DOE Bioenergy Technologies Office (BETO) 2023 Project Peer Review

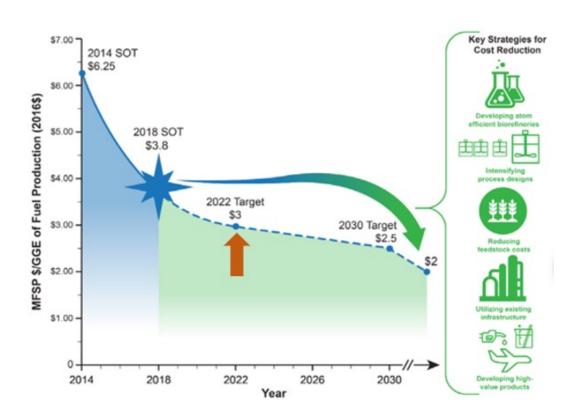
Pilot-Scale Biochemical and Hydrothermal Integrated Biorefinery (IBR) for Cost-Effective Production of Fuels and Value-Added Products

Date: April 3-7, 2023 Technology Area Session: Systems Development and Integration

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

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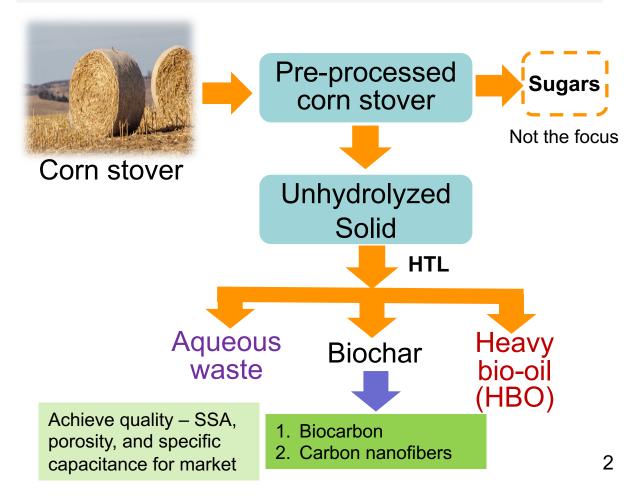
Project Overview



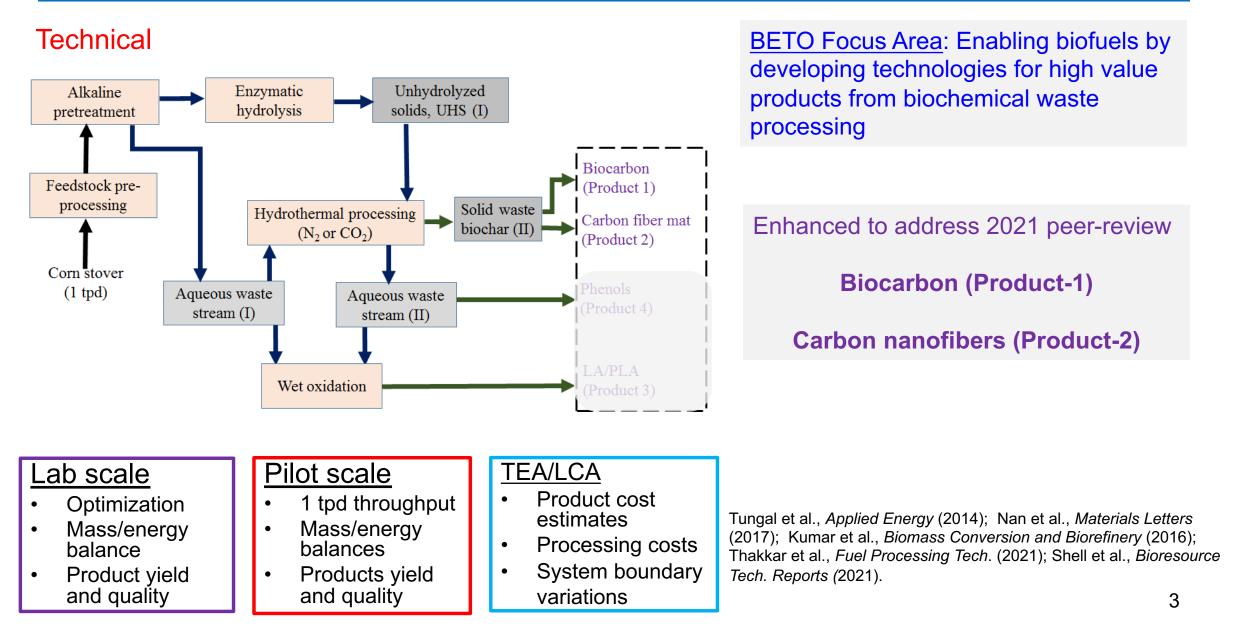
<u>https://www.energy.gov/sites/prod/files/2020/07/f76/betointegrated-strategies-to-enable-low-cost-biofuels-july-2020.pdf</u>; Ruan et al., Integrated Processing Technologies for Food and Agricultural By-Products, Academic Press (2019); 2016 Billion-Ton Report, USDOE.

Relevance to BETO's Programmatic Goals

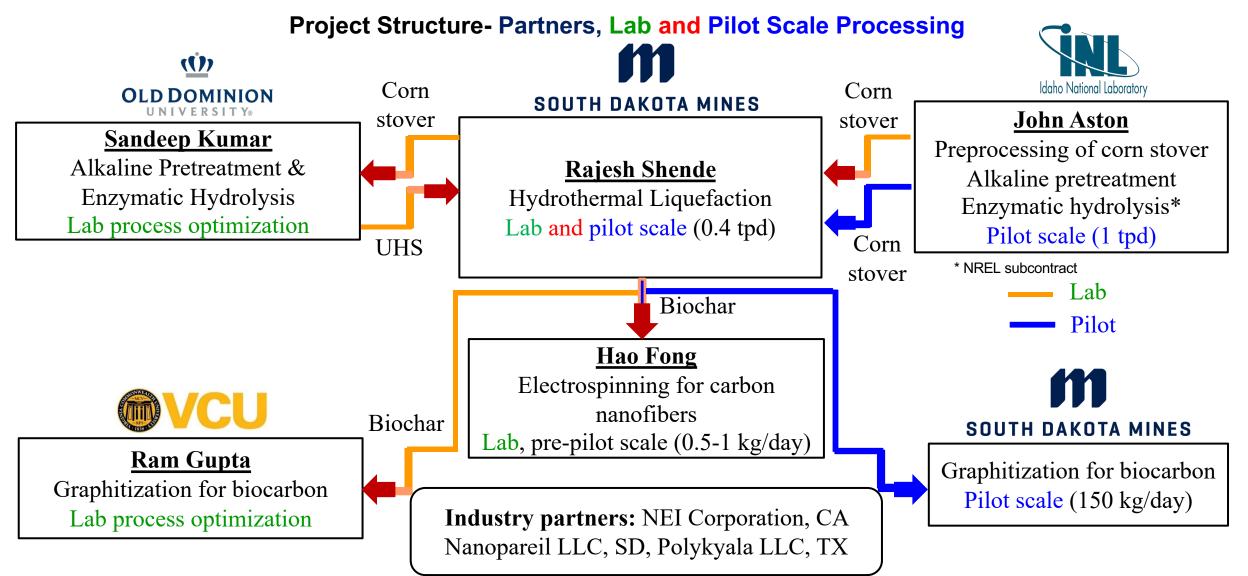
Development of conversion technologies for high value products from the waste solid residue derived during biochemical processing to generate revenue



1 - Approach



Project Management

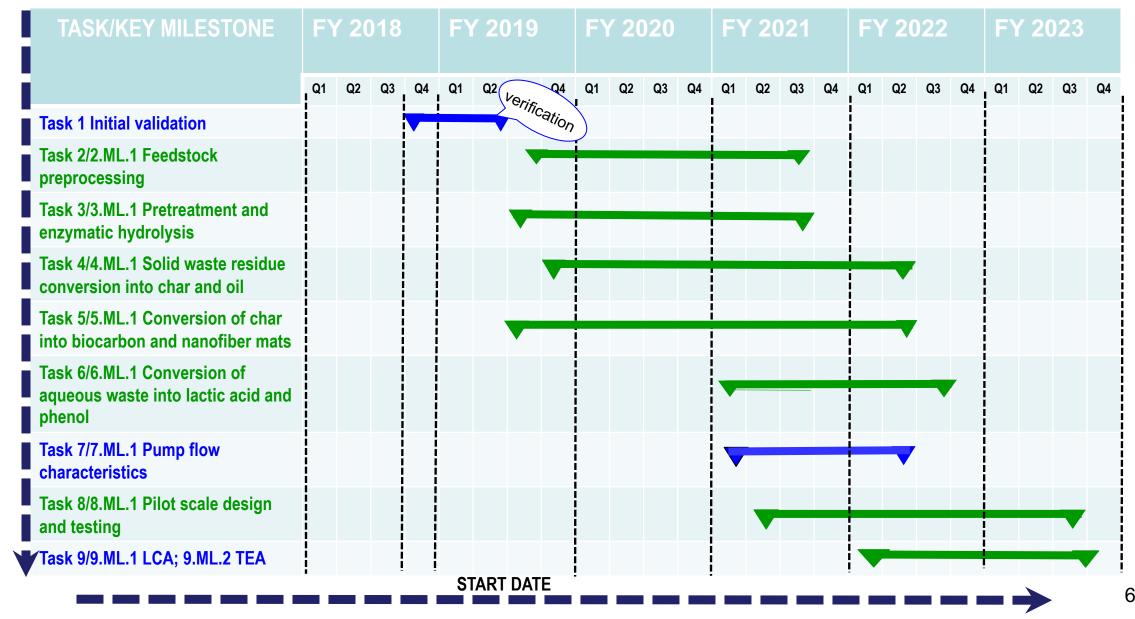


Demonstrate production of selected high value products from unhydrolyzed solid (UHS) waste generated during conventional biochemical processing of corn stover at a pilot scale level with 1 tpd throughput.

* Outcomes:

- □ High value products 1) graphitic carbon (referred as biocarbon) and 2) carbon nanofibers
- Quality of the carbon products in terms of specific surface area, porosity, and specific capacitance
- □ Potential revenues and the GHG emission.

Project Timeline



NOTE: GREEN lines and text indicate Active Status; BLUE lines and text indicate Inactive Status

Potential Challenges, Key Success Factors

Potential Challenges

- Yield and quality of selected high-value products at a pilot scale level (technical)
- HTL pilot plant design and fabrication (technical)
- Integrate processing at different locations (scheduling, transport of UHS and processed material) (Management)

Key success factors

- Quality assessment of high –value products; biocarbon and carbon nanofibers
- Evaluation and testing of the products by our industry partners
- Estimation of revenue from the high-value products and the GHG emission

2 - Progress and Outcomes

Preprocessing: Biomass Feedstock National User Facility

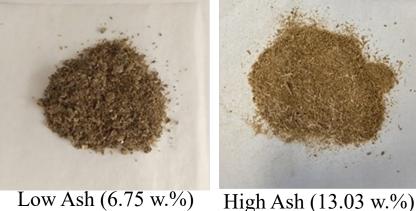
The BFNUF comprises:

- A full-scale, fully integrated Process Development Unit (PDU) with modular design with a capacity of 2 ton/hr for herbaceous biomass and 5 dry ton/hr for woody biomass.
- Single-stage or two-stage hammer mill grinding, knife milling, various separations screens and classifiers, in-line characterizations
- Various mechanical conveyors with Feedstock metering bin
- Pelleting (Bench, 1 ton/hr and 5 ton/hr)

Preprocessed corn stover and supplied to partners

The values determined for $Sieve_{10}$, $Sieve_{50}$, and $Sieve_{90}$ are within the target values ($Sieve_{10} > 0.1 \text{ mm}$ and $Sieve_{90} < 3 \text{ mm}$) the project.



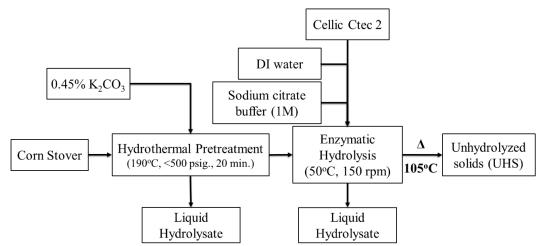




Pretreatment and Enzymatic Hydrolysis



INL chemical preprocessing system (CPS)



Optimized alkaline pretreatment process of corn stover

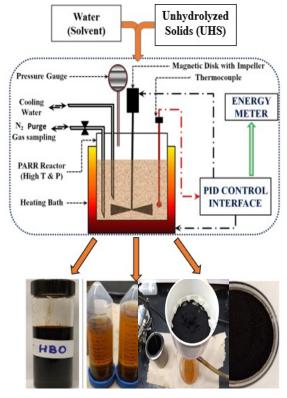
Operating parameters of the chemical preprocessing system and the corresponding maximum operational pressure achieved.

Material	Flow rate (kg/min)	Moisture Content (wt%)	Hopper Screw Speed (RPM)	Plug Screw Speed (RPM)	Maximum Operational Pressure (psig)
Corn stover knife milled to pass $\frac{1}{2}$ - inch screen	1	30	10	18	180
Corn stover crumbled to pass a 4-mm screen	1	50	10	18	120
Corn stover knife milled to pass $\frac{1}{2}$ - inch screen	1	30	10	24	160
Corn stover crumbled to pass a 4-mm screen	1	50	10	24	145
Corn stover knife milled to pass $\frac{1}{2}$ - inch screen	1	30	10	18	180
Corn stover crumbled to pass a 4-mm screen	1	50	10	18	140
Corn stover knife milled to pass $\frac{1}{2}$ - inch screen	1	30	10	24	135
Corn stover crumbled to pass a 4-mm screen	1	50	10	24	105

INL Chemical Preprocessing System (CPS)

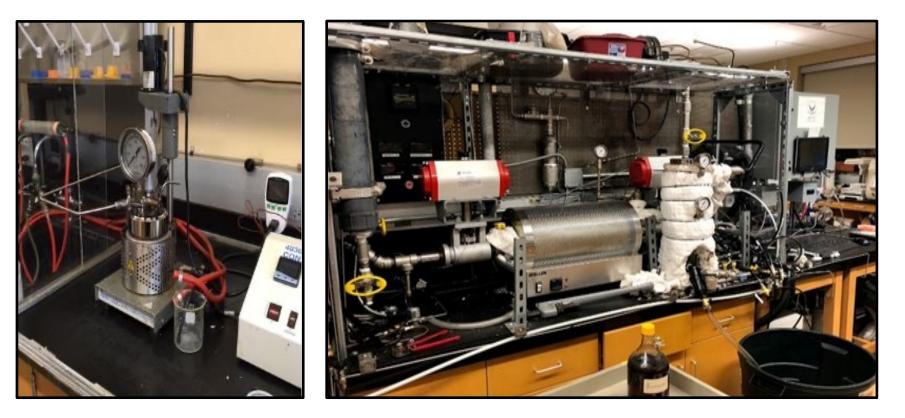
Unique pretreatment/preprocessing system Ambient $\leq T \leq \sim 200 \,^{\circ}$ C, Ambient $\leq P \leq \sim 200 \,^{\circ}$ psig Wide pH (0.5 – 13.5), numerous approved solvents Continuous up to 1 dry ton/day Batch reactors (3×10-L, non-agitated) Currently being configured to achieve 3.ML.1

Hydrothermal Liquefaction of UHS



Bio-oil Aq. Stream Hydrochar

Advantages - Direct wet processing (avoid drying costs), diverse biomass substrates, high value products, bio-oil with high HHV, energy recovery, better char characteristics



Processing	Char		НВО		Pher (GCN area	\mathbf{V}	ic wt%
	wt%	HHV (MJ/kg)	wt%	HHV (MJ/kg)			
Non-catalytic	36%	18	28%	30	18		7%
Catalytic	43%	17	23%	31	1:		4%

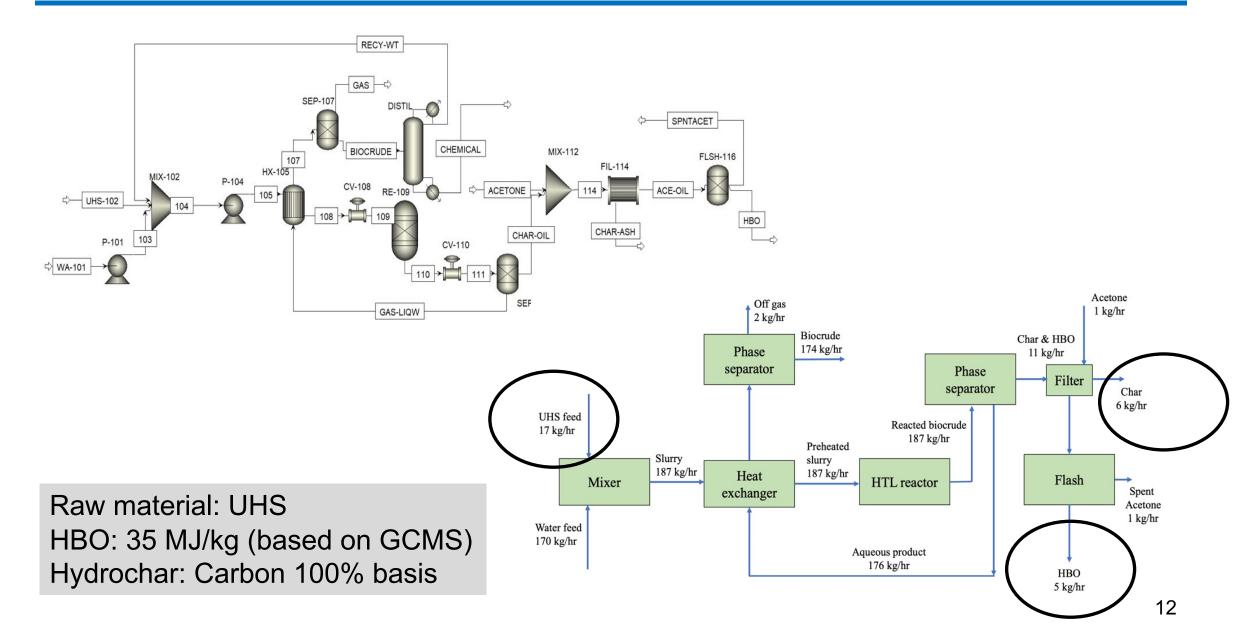
Hydrothermal Liquefaction of UHS Pilot Scale



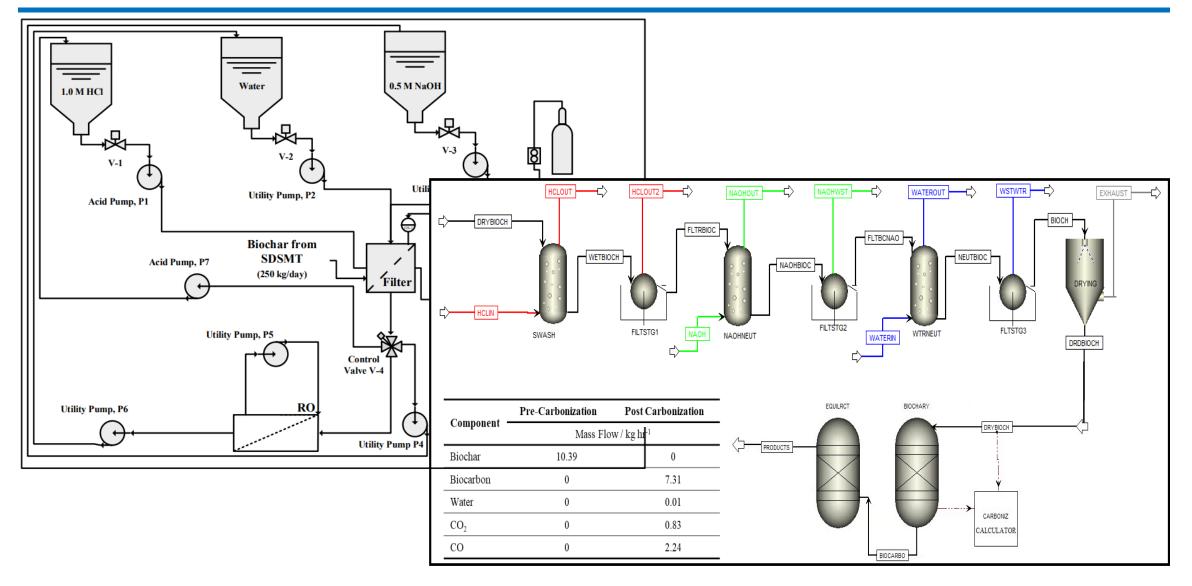
Designed and fabricated pilot scale HTL SS316 – 300 °C, 2200 psi rated (SDSMT) 11

motor)

HTL Modelling ASPEN PLUS



Hydrochar to Biocarbon – ASPEN Plus Modeling



At 10 ton/hour Capacity

Pilot Scale	Capacity Material of Cost per Company		Company			
Equipment	Capacity	Construction	unit	Company		
HCl Tank	200 gal	Plastic	\$2,321	Tamco		
Water Tank	500 gal	Plastic	\$1,263	Ace Roto-Mold		
				/ Den-Hartog		Manual
NaOH Tank	100 gal	Plastic	\$483	Tamco	Description	Calculations
Filtration Unit	85 kg	Hastelloy	\$80,000	Nano-Mag		
Box Furnace	4.2 cu ft	Multiple	\$35,297	Grieve	Equipment Costs	\$140,600
Reverse Osmosis	sis 7,000 gal day-1	Multiple	\$7,190	Crystal Quest	Installation Costs	\$370,000
(pump incl.)	7,000 gai uay	Multiple			Total Capital Costs	\$524,000
Acid Pump X 2	50 GPM	Metal	\$6,910	Magnatex Pumps	Utility Costs (yearly)	\$28,100
Utility Pump X 4	40.5 ft head	Metal	\$208	Little Giant	Operating Costs	
Total			\$140,582		(yearly)	\$249,000
	1 1	11 4 0 (0 0 /1			Yearly Return (Est.)	\$330,000
*		old at \$6-\$20/kg			Time to Payoff	7 yrs.
total char fee	dstock = 87,60	UKg				

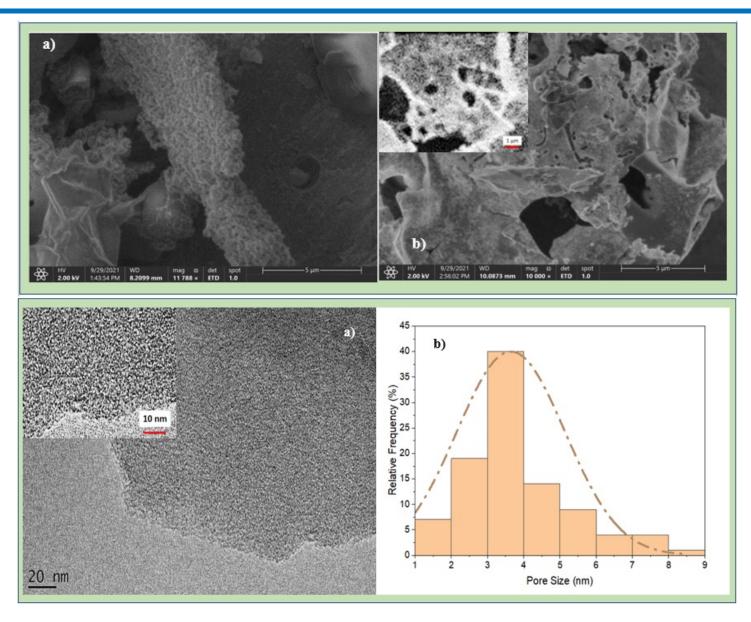
Biocarbon = 56,940 kg (C: avg. 85%)

Graphitized Carbon Characteristics

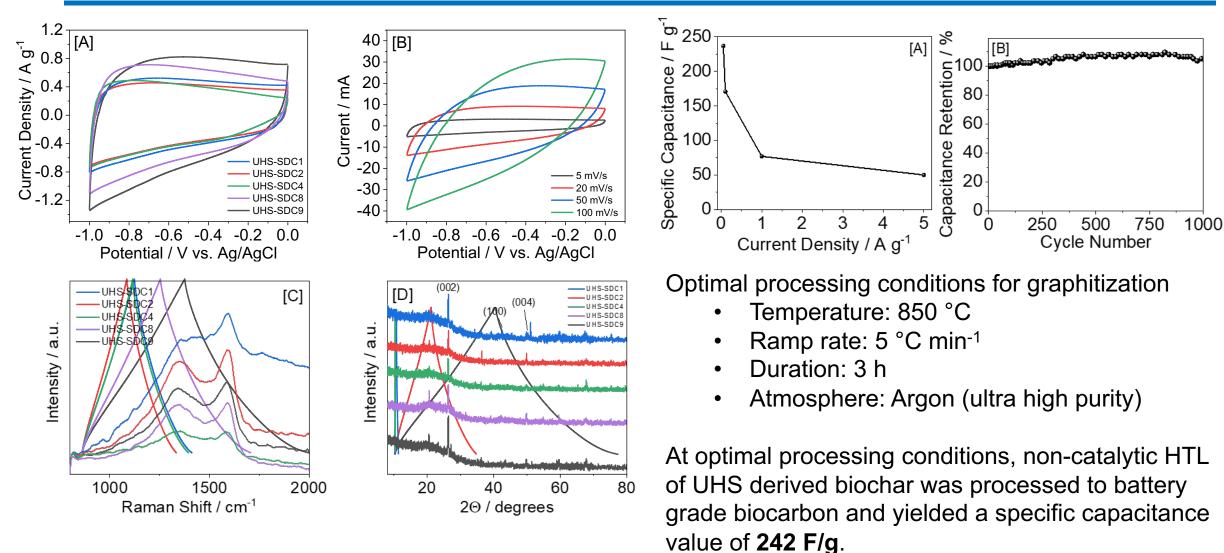
Quality – unique features

Raman microcrystalline size: 4 nm Specific surface area: >2000 m²/g Avg. pore size: 2.2 nm Pore volume: > 1.0 cm³/g

Amar et al., *Renewable Energy*, (2021) Shell et al, I&EC (2021) Amar et al., *Int. Journal of Energy Research* (2020) Choudhary et al., *Advanced Materials* (2017) Khang Huynh, MS Thesis (2022) Bharath Maddipudi, PhD current (2023) Huynh Khang, PhD current (2023)



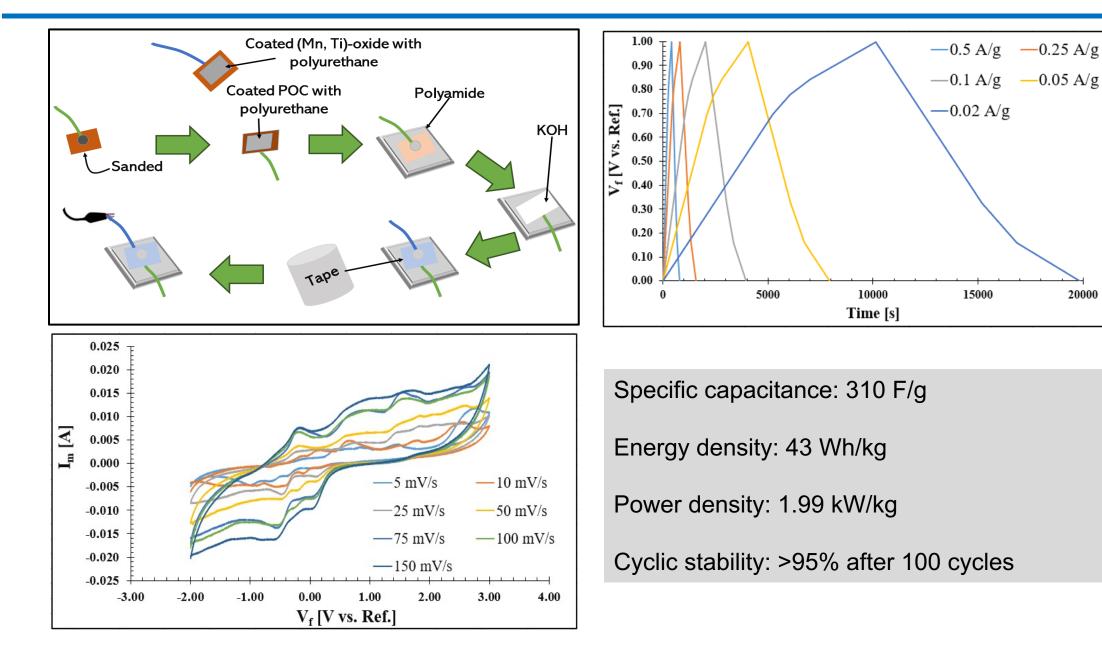
Graphitization of biochar to biocarbon



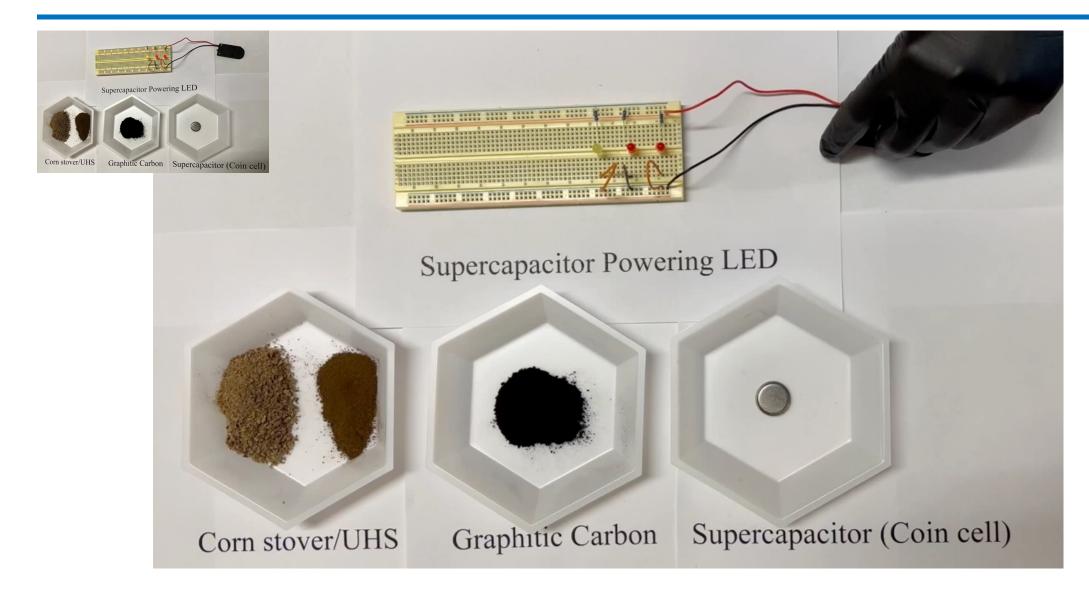
Electrochemical performance compares most closely with corn stover derived biocarbon (246 F g⁻¹) Jin et al. (2014).

Shell et al., *Bioresource Tech. Reports* (2021); Jin et al., *J of Analytical and Applied Pyrolysis* (2014); I&EC (2021)

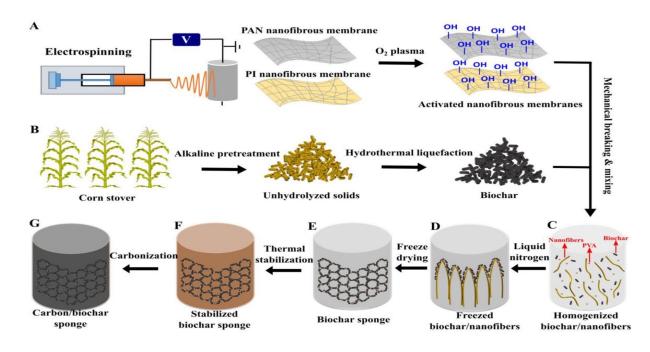
Asymmetric Supercapacitors



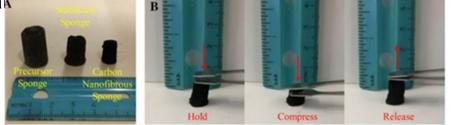
Carbon from Cornstover/UHS for Energy Storage



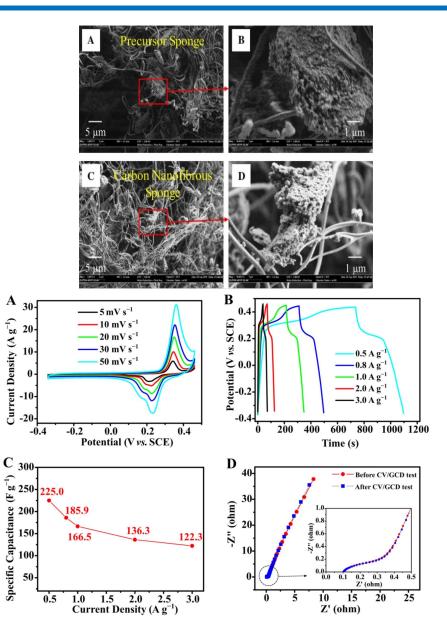
Process for Carbon Nanofibrous Sponge



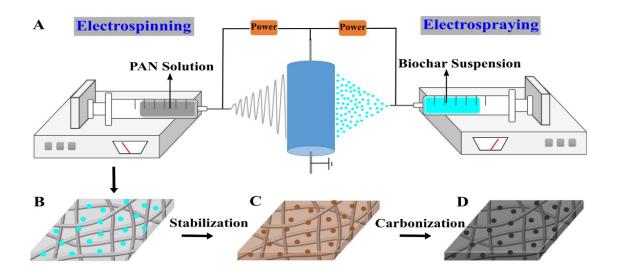
A. electrospun PAN and PI nanofibrous membranes, B.
Hydrothermally generated biochar particles, as well as the five steps in the fabrication of carbon nanofibrous sponge, C. Homogenization,
D. Fast freezing, E. Freeze drying, F. Thermal stabilization, G.

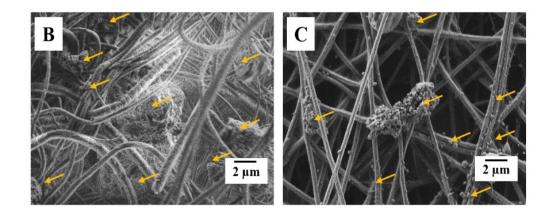


Tao et al., Advanced Fiber Materials (2020)

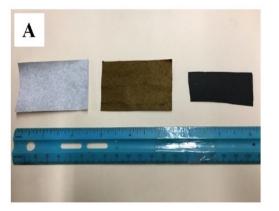


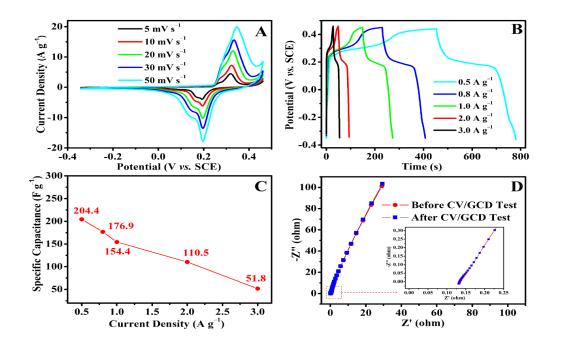
Preparation of Carbon Nanofibrous Felt





A. Simultaneous electrospinning and electrospraying, **B.** PAN/biochar precursor membrane, **C.** Stabilized membrane, **D.** Carbon nanofibrous felt.





3 – Impact (DOE/BETO)

- Directly impacting BETO's Mission of developing cost-effective technologies for high-value products from waste streams
- Integrated technologies developed here will generate additional revenue stream from the value-added products and help meeting BETO's 2022 target of \$3/gge.
- Expected positive impact on the GHG emission, reduction by > 50%.
- Impacting readily available market with increasing demand for the high value products (in 2028) biocarbon/ Graphitic carbon (\$25.7 billion) carbon nanofibers (\$1.7 billion), phenol (\$27.2 billion), lactic acid (\$2.43 billion)

3 – Impact (Partnerships and SD)

Positively impacted collaboration among 3 universities, INL, and SwRI with future collaborations with NREL, PNNL and industries.

Technology transfer to industries/commercialization- NEI corporation, Nanopareil LLC, Polykyala LLC and EIR at SDSMT ((https://www.sdsmt.edu/ Research/Economic-Development/Entrepreneur-In-Residence-Program/)

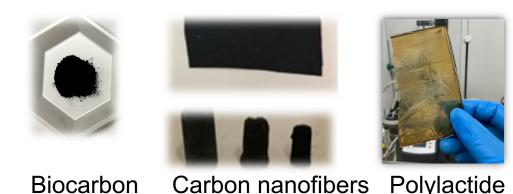
- Publications: 13, presentations 21 and invention disclosure 1
- Positively impacting South Dakota with bioprocessing pilot scale facilities initiative.

3 – Impact (Outreach and Education)

- IBR related educational workshop activities are being planned for the local high school and Native American students (Summer 2023) (https://www.sdsmt.edu/learn/).
- Training undergraduate students at SD Mines (CBE 467) to develop process engineering design for integrated bioprocessing. Learning outcomes from the current research activities will be included in the elective courses (CBE485/CBE485L Renewable and Sustainable Energy lecture/lab courses).
- Positively impacting GRA and salary support to the graduate students, post-docs, and research associates at collaborating institutions.
- COVID-19 pandemic situation affected project schedule. Extension was requested to complete the remaining pilot scale trials.

Summary

Products:



Total char feedstock = 87,600 kg Biocarbon = 56,940 kg (C: avg. 85%) Biocarbon can be sold at \$6-\$20/kg

Product for commercialization: A start-up company is interested in commercializing the Biocarbon for battery and supercapacitors

Hydrochar and lactic acid yields: 40 wt.% and 22 wt.%

Quality: Biocarbon (specific surface area ~2900 m²/g), specific capacitance 225 - 310 F g⁻¹ carbon nanofiber felt (up to 40 wt% hydrochar) and sponge (50% wt% hydrochar)

Response to FY-2021 Peer-Review Comments

Comment 1: Is this waste or a specialized solid product? Four products downselected to two? **Response**: The focus of the proposed work is to utilize the waste streams generated from the biochemical technology platform and convert it into high-value products. It is expected that the revenue stream generated from these high-value products will reduce the fuel cost to meet the objective of \$3/GGE. Originally, we proposed to derive four products—product 1, biocarbon; product 2, carbon nanofibers; product 3, phenol; and product 4, lactic acid—from the UHS recovered from the biochemical platform. Among these, products 1 and 2 are solid products, whereas products 3 and 4 are liquid side products. Product 1, biocarbon, which is also used for battery carbon electrodes, has outperformed in terms of specific capacitance (>300 F/g) and cyclic stability over 10,000 charging/discharging cycles. This type of carbon is currently being sold in the market at approximately \$20,000/ton.

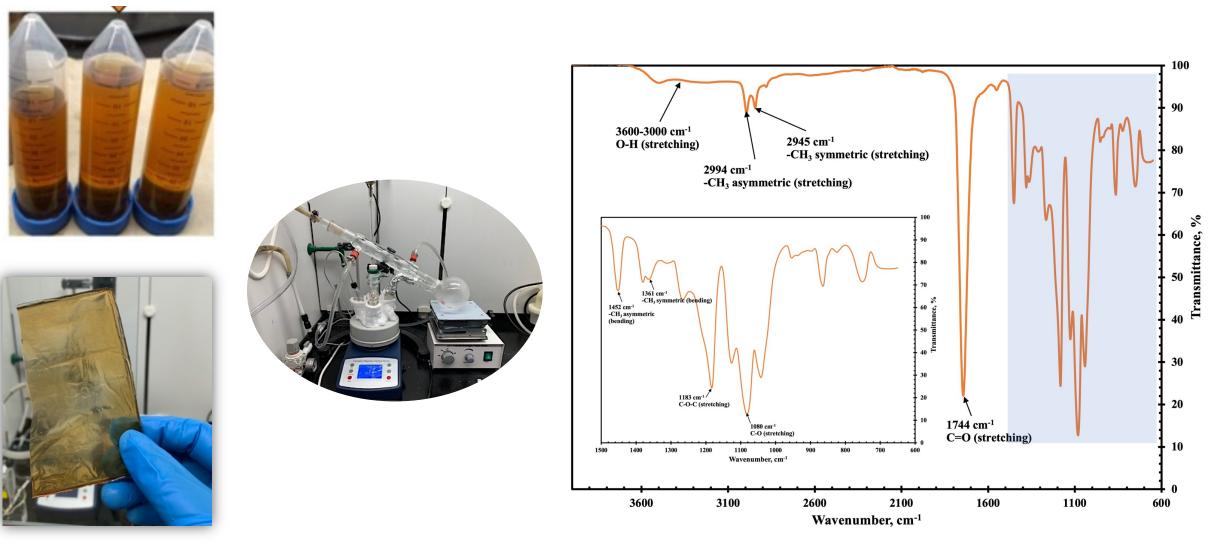
Weakness: This project is badly in need of industry advisors. The goals are not realistic, **Response:** Currently, my team is working with commercial entities such as NEI Corporation, Nanopareil LLC, Polykala LLC, and Lonza Group AG. In addition, we have contacted the Office of Economic Development at the SDSMT to share technology concepts at the Entrepreneur in Residence program. This program offers entrepreneurship activities, business formation and networking, market trend analysis, team building, IP, etc. Many CEOs will be on the South Dakota School of Mines and Technology campus, and it is the PI's intent to meet these chief executive officers and present the technology concept, products, and their quality, and to seek guidance on the commercialization strategy. Our industry advisors will help us set up the commercial goals, market assessment, investments, and profit estimation. There was a business competition, our group won the best CEO competition award for the carbon product.

Quad Chart Overview

 Timeline Project start date: 07/01/2018 Project end date: 01/14/2024 (tentative) 			 Project Goal Demonstrate production of high value products from solid waste generated during conventional biochemical processing at a
	FY22 Costed	Total Award	pilot scale level with 1 tpd throughput. End of Project Milestone
DOE Funding	(10/01/2021 – 9/30/2022) \$343,394	\$1,926,160	~80% Funding Mechanism FOA_DE-FOA-0001689_Optimization
Project Cost Share *		\$231,835	
	Project Start: 3 Project End: 5		 Project Partners* Virginia Commonwealth University Old Dominion University Idaho National Lab

*Only fill out if applicable.

Lactic Acid to PLA



Preprocessing: Biomass Feedstock National User Facility

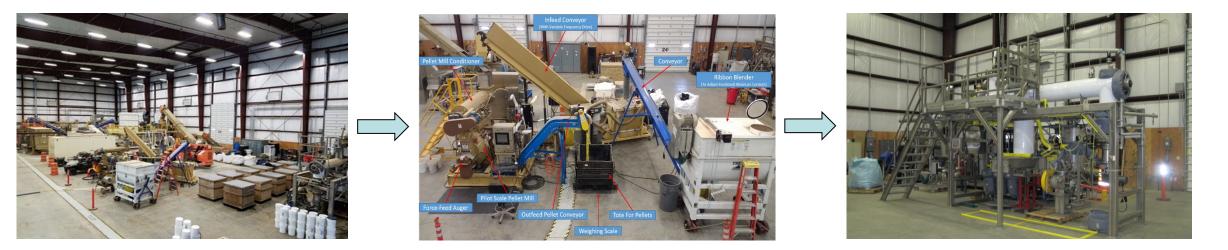


Table 3. Breakdown of modeled, estimated cost per ton of each unit operation for various preprocessing pathways using combined mechanical and chemical preprocessing [1,2,3].

Feedstock Pathways	Corn Stover Bale Preprocessing
Rotary Dryer (optional)	\$12.50
Conveyor	\$0.70
Mechanical Separation (optional sieving)	\$1.00
Air Classifier (optional)	\$2.81
Chemical Pretreatment and Drying	\$29.15
Size Reduction	\$17.21
Pelletization	\$7.68

References

- Hartley, D.S., Thompson, D.N., Cai, H. 2020. Woody Feedstocks 2019 State of Technology Report. Idaho National Laboratory. Idaho Falls, ID INL/EXT-20-57181. OSTI ID: 1607741
- Hu, H., Westover, T.L., Cherry, R. et al. Process Simulation and Cost Analysis for Removing Inorganics from Wood Chips Using Combined Mechanical and Chemical Preprocessing. *Bioenergy Research* 10, 237–247 (2017).
- Jaya Shankar Tumuluru, Kara G Cafferty, Kevin L Kenney. 2014. Technoeconomic Analysis of Conventional, High Moisture Pelletization and Briquetting Process. ASABE Annual meeting, Paper #141911360, Montreal, Quebec Canada July 13 – July 16, 2014.

Publications

- 1. Shell, Katelyn; Amar, Vinod; Bobb, Julian; Hernandez, Sergio; Shende, Rajesh; Gupta, Ram 2021. Graphitized Biocarbon Derived from Hydrothermally Liquefied Low-Ash Corn Stover. Ind. Eng. Chem. Res. 2022, 61, 392–402
- 2. Amar, V.S., Houck, J.D., Maddipudi, B., Penrod, T.A., Shell, K.M., Thakkar, A., Shende, A.R., Hernandez, S., Kumar, S., Gupta, R.B. and Shende, R.V., 2021. Hydrothermal liquefaction (HTL) processing of unhydrolyzed solids (UHS) for hydrochar and its use for asymmetric supercapacitors with mixed (Mn, Ti)-Perovskite oxides. *Renewable Energy*, *173*, pp.329-341.
- 3. Shell, K.M., Rodene, D.D., Amar, V., Thakkar, A., Maddipudi, B., Kumar, S., Shende, R. and Gupta, R.B., 2021. Supercapacitor performance of corn stover-derived biocarbon produced from the solid co-products of a hydrothermal liquefaction process. *Bioresource Technology Reports*, *13*, p.100625.
- 4. Li, X., Xu, T., Liang, Z., Amar, V.S., Huang, R., Maddipudi, B.K., Shende, R.V. and Fong, H., 2021. Simultaneous Electrospinning and Electrospraying for the Preparation of a Precursor Membrane Containing Hydrothermally Generated Biochar Particles to Produce the Value-Added Product of Carbon Nanofibrous Felt. *Polymers*, *13*(5), p.676.
- 5. Thakkar, A., Shell, K.M., Bertosin, M., Rodene, D.D., Amar, V., Bertucco, A., Gupta, R.B., Shende, R. and Kumar, S., 2021. Production of levulinic acid and biocarbon electrode material from corn stover through an integrated biorefinery process. *Fuel Processing Technology*, *213*, p.106644.
- 6. K. Shell, B. Maddipudi, V. Amar, A. Thakkar, R.V. Shende, S. Kumar, and R.B. Gupta **Production of Supercapacitor Carbon Electrodes from Corn-stover via a Facile Thermal Activation**. *TechConnect briefs (2021)*
- 7. Amar, V.S., Houck, J.D. and Shende, R.V., 2020. Catalytic HTL-derived biochar and sol-gel synthesized (Mn, Ti) -oxides for asymmetric supercapacitors. International Journal of Energy Research, 44(15), pp.12546-12558.
- 8. Amar, V., Houck, J., Maddipudi, B. and Shende, R., 2020. NiFe2O4/ZrO2 Core-Shell Nanoparticles for Hydrogen Generation from Thermochemical Water-Splitting Process. *J Catal Chem Eng Adv*, 7(1), p.105.
- 9. Jaswal, R., Shende, A., Nan, W., Amar, V. and Shende, R., 2019. Hydrothermal liquefaction and photocatalytic reforming of pinewood (pinus ponderosa)-derived acid hydrolysis residue for hydrogen and bio-oil production. *Energy & Fuels*, *33*(7), pp.6454-6462.
- 10. Amar V.S., Shende A., and Shende R.V., 2019. **BTMO catalyzed hydrothermal liquefaction of lignocellulosic biomass**. *TechConnect Briefs*, pp.195-198.
- 11. V.S. Amar, J.D. Houck, and R.V. Shende, "Sol-gel synthesis of mesoporous MnTi- oxides for Supercapacitors," Supercapacitors: TechConnect Briefs (2019).

Publications

- 12. Khang T. Huynh, Vinod S. Amar, Bharathkiran Maddipudi, Anuradha R. Shende, Rajesh V. Shende **KOH Activated Hydrothermal Liquefaction** (HTL) Derived Hydrochar from Corn Stover as Electrode Material for Asymmetric Supercapacitor (Under revision).
- 13. Huynh, K., Maddipudi, B., Amar, V., Houck, J., & Shende, R. **HTL Derived Biochar for Supercapacitor Electrodes**. In TechConnect World Innovation Conference, TechConnect Briefs, pp. 184-187, 2021
- 14. Huynh, K., Maddipudi, B., Amar, V., Bauer, G., Shende, A., & Shende, R. Asymmetric supercapacitor fabricated with ferrite/CHTL-derived hydrochar electrodes. In TechConnect World Innovation Conference, TechConnect Briefs, pp. 130-133, 2022.
- 15. Valorization of corn stover derived unhydrolyzed solids to value added products via non-catalytic hydrothermal liquefaction at non-catalytic optimized processing conditions, Chemical Engineering Journal, February 2023 (under preparation).
- 16. Optimization of catalytic hydrothermal liquefaction of lignin rich unhydrolyzed solids and extraction of value-added products. Bioresource Technology, February 2023 (under preparation).

Awards

1. Secured 1st place in SD Mines CEO Business plan competition with proposed plan in turning corn stover into a valuable form of biocarbon. Ref: https://www.sdsmt.edu/News/2022-CEO-Business-Competition/#.Y_Miuy-B1qs

Presentations

- Bharathkiran Maddipudi, Khang Huynh, Vinod Amar, Anuradha Shende, Grant Bauer, Rajesh Shende, Hydrothermal co-liquefaction of crop residue and plastic waste for sustainable production of oil and hydrochar, TechConnect World Innovation Conference, June 13-15, 2022, Washington, DC, USA.
- Vinod Amar, Bharathkiran Maddipudi, Grant Bauer, and Rajesh V. Shende, Core-shell and immobilized ferrite nanoparticles for CO2 free sustainable hydrogen production via thermochemical water-splitting process, TechConnect World Innovation Conference, June 13-15, 2022, Washington, DC, USA.
- 3. Khang Huynh, Bharathkiran Maddipudi, Vinod Amar, Anuradha Shende, Rajesh Shende, **Asymmetric supercapacitors fabricated with** ferrite/CHTL-derived hydrochar electrodes, TechConnect World Innovation Conference, June 13-15, 2022, Washington, DC, USA.
- 4. Khang Huynh, Bharath Maddipudi, Anuradha Shende, Rajesh Shende, Effect of surfactant on HTL derived hydrochar physicochemical characteristics 2022 AIChE Annual Meeting, Nov 13-18, 2022.
- 5. Khang Huynh, Bharath Maddipudi, Anuradha Shende, Rajesh Shende **Glycerol as a co-solvent for hydrothermal liquefaction of corn stover** 2022 AIChE Annual Meeting, Nov 13-18, 2022.
- 6. Khang Huynh, Bharath Maddipudi, Anuradha Shende, Rajesh Shende **Sol-gel synthesis of doped (Mn, Ti)-oxides for asymmetric supercapacitors** 2022 AIChE Annual Meeting, Nov 13-18, 2022.
- 7. Bharathkiran Maddipudi, Khang Huynh, Vinod Amar, Anuradha Shende, and Rajesh Shende **Catalytic hydrothermal liquefaction of lignocellulosic biomass for fuels and value-added products.** 2022 AIChE Annual Meeting, Nov 13-18, 2022.
- 8. Bharathkiran Maddipudi, Khang Huynh, Paiton Mueller, Rajesh Shende. **Surfactant assisted hydrothermal liquefaction of corn stover.** 2022 AIChE Annual Meeting, Nov 13-18, 2022.
- 9. Bharathkiran Maddipudi, Khang Huynh, Vinod Amar, Katelyn Shell, Anuj Thakkar, Anuradha Shende, Sergio Hernandez, John Aston, Sandeep Kumar, Ram Gupta, Rajesh Shende **TEA/LCA of integrated biochemical and hydrothermal processing of corn stover for fuels and high value products** 2022 AIChE Annual Meeting, Nov 13-18, 2022.
- Bharathkiran Maddipudi, Vinod S. Amar, Khang Huynh, Anuj Thakkar, Katelyn Shell, Runzhou Huang, Sergio Hernandez, Sandeep Kumar, Ram Gupta, Hao Fong, Anuradha Shende, and Rajesh Shende, Integrated Biochemical and Hydrothermal Processing of Corn Stover for Fuels and High Value Products, AIChE Annual Meeting, Nov 7 – Nov 11, 2021, Boston, MA.
- 11. Max Lampert, Bharathkiran Maddipudi, Vinod S. Amar, Anuradha Shende, and Rajesh Shende, **Evaluation of Ultrasonic Pretreatment on Catalytic Hydrothermal Liquefaction of Woody Biomass**, AIChE Annual Meeting, Nov 7 – Nov 11, 2021, Boston, MA.

Presentations

- 13. Vinod S. Amar, Bharath Maddipudi, Anuradha Shende, and Rajesh Shende, **Thermally Induced Two-step Chemical Looping Process for Hydrogen, Syngas, and Ammonia Production**, AIChE Annual Meeting, Nov 7 – Nov 11, 2021, Boston, MA.
- 14. Khang Huynh, Bharathkiran Maddipudi, Vinod S. Amar, Anuradha Shende, and Rajesh Shende, Fabrication and Testing of Asymmetric Supercapacitors Fabricated with (Mn, Ti)-Oxides and CHTL Derived Hydrochar/Graphene, AIChE Annual Meeting, Nov 7 Nov 11, 2021, Boston, MA.
- 15. Khang Huynh, Bharathkiran Maddipudi, Vinod S. Amar, Anuradha Shende, and Rajesh Shende, Adsorption Kinetics Studies of Catalytic HTL Derived Biochar, AIChE Annual Meeting, Nov 7 Nov 11, 2021, Boston, MA.
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