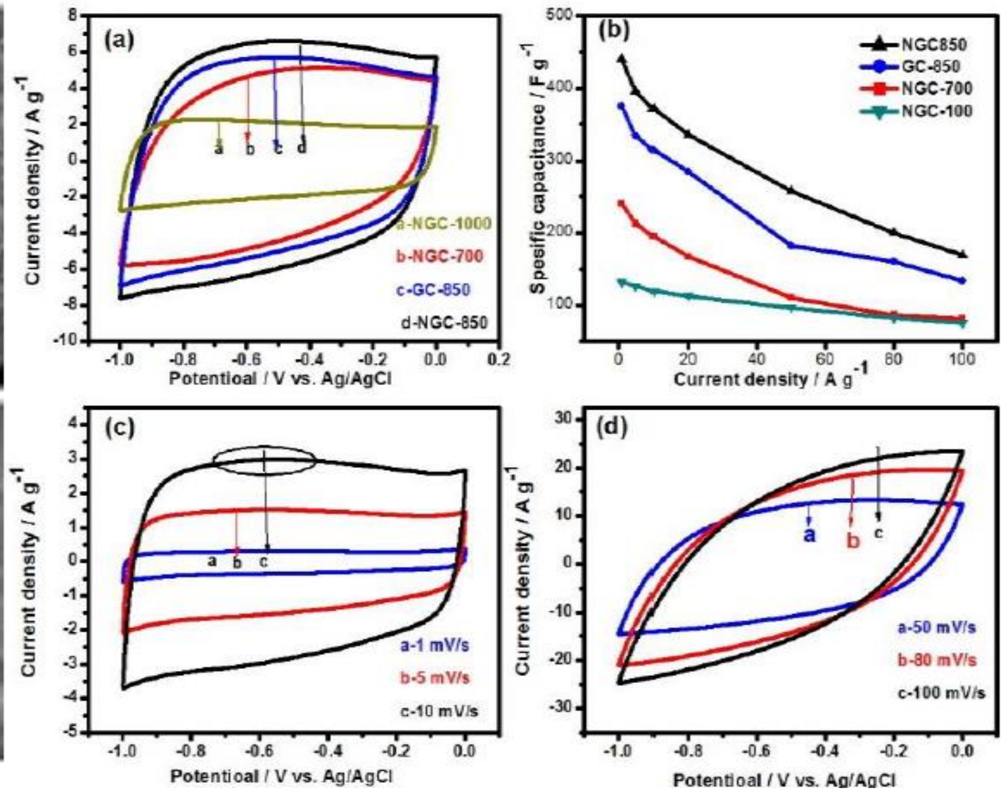
- % agreement between lab and pilot scale trials
- In-depth estimation of revenue from the products



2 – Approach (Technical)

Biocarbon (product-1)

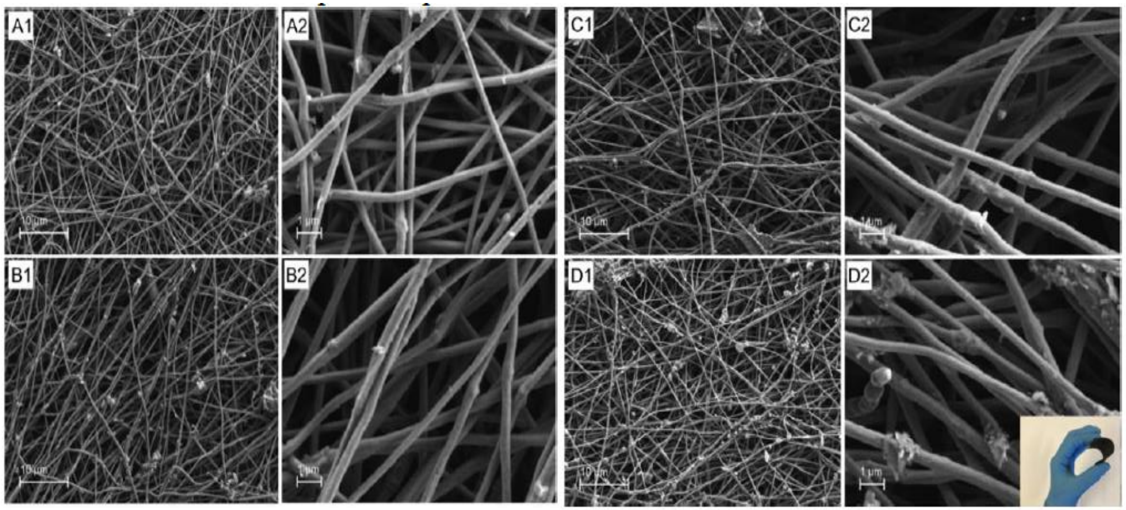


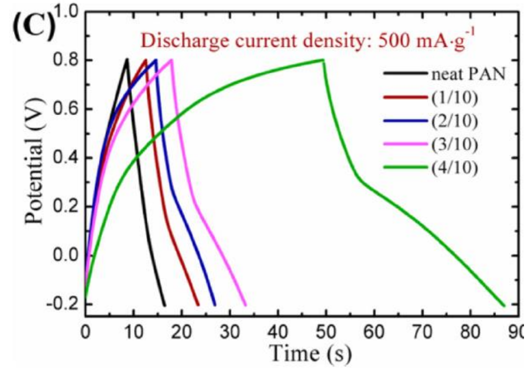


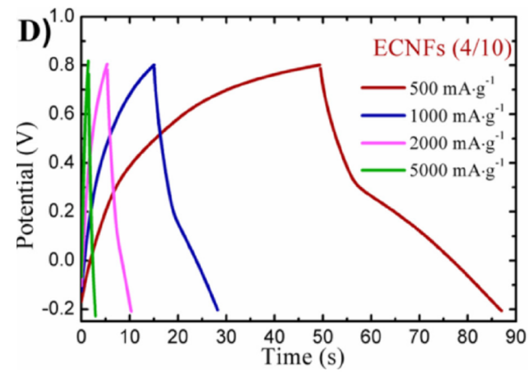
Demir, M., et al., *Graphitic Biocarbon from Metal-Catalyzed Hydrothermal Carbonization of Lignin.* Industrial & Engineering Chemistry Research, 2015. **54**(43): p. 10731-10739

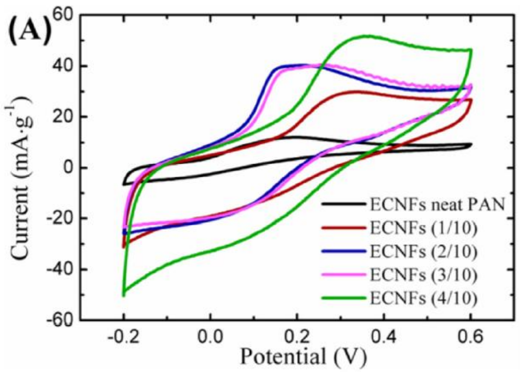
2 – Approach (Technical)

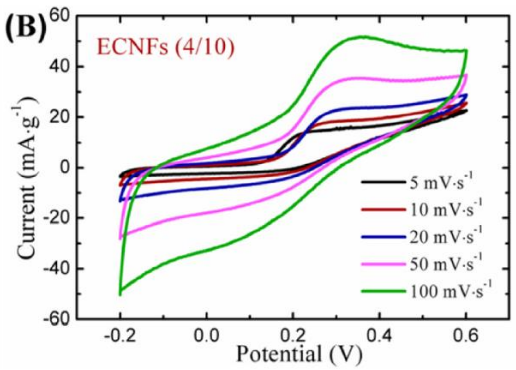
Carbon nanofibers (product-2)







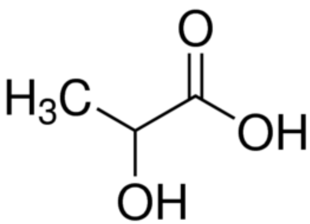


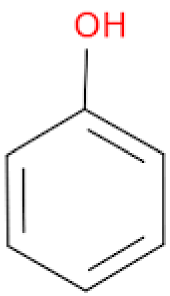


Wei et al. *Mechanically flexible electrospun carbon nanofiber mats derived from biochar and polyacrylonitr*ile, Materials Letters, 2017, 205: p 206-210.

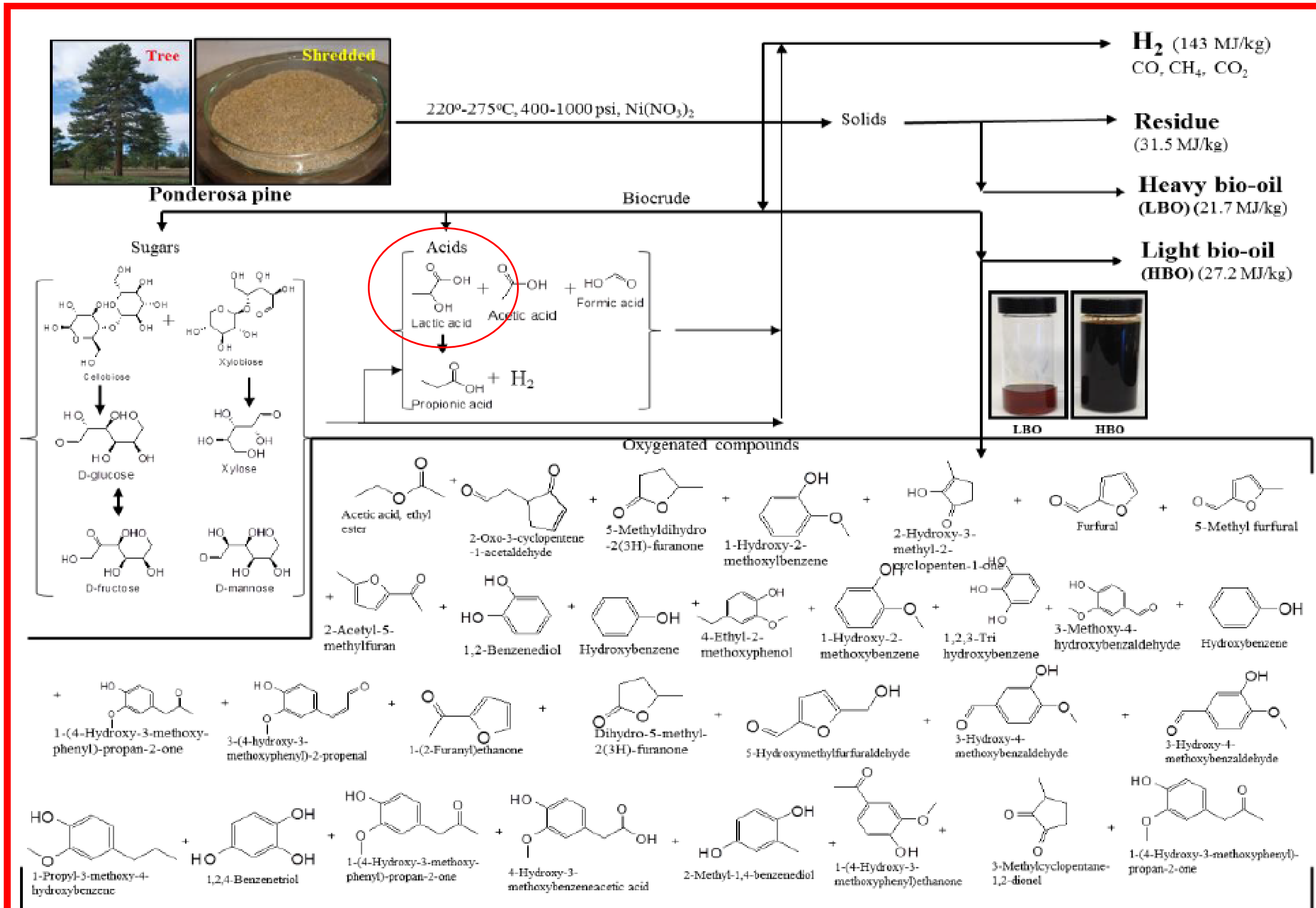
2 – Approach (Technical)

Lactic acid and phenols (products 3 & 4)





Tungal and Shende, *Hydrothermal liquefaction of pinewood (Pinus ponderosa) for H₂, biocrude and bio-oil generation* 2014, 134, p: 401-412.



2 – Approach (Management)

- **Validation visits: plan and coordinate**
  – SDSMT&T, ODU and VCU

- **Task leadership: plan, prioritize, coordinate, review progress**
  – Bi-weekly team coordination meetings
  – Bi-weekly team coordination meetings with DOE personnel
  – Quarterly BETO review meeting

- **Create and follow approved management plans**
  – Milestones (quarterly) and deliverables
  – Performance metrics

- **Responsibilities**
  – Corn stover preprocessing and pilot scale alkaline pretreatment and enzymatic hydrolysis (1 batch): Tyler Westover
  – Alkaline pretreatment and enzymatic hydrolysis (lab scale): Sandeep Kumar
  – Hydrothermal liquefaction and wet oxidation (lab and pilot): Rajesh Shende
  – Conversion of char into biocarbon (lab): Ram Gupta, (pilot) Mark Feng
  – Conversion of char into carbon nanofibers (0.5-1 kg/day): Hao Fong

3 – Technical Accomplishments/ progress/Results

- **New project**
  - Start date 07/01/2018
  - Project is currently under verification phase

    (IE completed verification for ODU and VCU sites; Feb 19 – Feb 21, 2019 SDSMT site)

- ***Progress***
  - SDSMT Site is being prepared for the visit
  - Laboratory and analytical equipment are being tested
  - Personnel are being trained to master processes

  - Experimental work- INL has prepared and delivered 2 kg of pelletized corn stover. ODU is performing alkaline pretreatment and enzymatic hydrolysis. SDSMT is working on hydrothermal liquefaction of corn stover derived UHS to establish baseline values for different products.

4 – Relevance

Enabling biofuels by developing technologies for high value products from biochemical waste processing

- ❖ Directly supporting BETO's Mission:
  - – Developing cost effective technologies for bio-based products to economically leverage production of hydrocarbon fuels
  - – Developing technologies for converting waste streams into high value products is in DOE/BETO programmatic interests
  - – Proposed products will offset fuel cost meeting BETO's 2022 cost target of $3/gge.

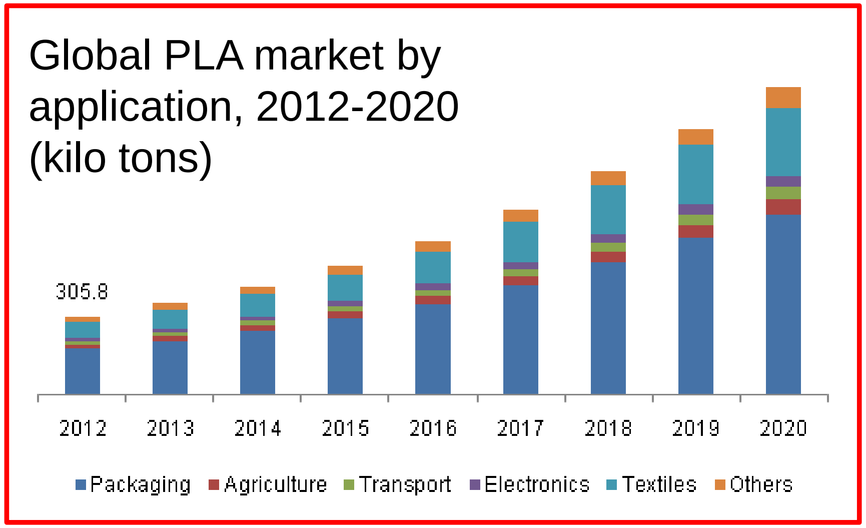
- ❖ Project metrics and targets are driven by TEA and LCA

4 – Relevance

Market potential of the products

- **Biocarbon** 22 kt/y of graphite will be required for the 0.5 million estimated batteries (70 kWh) by 2020.
- **Carbon nanofibers** $1 billion by 2021 at CAGR 24-25%
- **Lactic acid/PLA** Lactic acid 1961 kt and $3.82 billion by 2020 (CAGR 18.6%) and PLA $5.16 billion by 2020 (CAGR 20.9%)
- **Phenols** $19.78 billion by 2026 at CAGR 4.6%

http://www.prnewswire.com/news-releases/biochar-market-size-to-reach-589-billion-by-2022-grand-view-research-inc-565120141.html (accessed April 15, 2016)



http://www.grandviewresearch.com/industry-analysis/lactic-acid-and-poly-lactic-acid-market (accessed April 15, 2016)

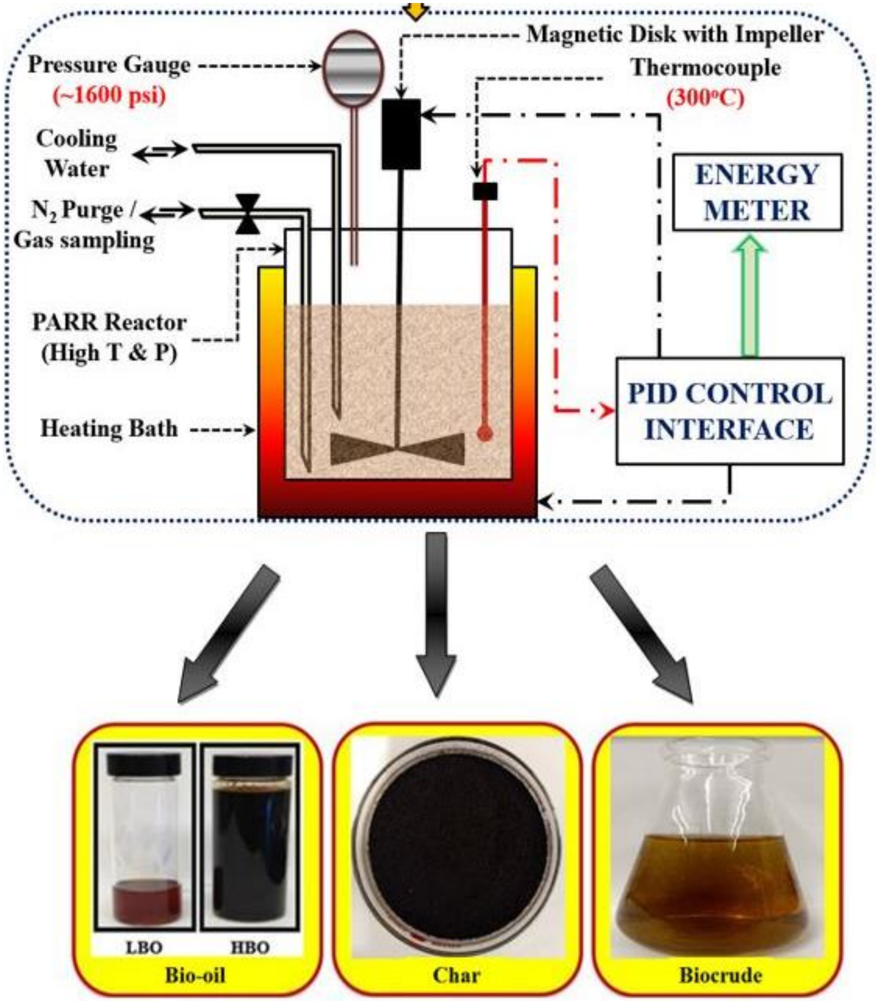
5 – Future Work: Overview

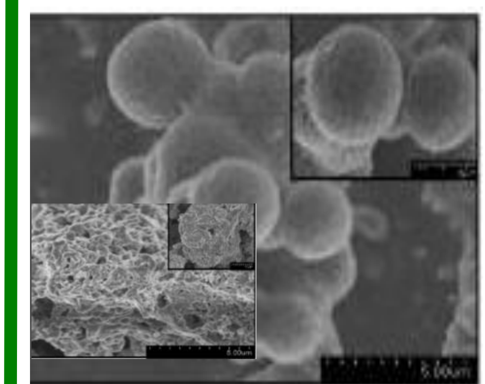
Project will address following areas:

- ❖ Thorough bench scale experimentation for waste conversion and characterization of different liquid and solid products
- ❖ Pilot scale testing of waste conversion technologies for high value products with throughput of 1 tpd
- ❖ Desired char characteristics for further conversion into biocarbon and carbon nanofibers
  - highly dependent on hydrothermal processing conditions
  - bench scale trials are successful but pilot scale need to be verified
- ❖ Enrichment of lactic acid by wet oxidation
  - bench scale experiments are needed to validate wet oxidation with biochemical aqueous streams
- ❖ In-depth TEA and LCA, and estimation of fuel cost by taking into account revenue generated from the products

5 – Future Work: Overview

Process/product optimization





5 – Future Work: Timeline

- ❖ Project validation 3 months

- ❖ Next 18 months: Bench scale experiments with corn stover
  - alkaline pretreatment and enzymatic hydrolysis
  - hydrothermal liquefaction of UHS
  - wet oxidation of aqueous streams
  - preparation of biocarbon and carbon nanofibers
  - characterization of different products

- ❖ Next 15 months: Bench/pilot scale experiments with corn stover
  - >lab - <pilot scale alkaline pretreatment and enzymatic hydrolysis
  - pilot scale hydrothermal liquefaction of UHS
  - >lab - <pilot scale wet oxidation of aqueous streams
  - pilot scale preparation of biocarbon
  - >lab - <pilot scale preparation of carbon nanofibers
  - pilot scale pump flow characteristics for UHS (15-20 wt% loading)
  - characterization of different products

Summary

Overview: This is a new project, which is currently under verification phase. Site verification for ODU and VCU sites have been completed. SDSMT site verification is planned from Feb 22 through Feb 24

Approach: Technical approach includes development of integrated technology platform to effectively convert wastes derived from corn stover processing into solid and liquid products, characterization of the products, and TEA and LCA

Technical Accomplishments/Progress/Results: INL has preprocessed corn stover with a hammer mill. ODU has performed alkaline pretreatment and enzymatic hydrolysis with this material on a lab scale. Hydrothermal liquefaction of corn stover derived UHS is being carried out at SDSM&T

Relevance: Enabling biofuels by developing technologies for high value products from biochemical waste processing

Future work: Project validation, followed by bench scale and pilot scale waste conversion technology development for high value products

Additional Slides

Publications, Patents, Awards, and Commercialization

This project: None so far as this project is newly awarded

Previous related project (few examples):

- ❖ Tungal, R.; Shende, R.V., Hydrothermal liquefaction of pinewood (Pinus ponderosa) for H_2, biocrude and bio-oil generation, *Applied energy* 2014, 134, p: 401-412.
- ❖ Wei Nan, Yong Zhao, Yichun Ding, Anuradha R. Shende, Hao Fong, Rajesh V. Shende, Mechanically flexible electrospun carbon nanofiber mats derived from biochar and polyacrylonitrile, *Materials Letters* 2017, 205: p 206-210.
- ❖ Kumar, S., et al., Hydrothermal pretreatment of switchgrass and corn stover for production of ethanol and carbon microspheres. *Biomass and Bioenergy*, 2011 35(2): p. 956-968.
- ❖ Muslum Demir, Zafer Kahveci, Burak Aksoy, Naveen K. R. Palapati, Arunkumar Subramanian, Harry T. Cullinan, Hani M. El-Kaderi, Charles T. Harris, and Ram B. Gupta, Graphitic biocarbon from metal-catalyzed hydrothermal carbonization of lignin, *I&EC Research*, 2015, 54, p: 10731-10739.

Status of technology transfer (commercialization): None

Commercialization plan

- Technology transfer activities are planned with the commercial entities to explore marketability of the products derived from the waste streams. Initial correspondence has been established.

- With Entrepreneur In Residence (EIR) program at SDSMT (and similar activities at partnering institutions), a successful plan will be developed for large scale product development systems for the waste streams originated in integrated biorefineries

- Partnership with NREL for future large scale trials.

Risks and Improvement Areas

Risks

1. Project is 3 years of focused effort on process/product optimization and pilot scale demonstration with 1 tpd throughput
2. Coordination among partnering institutions to ship the required materials for the next stage of processing
3. Technical risks such as product yields and characteristics of final products derived at the pilot scale

Mitigation Plan/Strategies

1. Need better planning and prompt communication to address the expectations
2. Focus on bench scale optimization in the initial phase and develop strategies from the beginning of the project for pilot scale testing
3. Simplify technology platform processing and product analysis