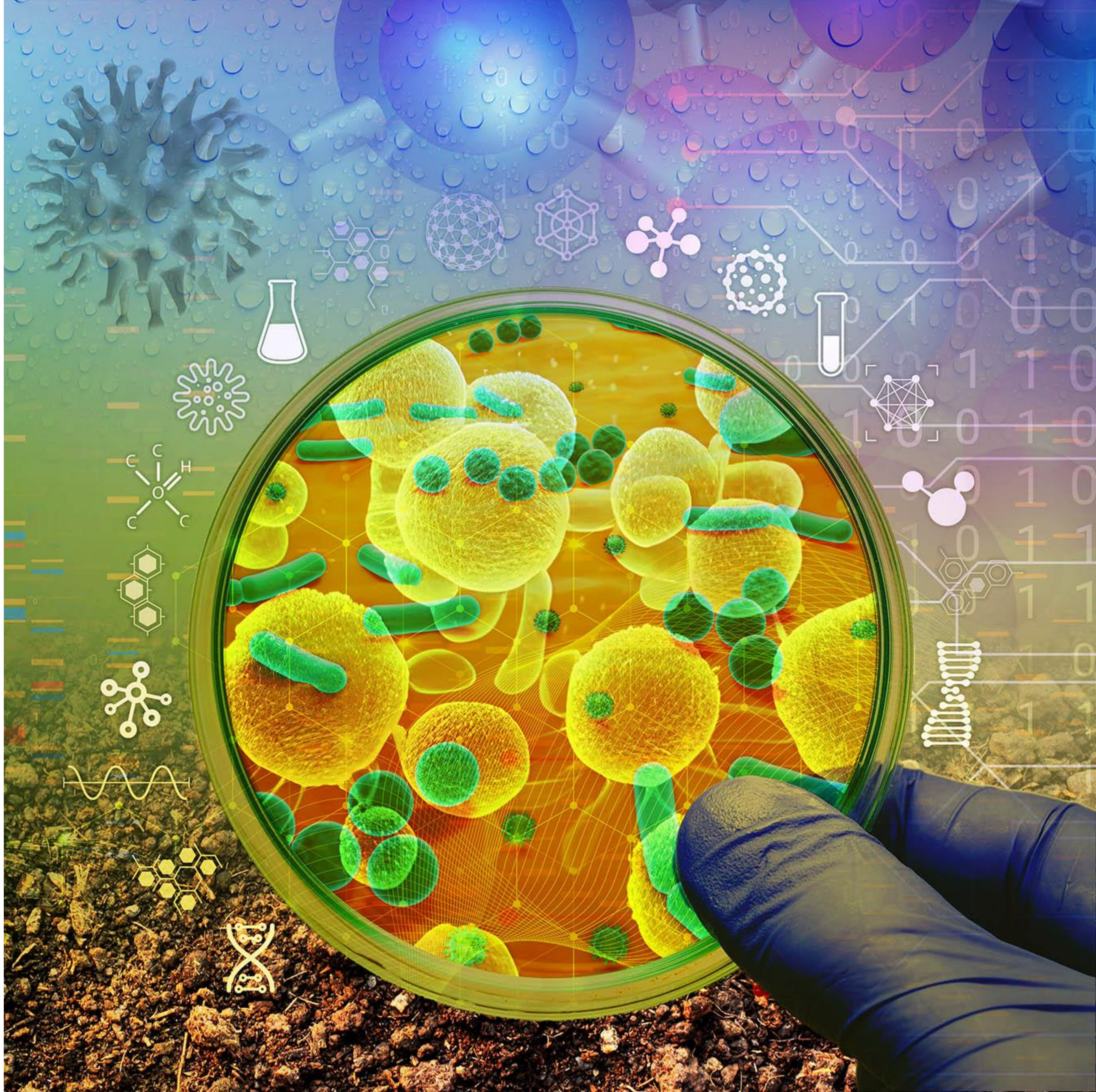
PNNL-FWP-78606 (DOE)

Integrated Capture and Conversion of CO_2 into Materials (IC^3M): Pathways for Producing CO_2-Negative Building Composites

Satish K. Nune

David J Heldebrant

U.S. Department of Energy
National Energy Technology Laboratory
Carbon Management Project Review Meeting
August 28 – September 1, 2023



Project Budget Overview

36-month Effort

- **BP1 - 09/30/2022,**
- **BP2 10/1/2022 - 09/30/2023**
- **BP3 10/1/2023 - 09/30/2024.**

DOE: $2.7M in Federal funds

- **(FY1 $841K, FY2 $980K, FY3 $885K)**

Cost Share: $540K, SoCalGas

Flavio da Cruz

Ethan Simonoff

Siari Sosa

Ron Kent

Project Team and Roles

Kristy Hahn

NETL PM

Satish K. Nune (PM and Co-PI)

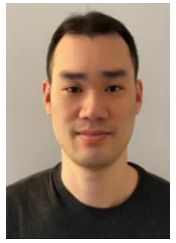
David J. Heldebrant (PI)

Operando Spectroscopy
Catalysis & Kinetics

Jaelynne King

Nick Nelson

Katarzyna Grubel



Wontae Joo

John Linehan

Composites & Solvents

Md. Reza-E-Rabby

Keerti Kappagantula

Deepika Malhotra

Aditya Nittala

Yelin Ni

Jose Ramos

TEA/LCA

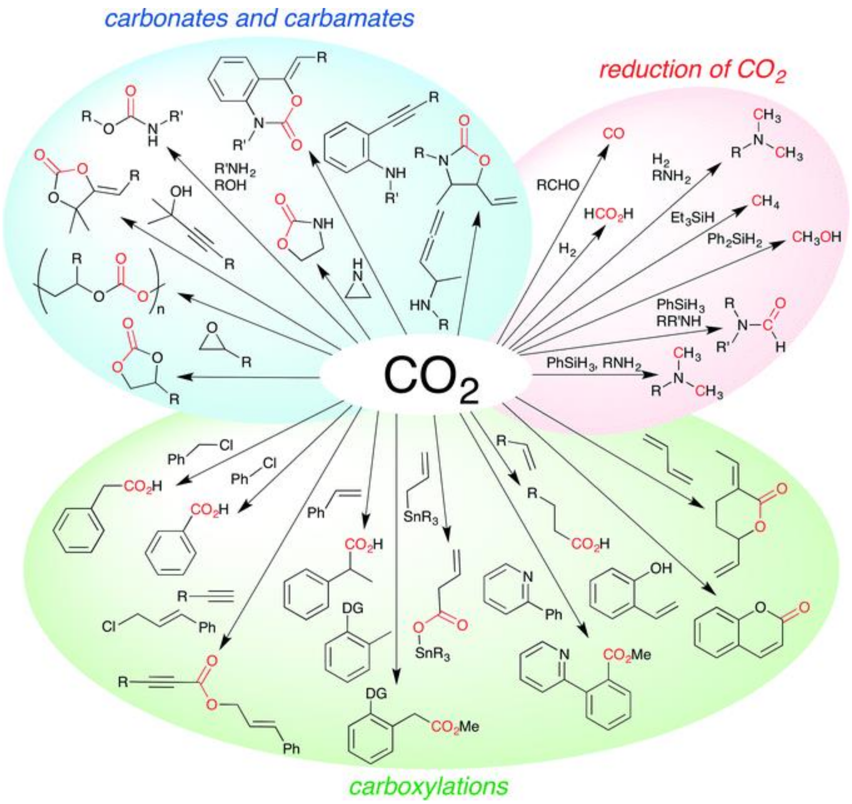
Yuan Jiang

Lesley
Snowden-Swan

Francesca
Pierobon

Integrated Capture and Conversion of CO_2 into Materials (IC^3M); A Multi-Product Platform for CCUS

Our Vision: To make a CO_2 capture unit a conversion plant like Shell's Pearl GTL facility, making materials from CO_2.



Near term targets
carbon-neutral fuels and chemicals:
CH_3OH, CH_4

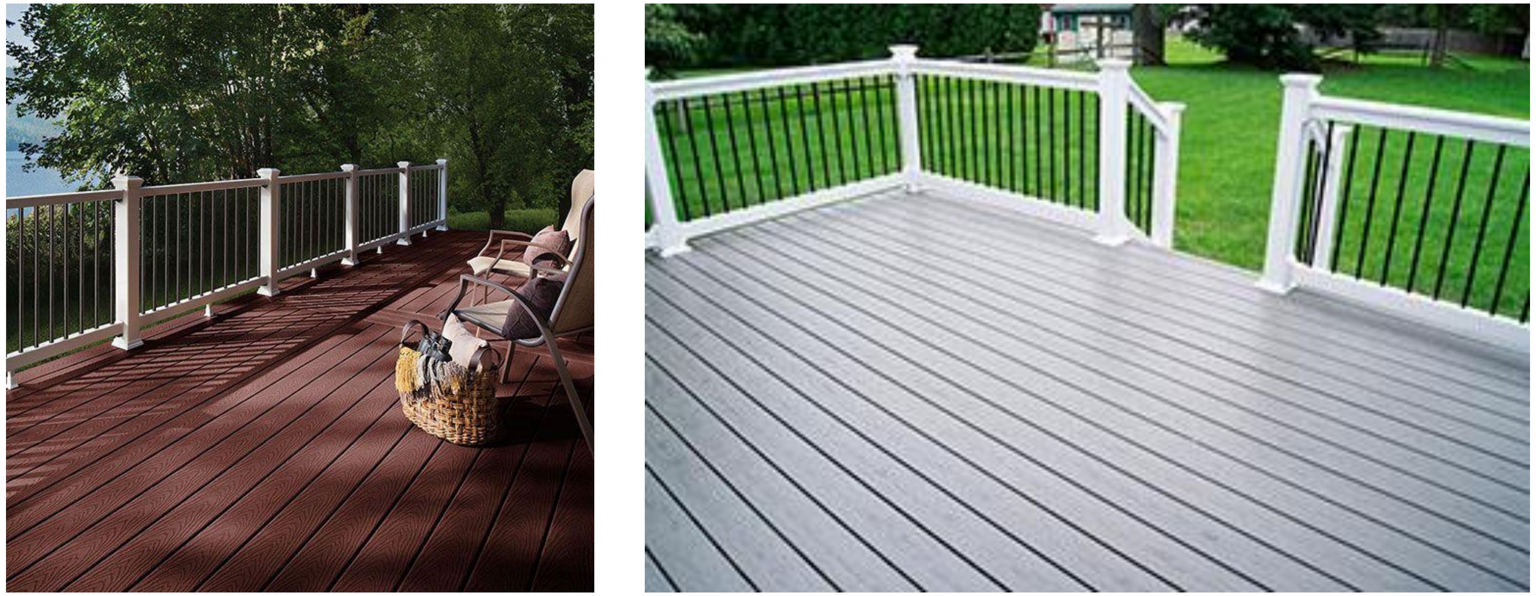
Intermediate term targets
carbon-negative building materials:
CO_2LIG

Long term targets
Mineralization materials:
$CaCO_3$ or $MgCO_3$

Bricket et al. Chem. Sci., **2022**,13, 6445-6456

Project Goal: Negative-Emission Composites

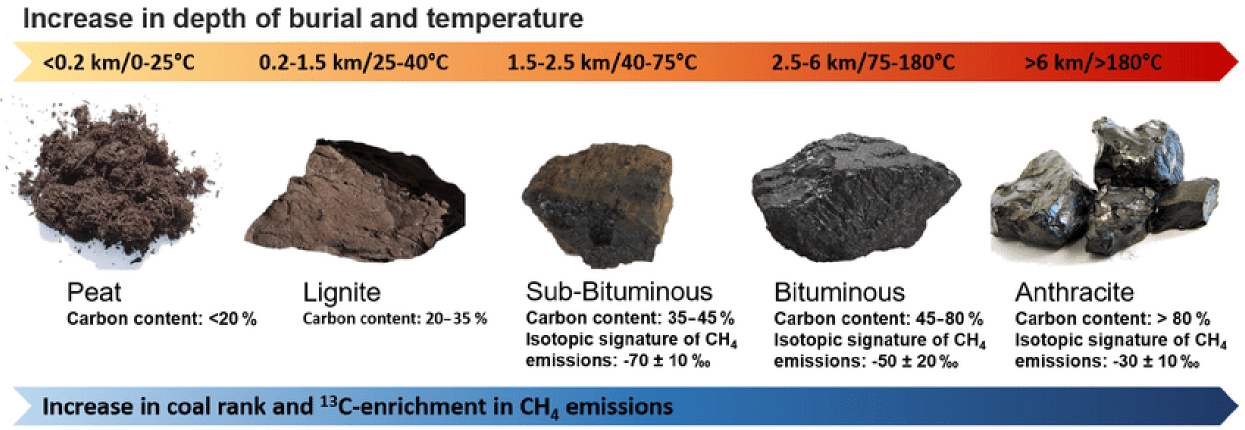
Large-volume biopolymers may be large volume CO_2 sinks, while being economically viable.



- 3.55 billion linear board feet of synthetic decking in the US per year.
- Annual $2.8 billion market in US.
- Made from wood flour (shavings) and plastic (~50 wt.% each).

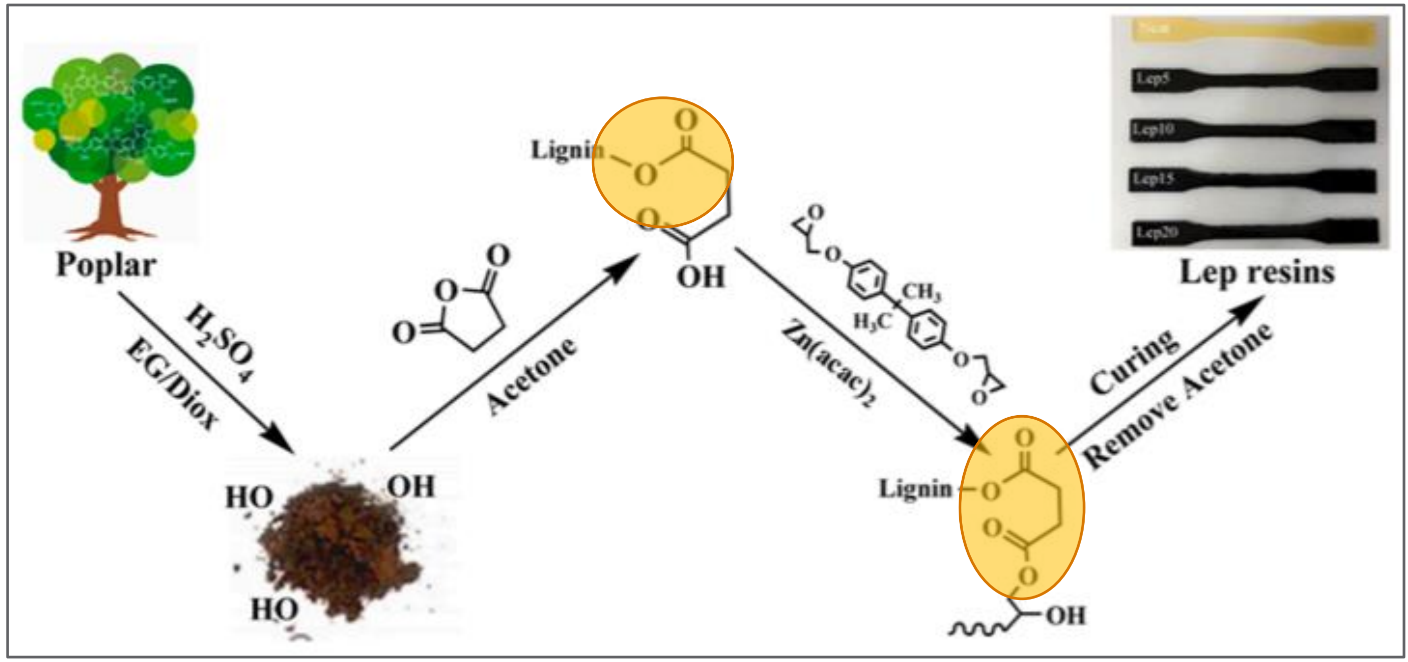
- Lignin is complex organic polymer that forms key structural materials in the support tissues of most plants.
- Lignite is a combustible sedimentary rock formed from naturally compressed peat.

Lignin

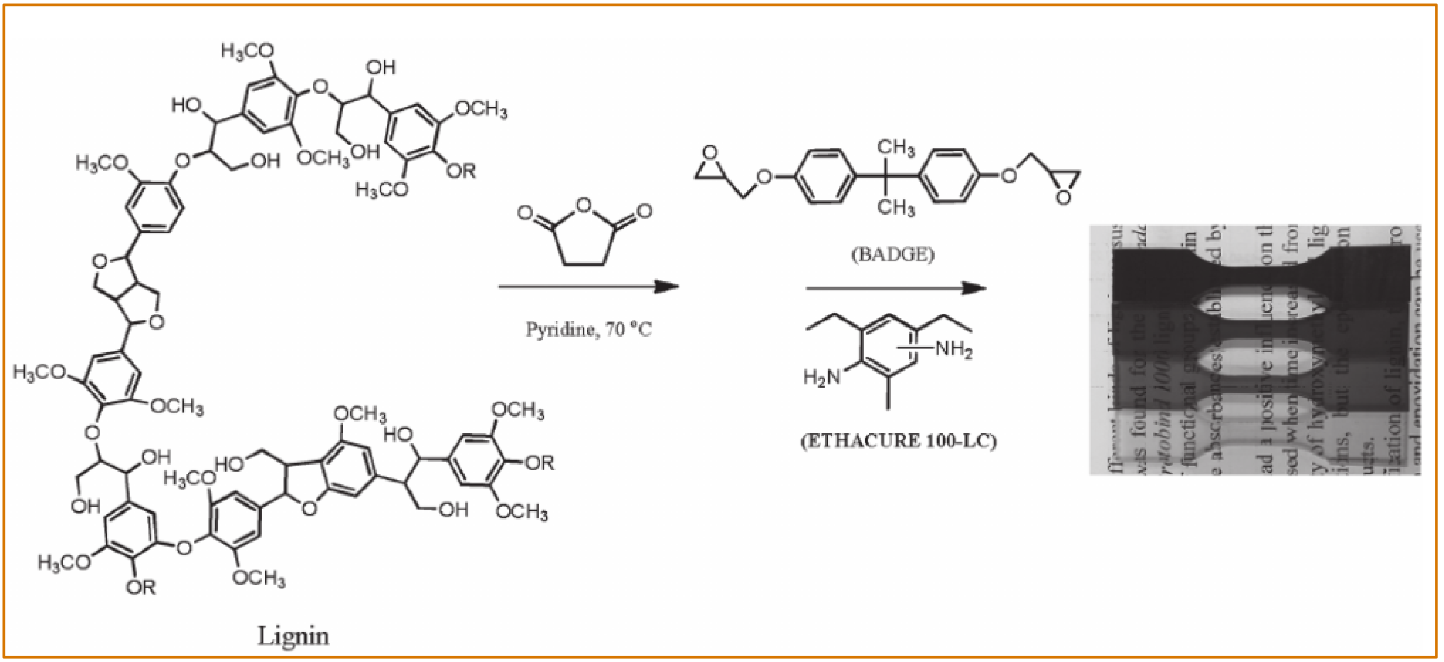


Lignin (Lignite) Filler Requires Upgrading

Lignin and lignite are strong, cheap, chemically durable filler materials, but they cannot bind well in polymer matrixes, requiring chemical modification.



Macromol. Mater. Eng. 2017, 302, 1700341



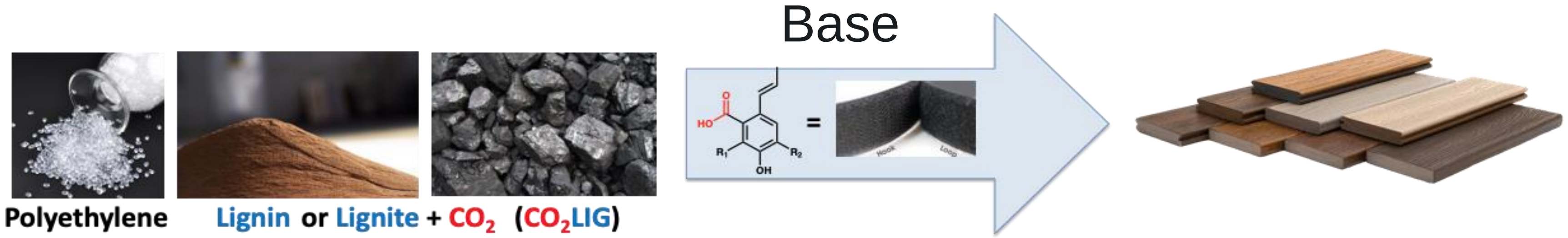
European Polymer Journal 150 (2021) 110389

- Currently a co-additive such as Maleic Anhydride Polyethylene (MAPE) is added
- Carboxylation achieved on phenolic hydroxyls instead on aromatic backbone.
  - C-O bonds are not very stable undergo hydrolysis.

- Addition of additives add cost and complexity to composite manufacturing

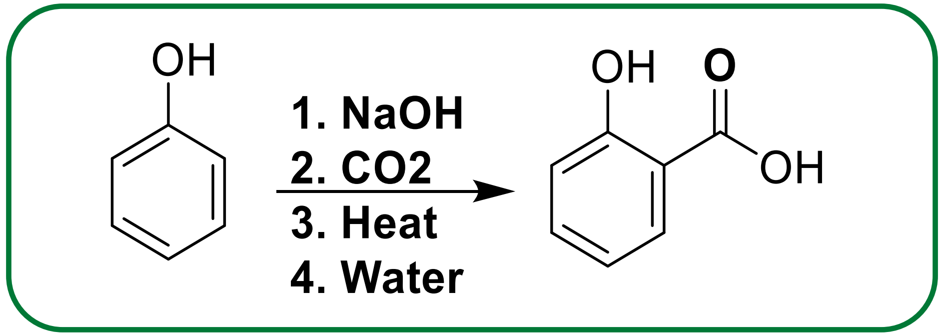
Upgrading Lignin (Lignite) via CO_2 Fixation

CO_2 can provide the "Velcro" to help lignin and lignite bind strongly to polymer matrixes.



Production of Salicylic Acid for Aspirin

Kolbe-Schmidt Reaction



- Upgrading lignin (with CO_2) results in chemically durable composite fillers (C-C bond, utilizes CO_2 in its entirety)
- With 5 wt. % carboxylation & 50-70 % filler ratio for producing Lignin-Polymer composites, we could sequester about 250 thousand metric ton/ year
- Equivalent to emissions from 54,000 cars/year in the US / 1.86 M cars/year globally (4.6 metric ton CO_2/car/year, from EPA)

Carboxylation of Lignin and Lignite

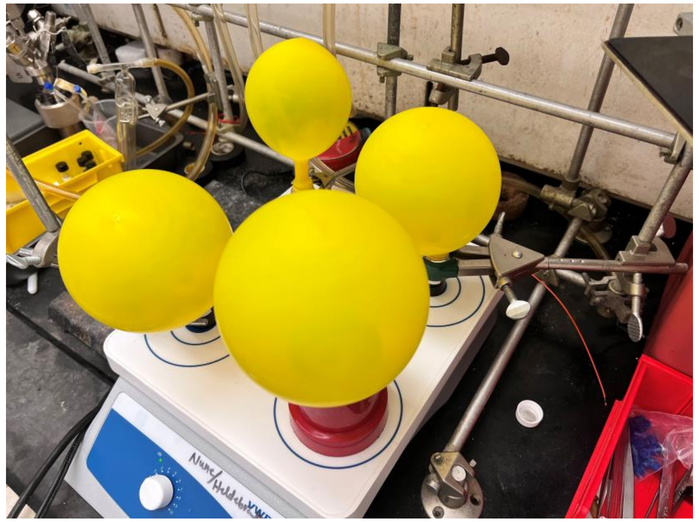
- Achieved carboxylation on lignin and lignite using NaOH/ DBU.

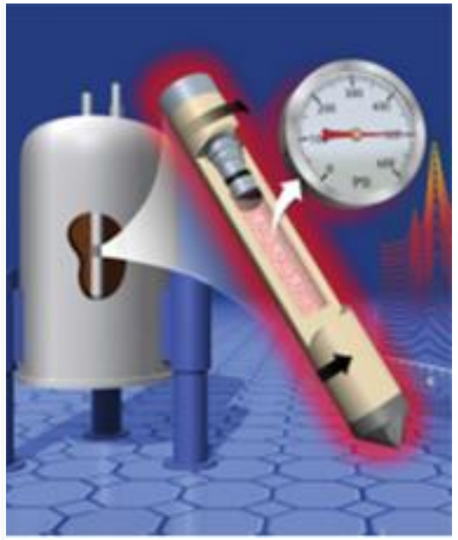
CO_2

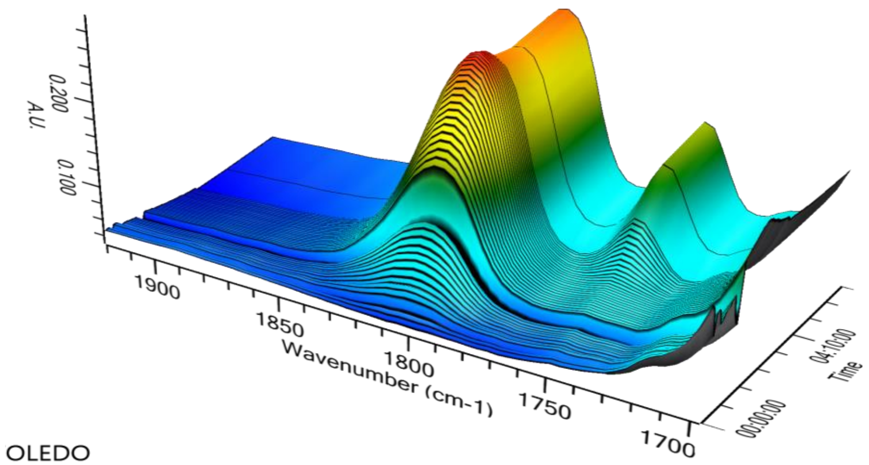
Base

Carboxylated Lignin Separation

Carboxylated Lignin



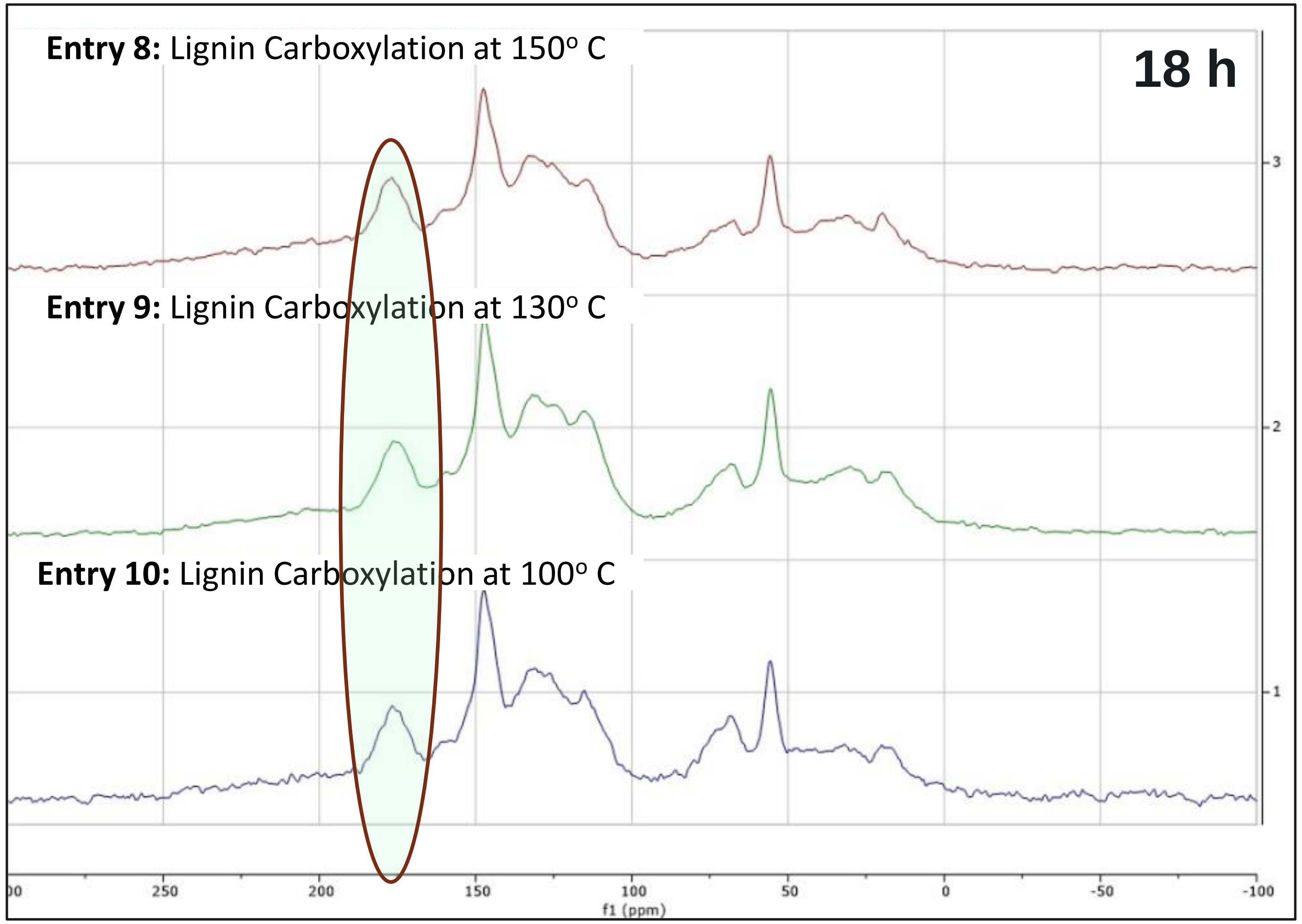




OLEDO

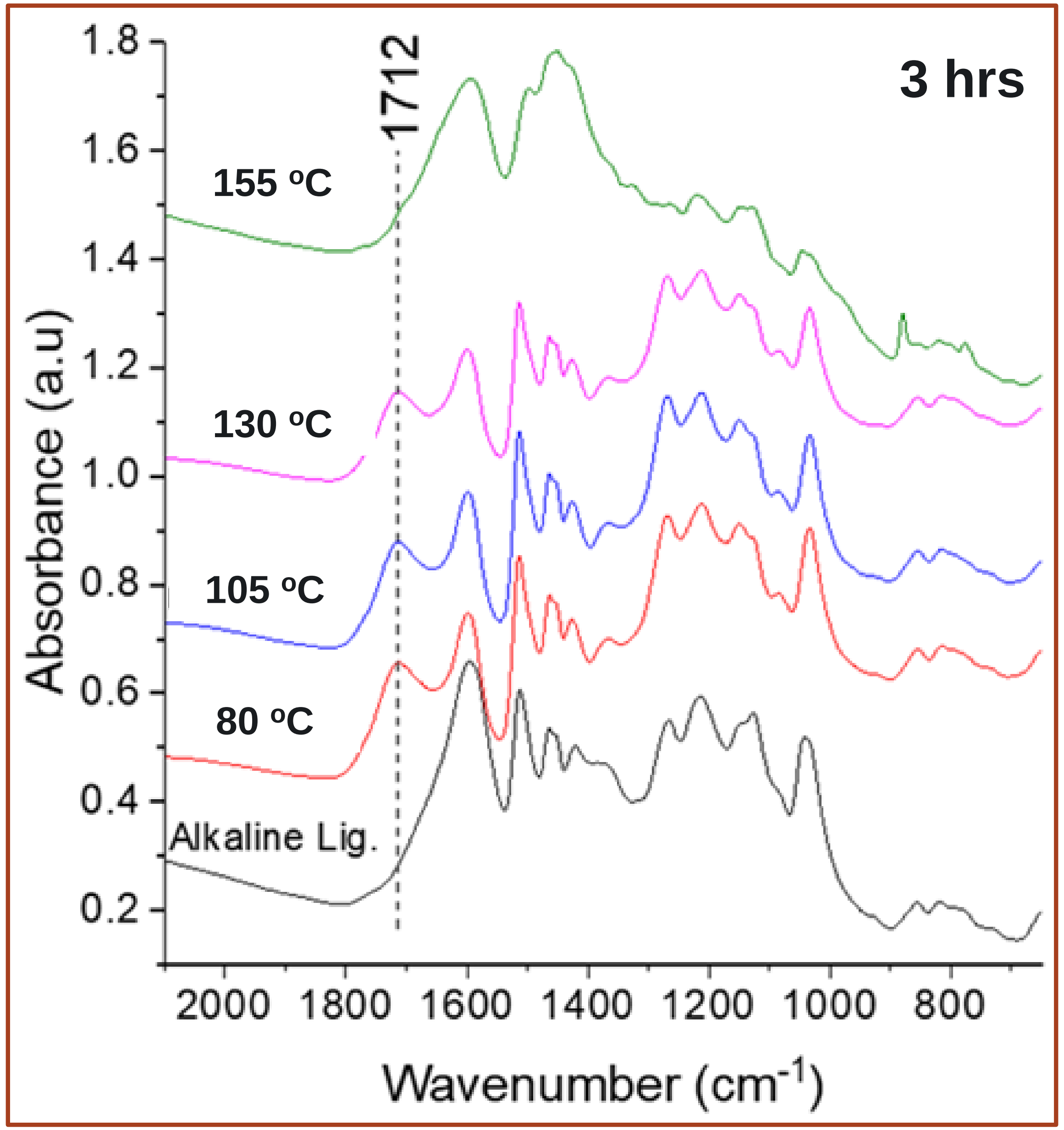
Carboxylation of Sodium Lignin sulfonate

Lignin (Sodium Lignin sulfonate; SLS) carboxylation using inorganic base (NaOH) at three different temperatures (500 mg scale)



- ➢ ^{13}C-SS NMR has peaks around 175-176 ppm indicative of carboxylation even at 100° C.
- ➢ These results suggests that the carboxylation reaction was successful at 500 mg scale, warranting to study this reaction at large scale (100 g scale).

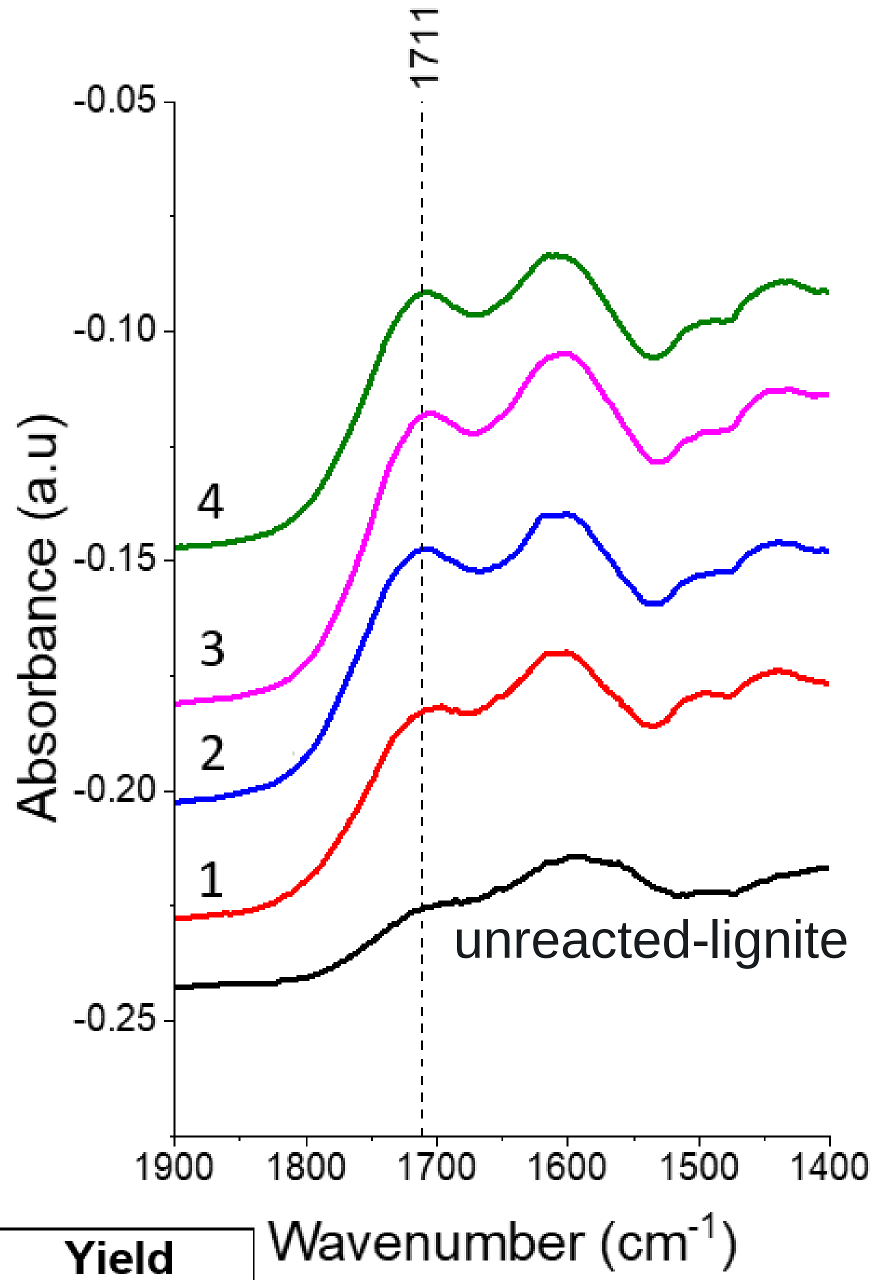
Carboxylation of Alkaline Lignin



- ➢ The IR spectrum of the acid treated alkaline lignin after reaction confirmed carboxylation can be achieved at <u>**80 ºC in 3 h**</u>

Carboxylation of Lignite

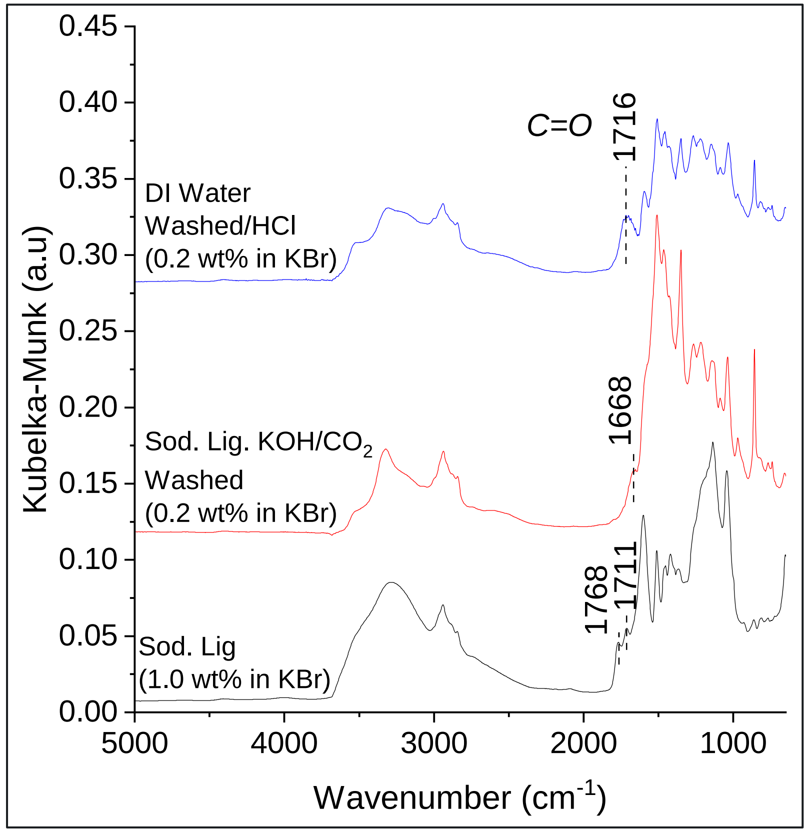
- ➢ Two different lignites DEC25, Buelah Zap Lignite, and DEC26, Wyodak Sub-bitiminous Coal was tested for carboxylation.
- ➢ Four different conditions was used to test the carboxylation of DEC25, Buelah Zap Lignite.
- ➢ All samples displayed increased intensity at 1711 cm^{-1} when it was compared to the spectrum of unreacted-lignite

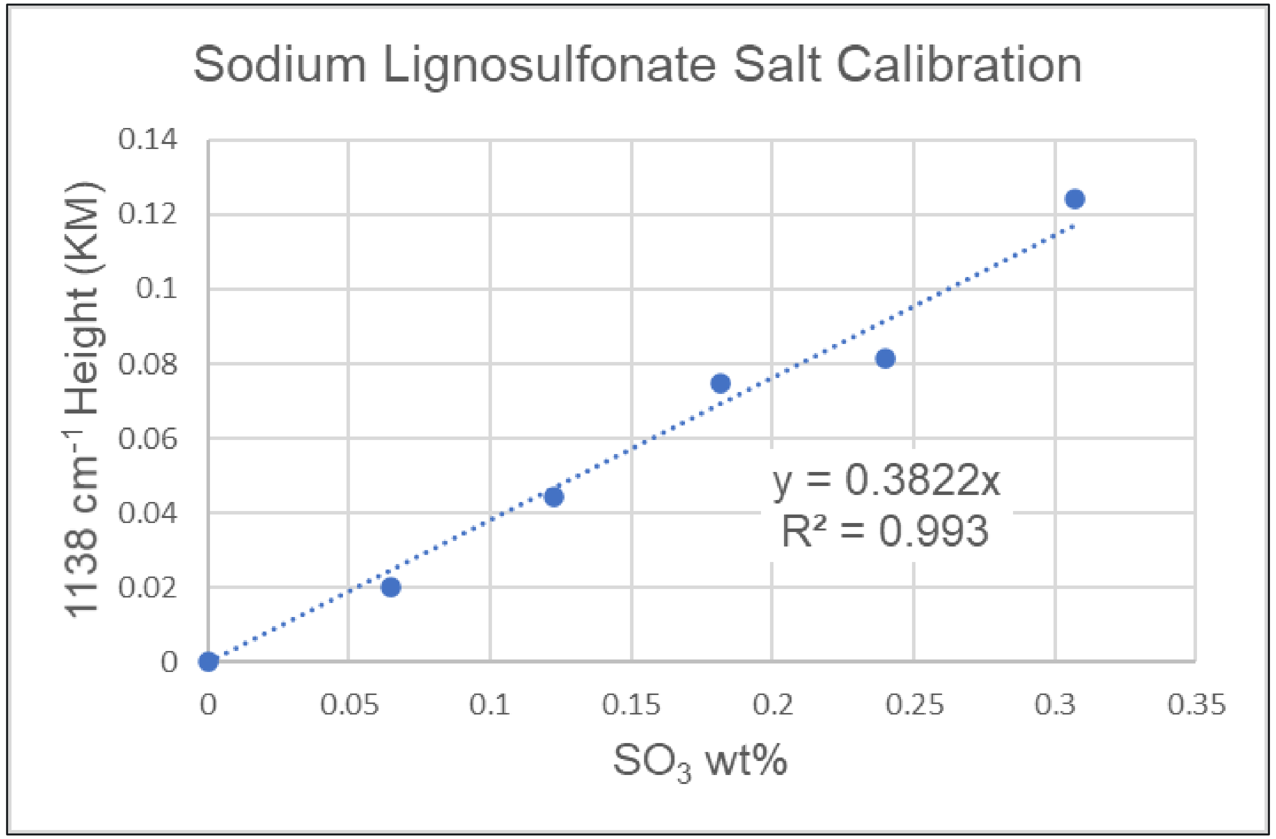


Entry	Reactant	Base	Solvent	Temp.	Pressure	Time	Yield
1	Lignite*	NaOH*	neat	130 ºC	250 psig	12 h	59%
2						6 h	39%
3						3 h	65%
4						9 h	57%

Quantification of Carboxylation on Lignin by FT-IR

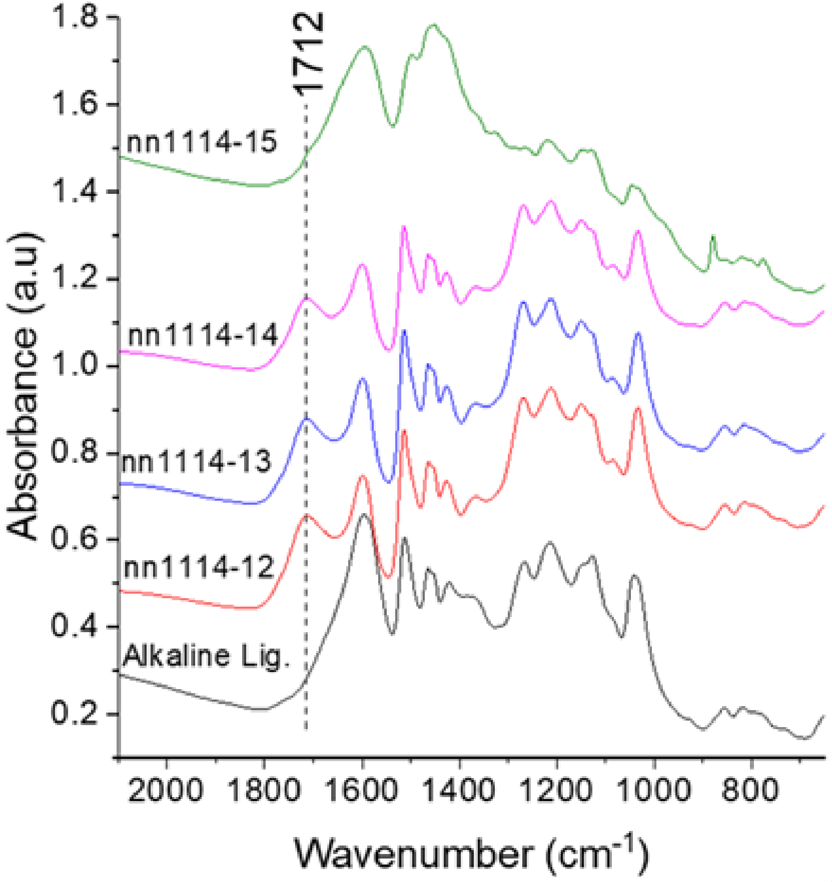
FT-IR can be used for the quantification of carboxylic acids on Lignin

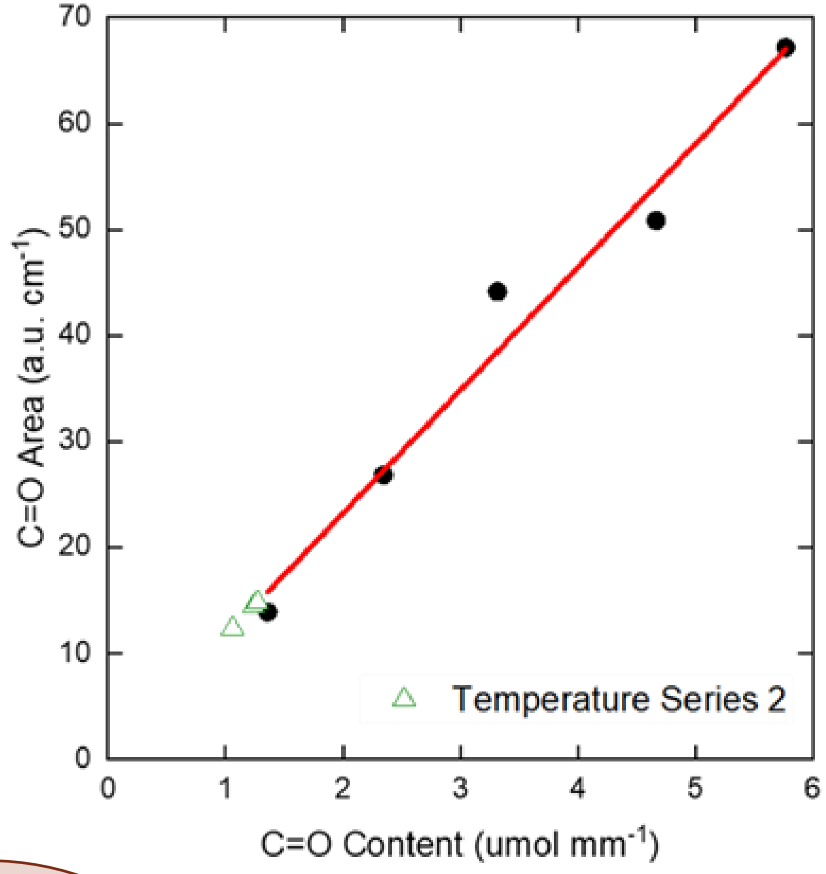




- ➢IR results conformed that acid washing is needed for quantifying carboxylic acids on lignins
- ➢IR results on sodium lignin sulfonate demonstrated an excellent fit between peak height of SO_3 groups and the SO_3 weight percentage.
- ➢IR results of sodium lignin sulfonate indicated that IR can be used to successfully quantify -SO_3 groups.

Quantification of COOH Functional Group in Lignin – Alkaline Lignin*





Sample ID	Temperature (°C)	Pressure (PSI)	C=O g/ Lignin g (wt%) (Calculated)	C=O g/ Lignin g (wt%) (1.72% Error)	C=O g/Lignin g (wt%) (18.9% Error)
nn1114-12	80	220	5.206208598	5.117703	4.378421
nn1114-13	105	216	5.102555356	5.015812	4.291249
nn1114-14	130	235	4.327287624	4.253724	3.639249

*Patent Pending

Composite Synthesis and ASTM Properties

Composite design

Polymer type
Filler type
Filler size
Filler content

Additives and content

Manufacturing process design

Thermophysical properties of constituents

Manufacturing approach

Tooling design

Process parameters

Composite performance

Filler distribution

Tensile and flexural properties
ASTM D6109
ASTM D7031

Water absorption

IBC metrics

Process development for baseline composites

Process development for functionalized lignin

Performance of composites with functionalized lignin

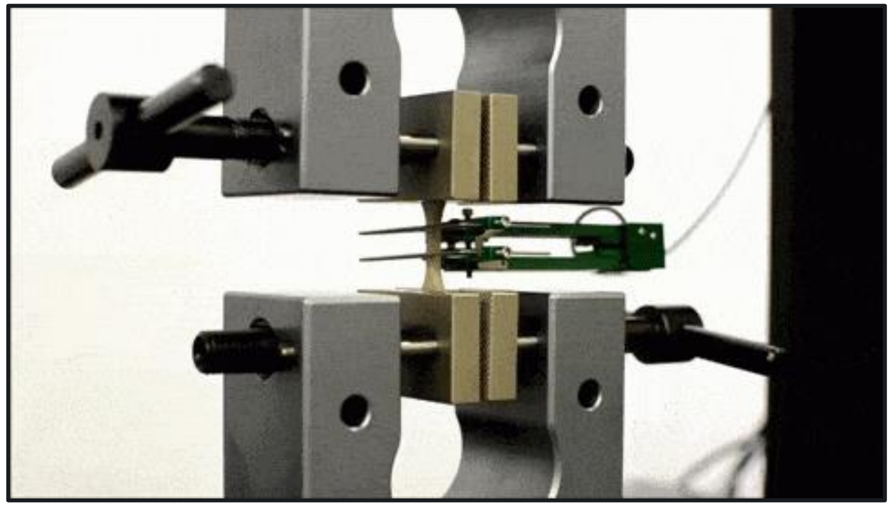
CO_2LIG Composite Manufacturing

Pacific Northwest
NATIONAL LABORATORY

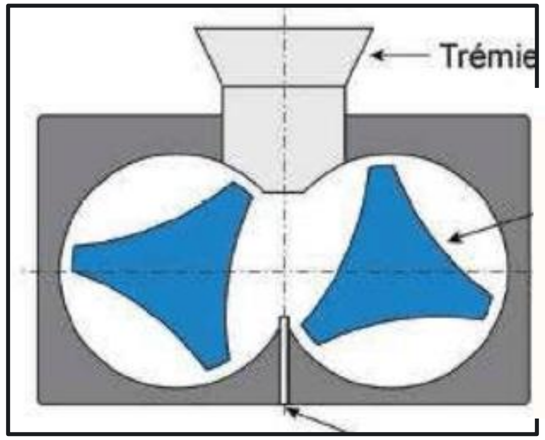
Manufacturing baseline composites to test properties.

1. Dry mixing
- Filler: wood flour, lignin, lignite
- Polymer: HDPE, MAPE

3. Granulation



2. Melt compounding



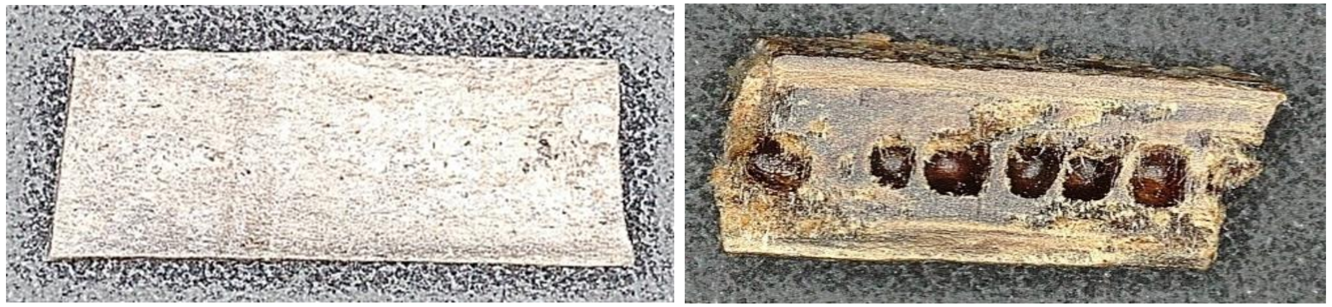
4. Injection molding

Composites
- Panel
- Tensile specimens
- Flexural specimens

Process Development for CO_2LIG-polymer Composites

Pacific Northwest
NATIONAL LABORATORY

Identified process conditions for CO_2LIG-polymer composites with less voids.



50% WPC

60% LPC

Optical photos of flexural specimens, manufactured using one-stage molding parameters

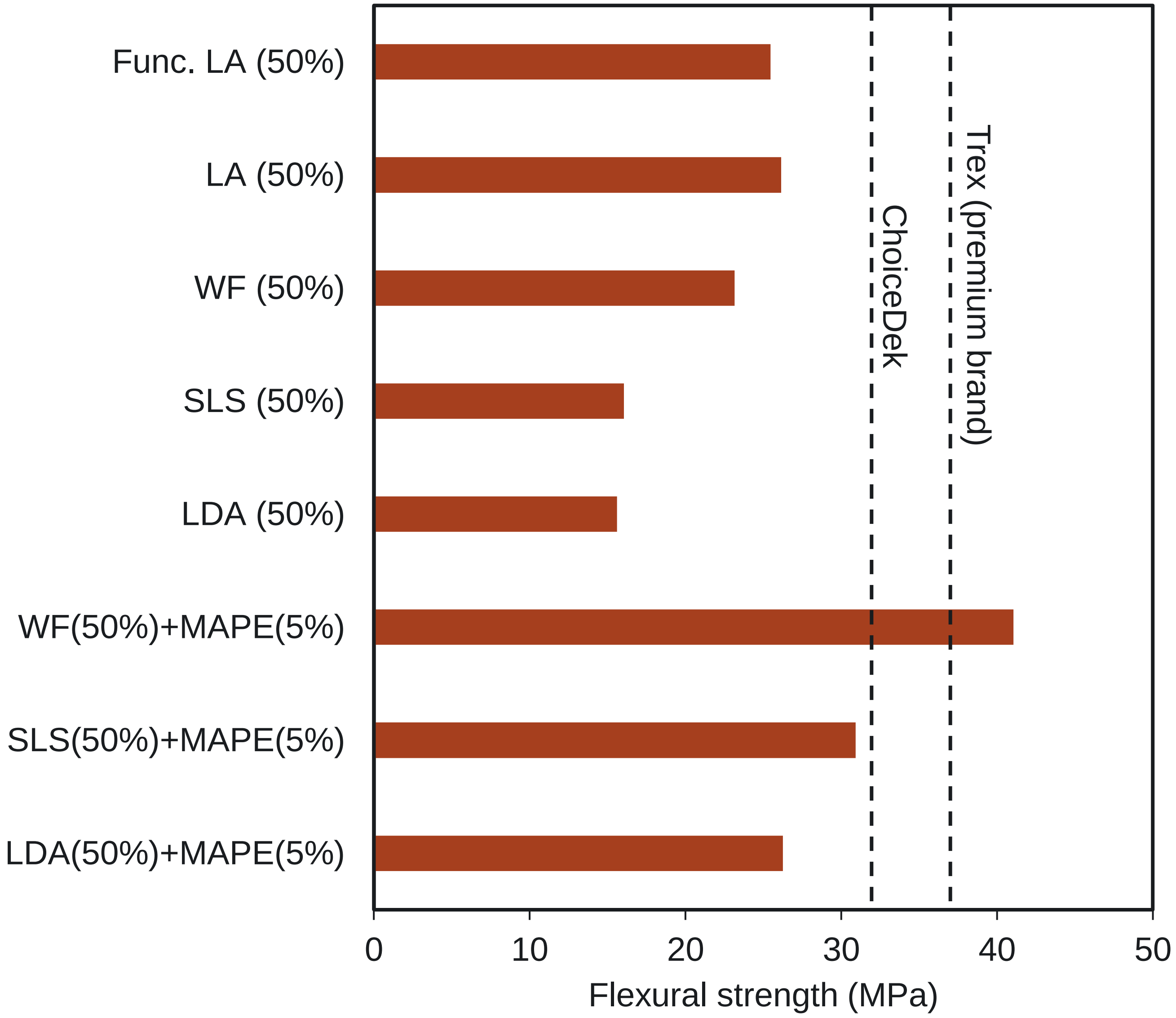
60% LPC

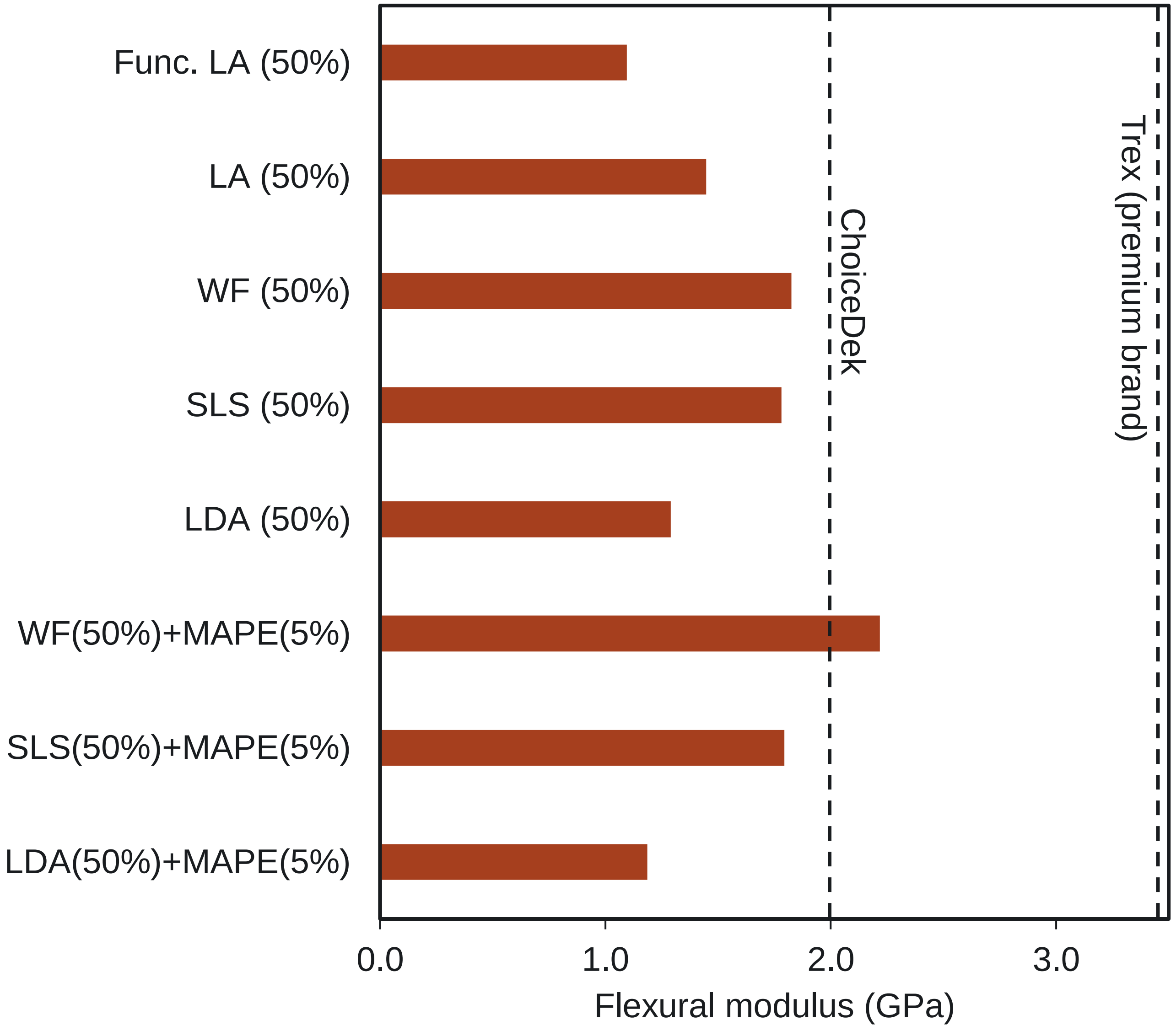
Cross-section of LPC's manufactured using two-stage molding parameters

	One-Stage, WPC	**One-Stage, LPC**	**Two-Stage, LPC**	**Two-Stage, Low-Temp, LPC**
Composite Temperature (°F)	365	365	365	320
Mold Temperature (°F)	200	200	200	200
Injection Rate (in/s)	1.25	0.85	1.1; 0.85	1.1; 0.85
Pack Fill Length (in)	0.3	0.15	0.15	0.15
Pack Hold Time (s)	5	5	5	5
Pack Pressure (psi)	20,000	20,000	20,000	20,000
Refill Screw Jog Speed (rpm)	120	120	120	120

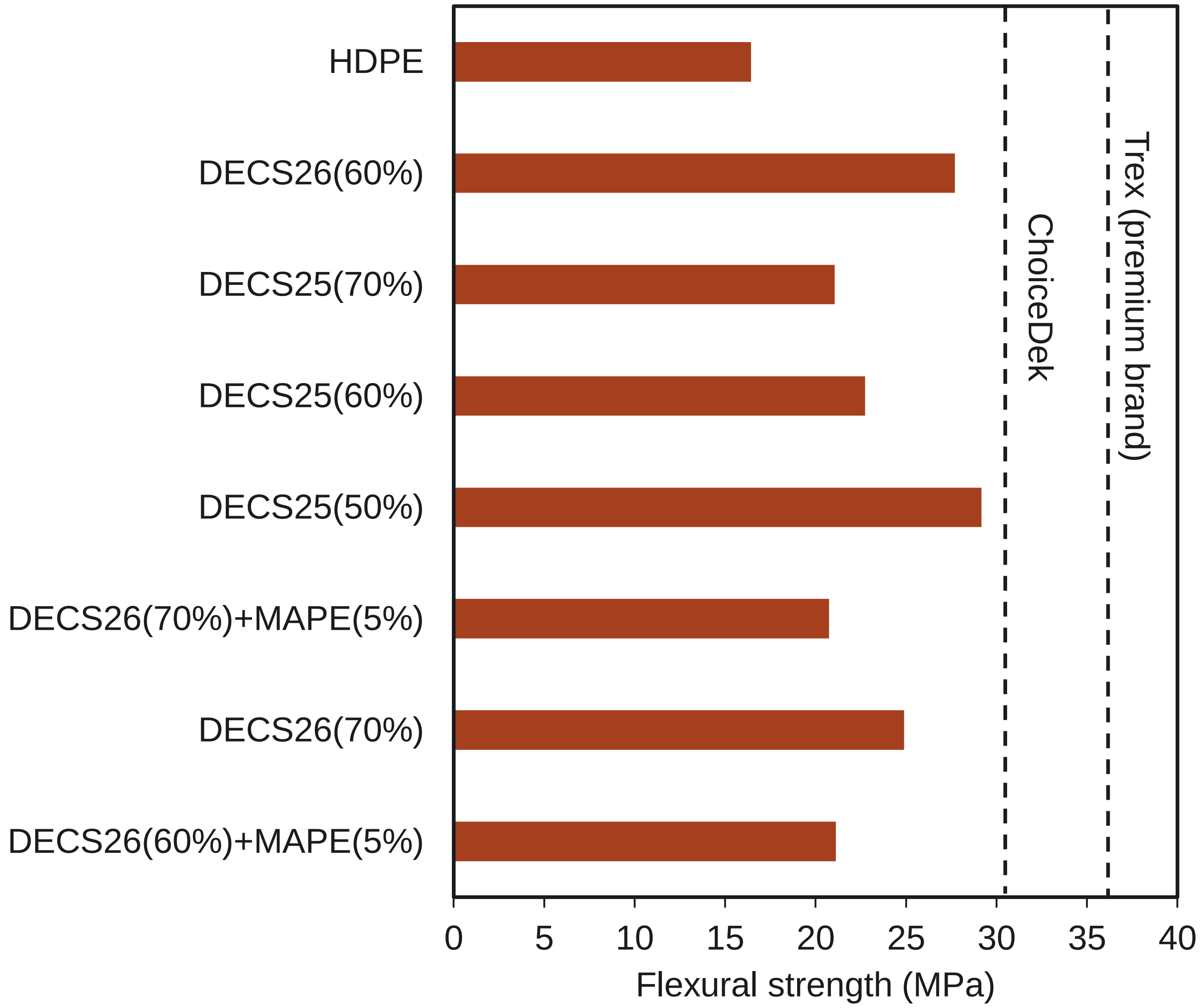
- Two-Stage injection process greatly reduced the occurrence of the voids
- The void distribution pattern was significantly different from that observed for all previous samples

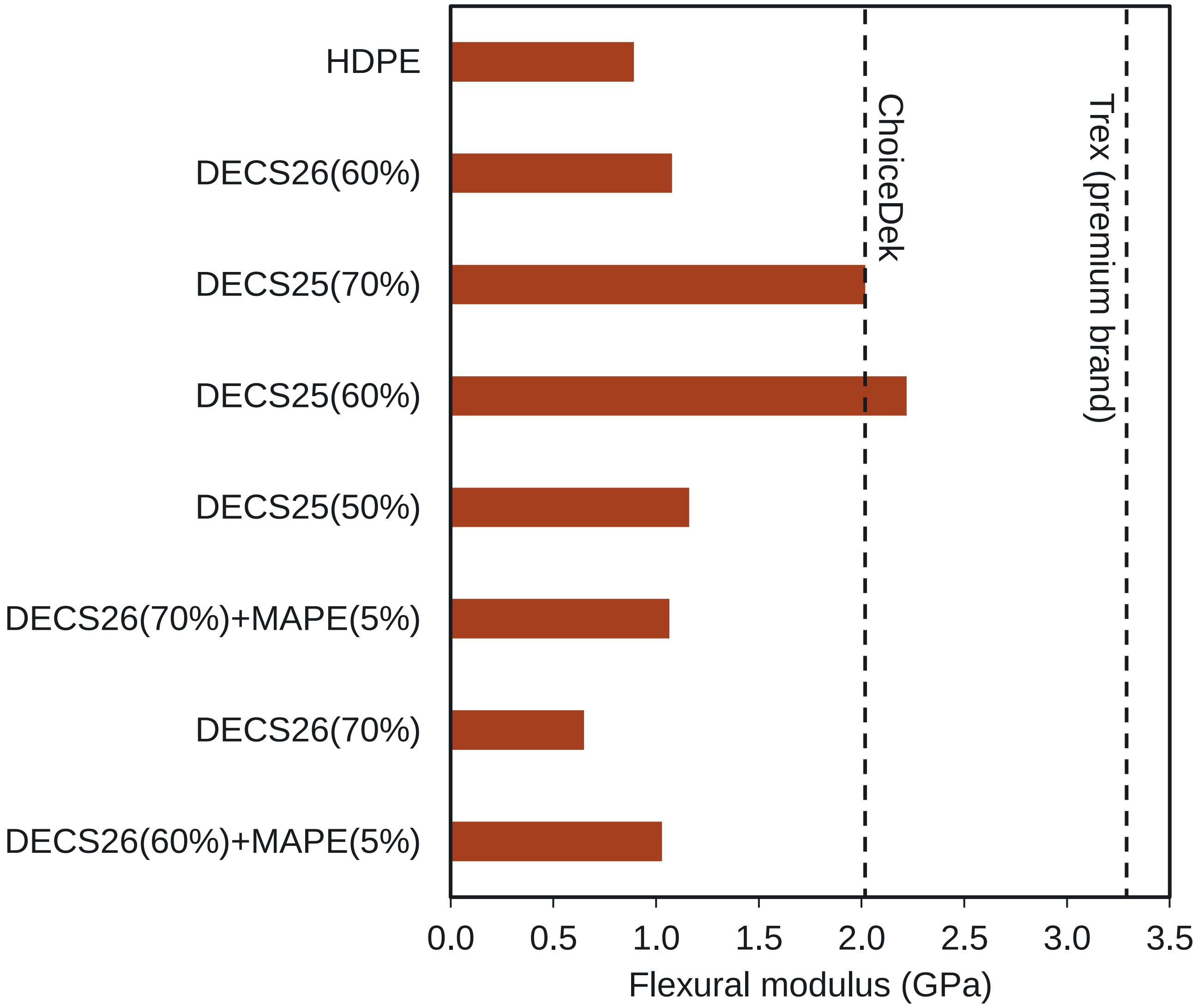
Mechanical Performance of Lignin Plastic Composites





Mechanical Performance of Lignite Plastic Composites





Load at Maximum Deflection (IBC Requirement)

*Meets Milestone 6.1

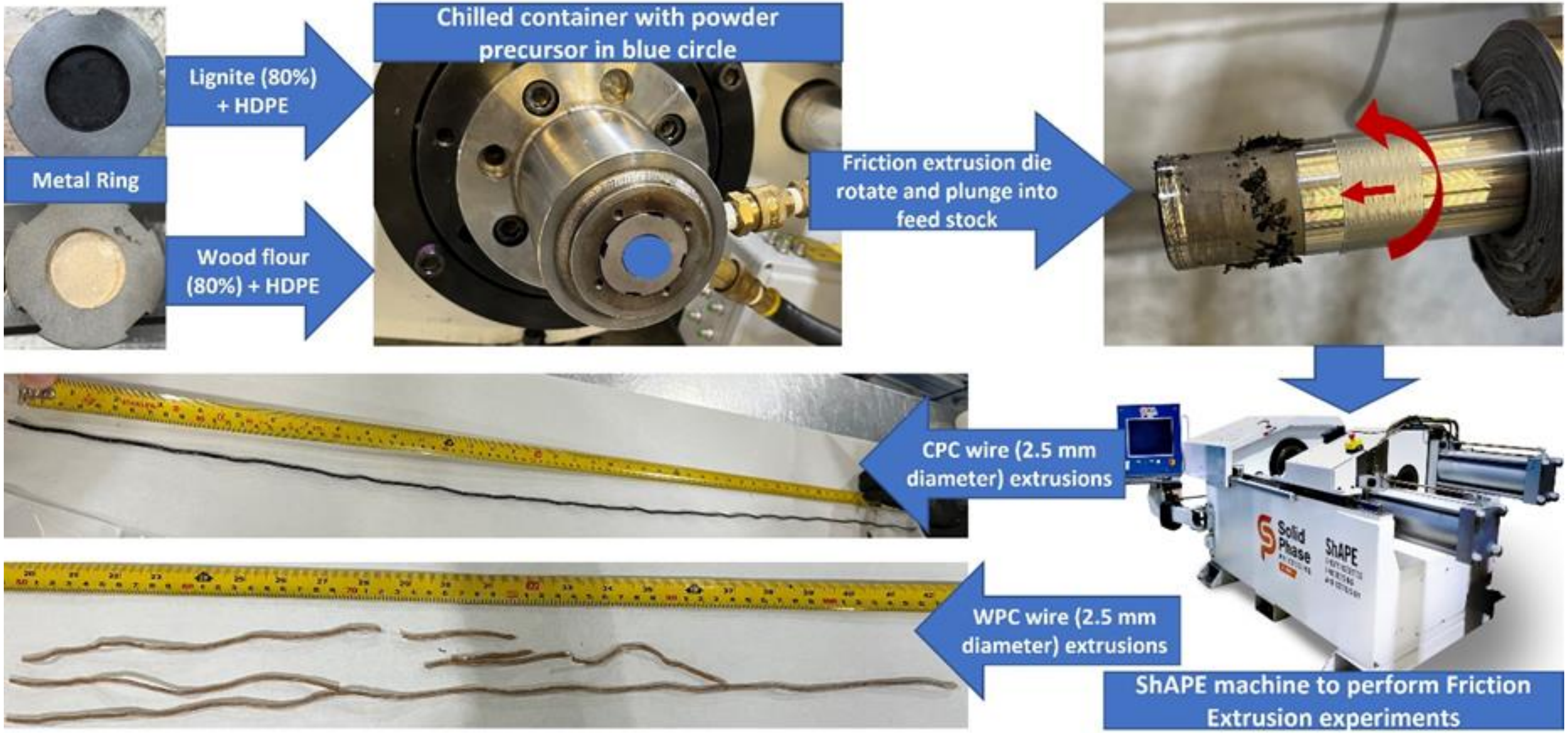
Carboxylated lignin polymer composites met IBC standard for flooring and decking material (>100 psf)

IBC
INTERNATIONAL
BUILDING
CODE

ASTM D7264 was used for Mechanical Performance

Material	Flexural strength (MPa)	Flexural modulus (GPa)	Uniform load at max deflection allowed (psf)	Notes
LPC with 50 wt.% LA (unfunctionalized)	**26.21**	**1.04**	**131.2**	Need to reduce the span rating/increase decking board thickness to meet the requirement
LPC with 50 wt.% fLA	**25.86**	**1.87**	**92.6 – 105.2**	Meets the requirement at 16” span rating and 1” thickness

Friction extrusion of CO_2LIG Composites



Metal Ring

Lignite (80%) + HDPE

Wood flour (80%) + HDPE

Chilled container with powder precursor in blue circle

Friction extrusion die rotate and plunge into feed stock

ShAPE machine to perform Friction Extrusion experiments

CPC wire (2.5 mm diameter) extrusions

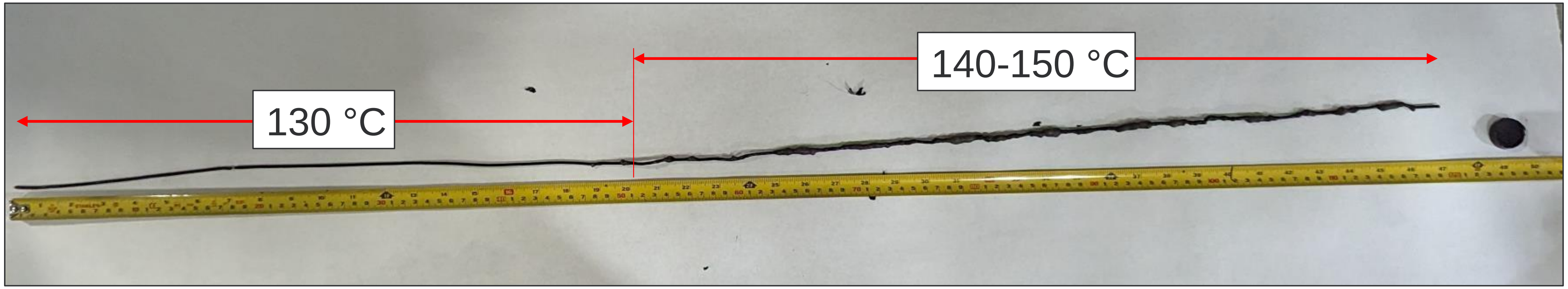
WPC wire (2.5 mm diameter) extrusions

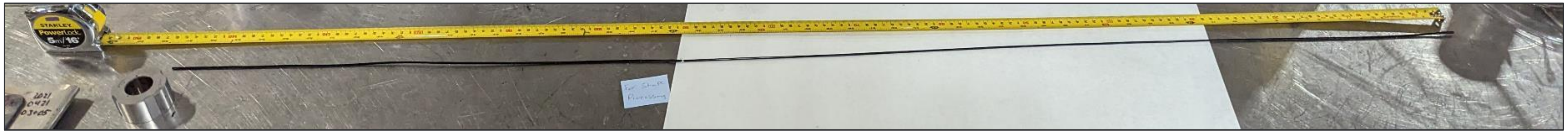
- A 1.2 m long (at 135 ^{o}C) and 2.5 mm diameter CPC wire was extruded that was continuous in nature

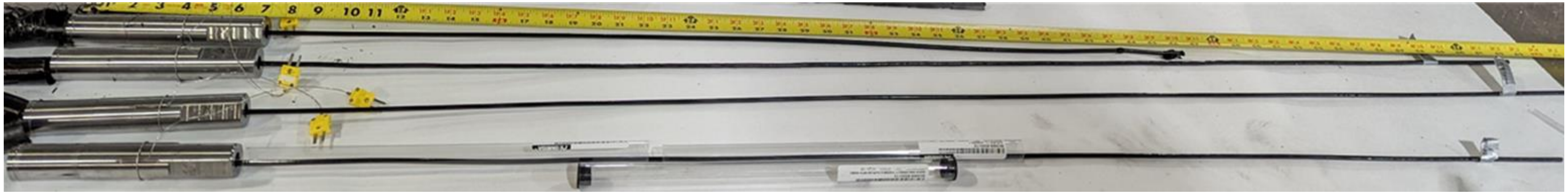
Friction extrusion of CO$_2$LIG Composites

ShAPE :Friction extrusion of CO_2LIG Composites Effect of Extrusion Temperature

Identified process conditions for extruding CPC’s.

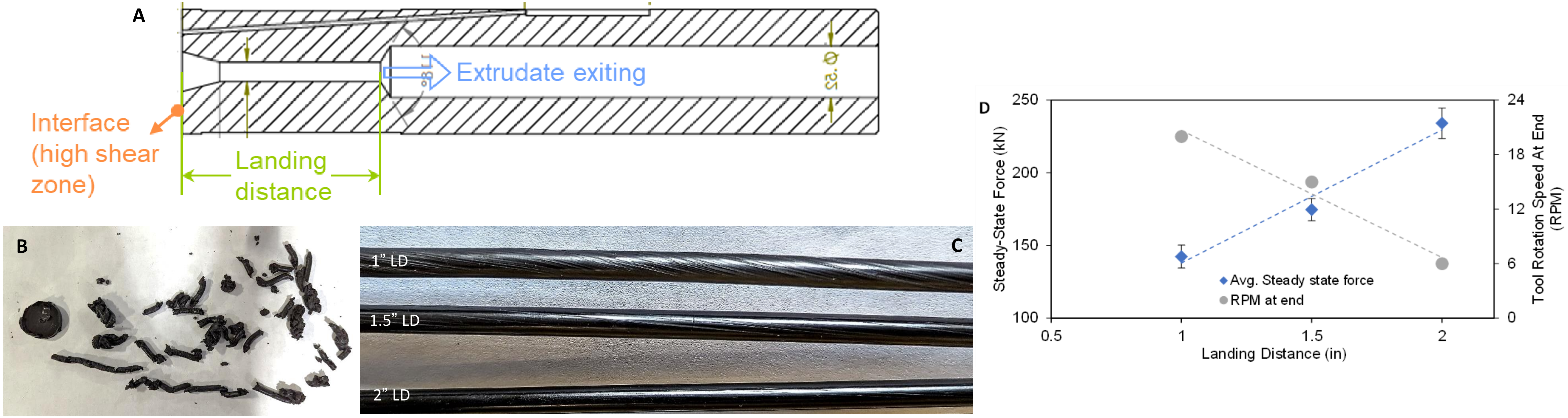






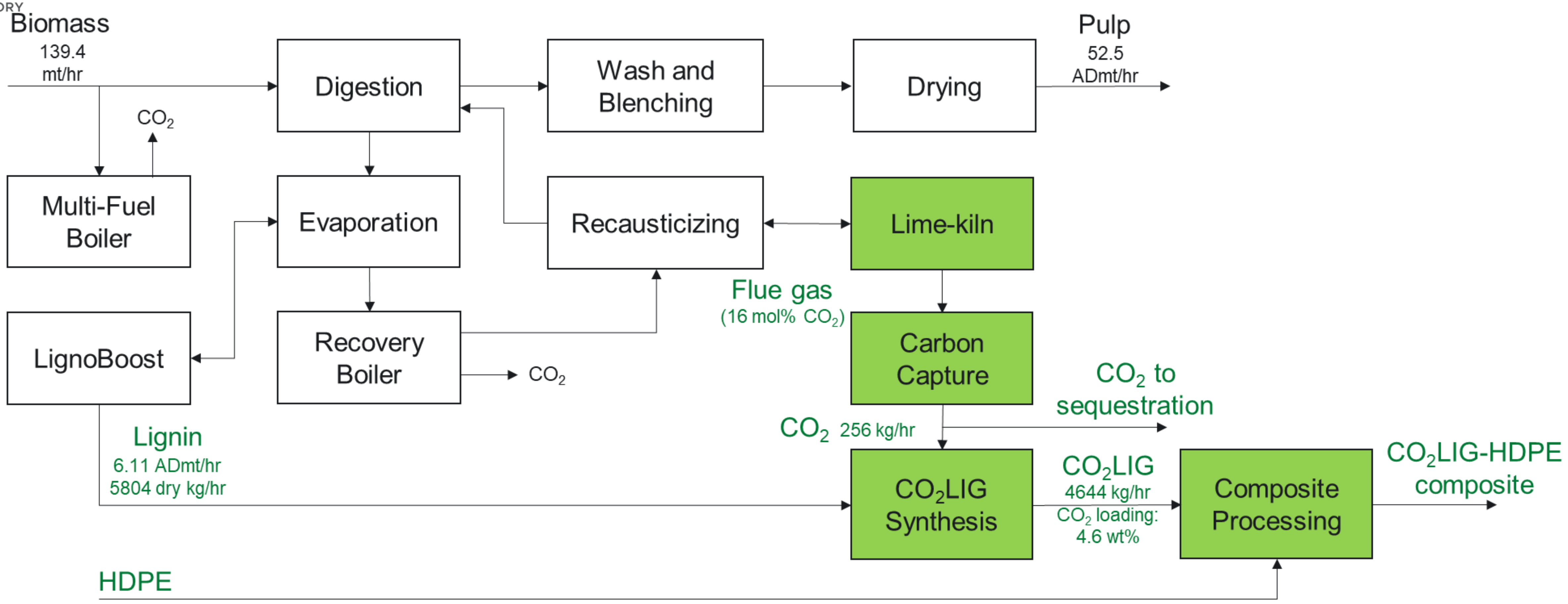
- At 130 °C extrusion temperature, the wire was smooth with uniform diameter throughout the extruded length

ShAPE: Effect of Landing Distance



- ➢ Effect of Tool landing distances ranging form 0.125’-2” on extrudability and surface finish of the Lignite-Plastic composites (CPC’s) with 80 % lignite was investigated.
- ➢ Surface was smoother with a longer landing distance (2”).

Techno-Economic Projections



Biomass
139.4 mt/hr

Digestion

Wash and Blenching

Drying

Pulp
52.5 ADmt/hr

CO_2

Multi-Fuel Boiler

Evaporation

Recausticizing

Lime-kiln

LignoBoost

Recovery Boiler

CO_2

Flue gas
(16 mol% CO_2)

Carbon Capture

CO_2 to sequestration

CO_2 256 kg/hr

Lignin
6.11 ADmt/hr
5804 dry kg/hr

CO_2LIG Synthesis

CO_2LIG
4644 kg/hr
CO_2 loading: 4.6 wt%

Composite Processing

CO_2LIG-HDPE composite

HDPE

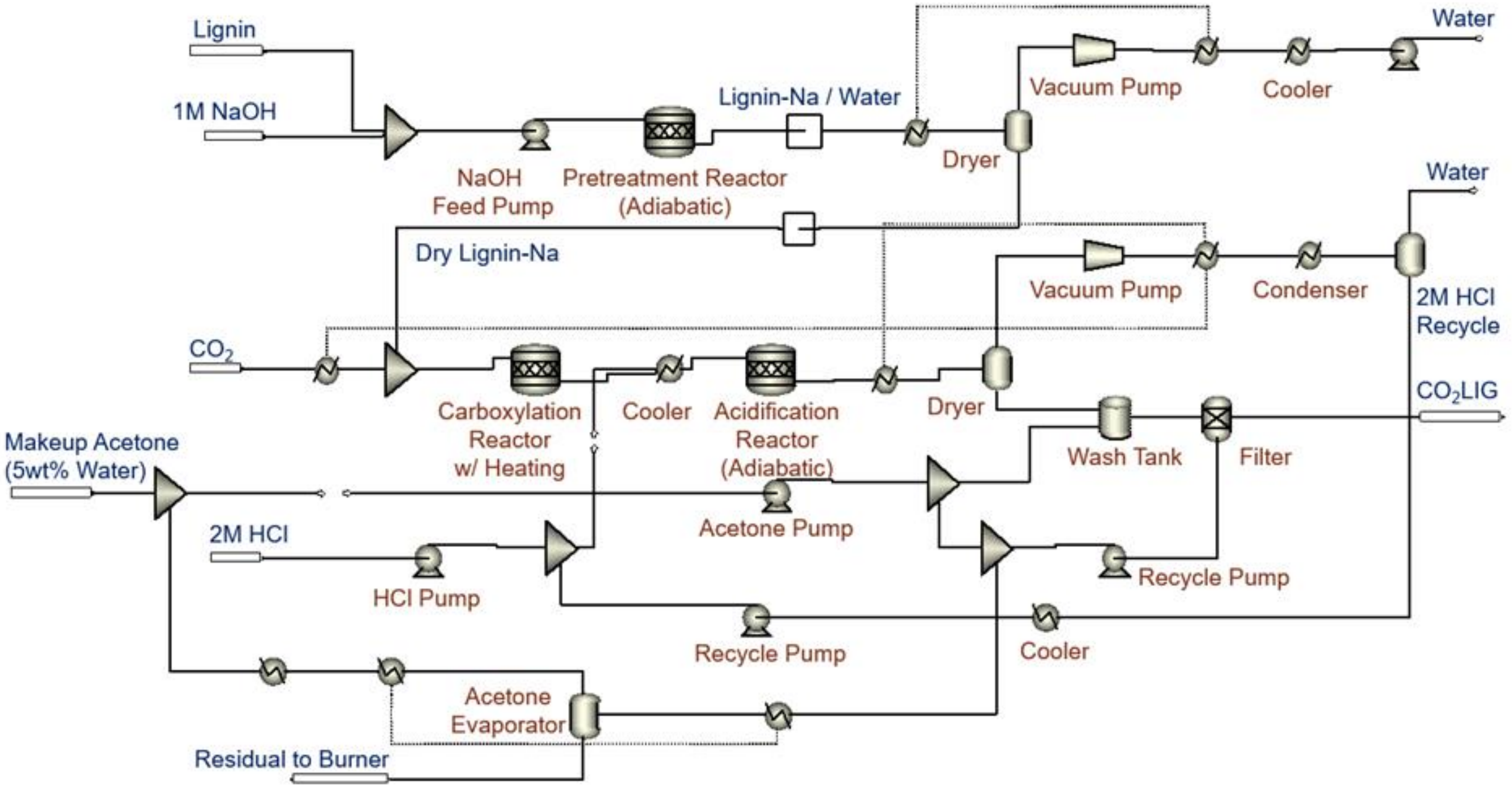
☐ Included in LCA, but not TEA; LCI collected from Culbertson et al., 2016; Lignin price collected from Hodasova et al., 2015

🟩 Included in both TEA and LCA; mass and energy balance, LCI, equipment sizing generated from process models

Model and experimental data-based TEA and cradle-to-gate LCA to quantify economic and environmental benefits

Techno-Economic Projections

Updated TEA results based on real experimental data suggests that *CO2LIG can be produced at a cost lower than the HDPE ($1/kg)*



Minimum CO_2LIG selling price ($/kg)	
Lignin	0.375
Chemicals	0.085
Utilities	0.028
By-product credits	-0.043
Capital depreciation	0.071
O&M	0.088
Return on Investment	0.106
Others (tax, etc.)	0.112
Total	0.820

Experimental	Temperature (°C)	Pressure (bar)	Residence time	Solution (/g lignin)
Pretreatment	room	1.2	18 h	1ml 1M NaOH
Carboxylation	130	16	3 h	
Acidification	room	1	5 min	1ml 2M HCl
Dryers	130	vacuum		
Acetone wash	room	room		200 ml acetone (5% H_2O)

Plans for Next Budget Period: Carboxylation

- Identify process for separation of carboxylated lignin
- Production of up to 5 kg quantities of carboxylated lignin and lignite
- By the end of BP3, the project team will design an integrated process for isolation of the pure carboxylated lignin product

BP2 Preliminary Life Cycle Analysis

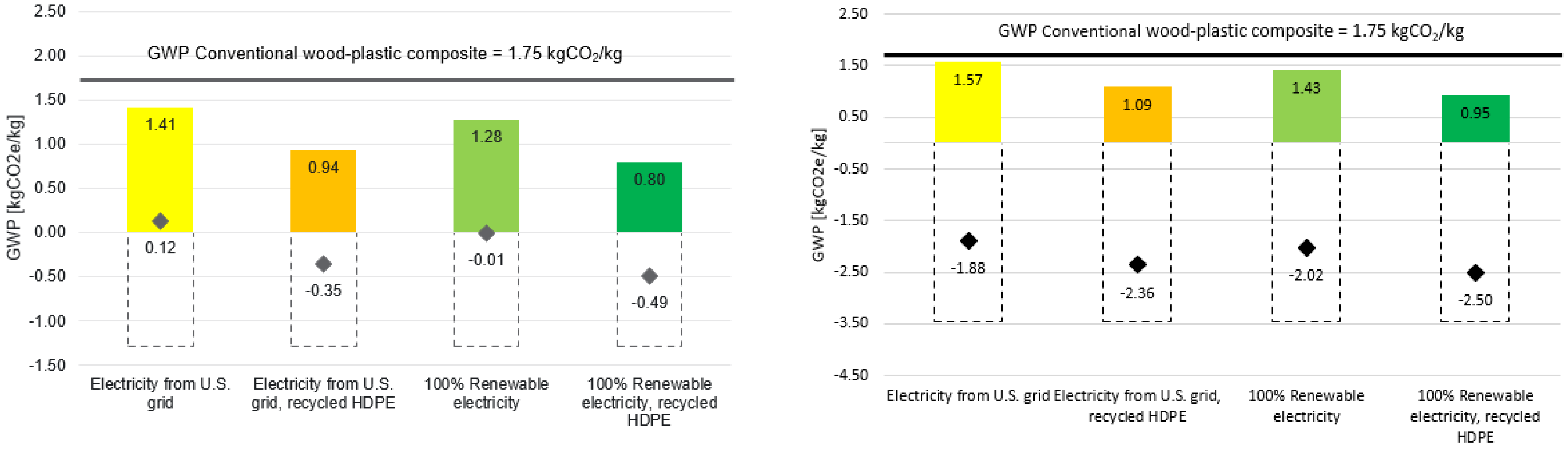
Pacific Northwest
NATIONAL LABORATORY

LCA suggesting the global warming potential (GWP) of CO_2LIG Panel is much lower than that of conventional wood-plastic composite

➢ **Approach**

- "Cradle-to-gate" LCA was conducted in SimaPro v9 using TRICI v.2.1 method with DATASMART and Ecoinvent v.3.5 databases. The functional unit is 1 kg of CO_2LIG-plastic composite panel.
- Main components of GWP include (1) **fossil-based GHG emissions** and (2) **carbon storage benefits** from storing carbon over the **lifetime of the panel** and CO_2 **sequestrated underground**.

➢ **Global warming potential of 1 kg CO_2LIG panel** (time horizon for fossil-based GHG emission and carbon storage benefits fixed to 100 year)



(a) Results include the CO_2 stored into CO_2LIG only

(b) Results include the CO_2 stored into CO_2LIG and the additional CO_2 sequestered and stored in the ground

➢ **Replacing virgin HDPE with recycled HDPE, and use of renewable electricity can further reduce GWP**

*Meets Milestone 7.1

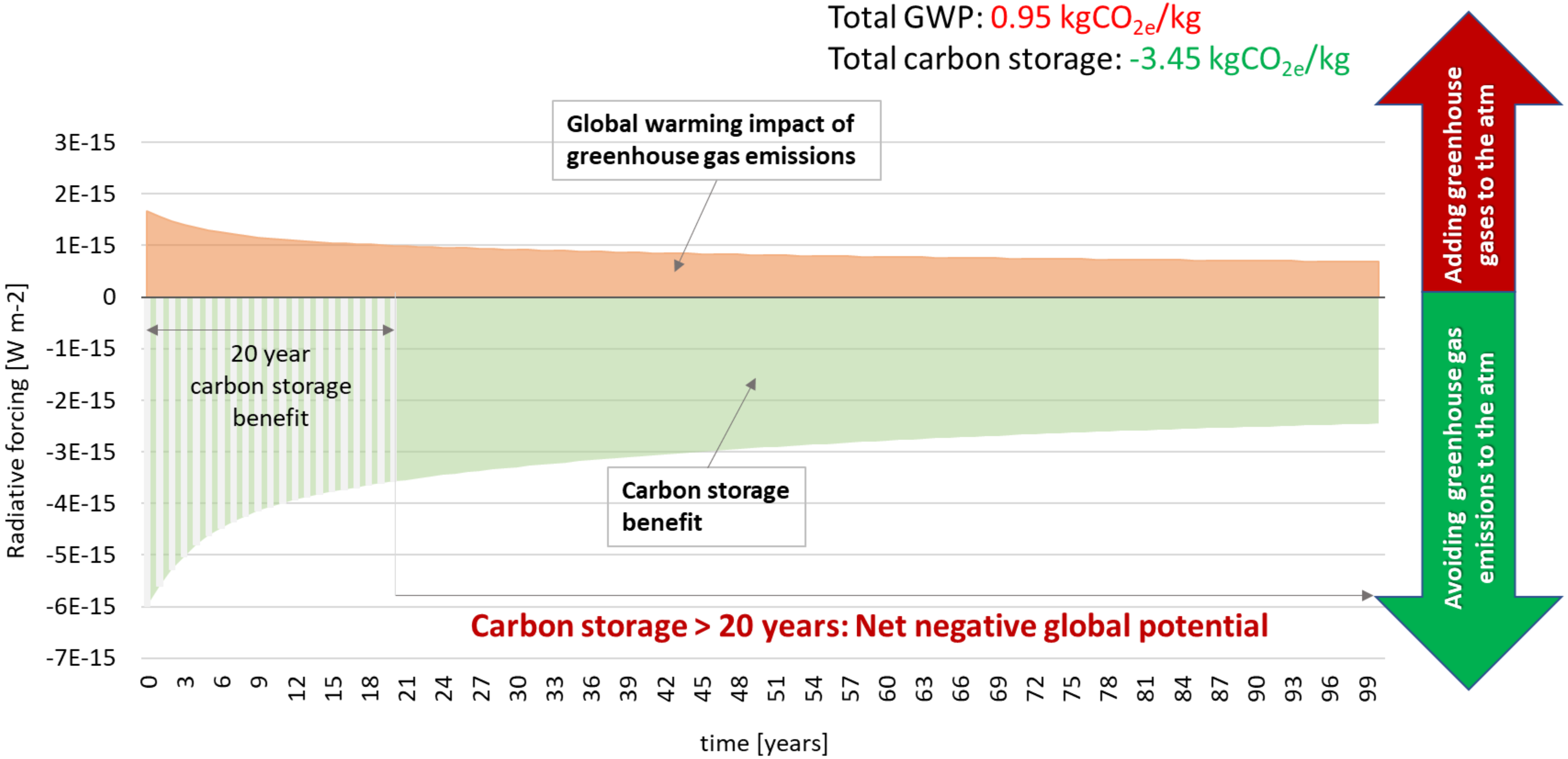
Pacific Northwest
NATIONAL LABORATORY

BP2 Preliminary Life Cycle Analysis

LCA was done for lignin carboxylation, suggesting the GWP of CO_2LIG Panel is can be carbon negative when the penal has a life time greater than 20 years

- ➢ Temporal radiative forcing analysis was performed to evaluate the number of years of storge of CO_2 on CO_2LIG-plastic composite (lifetime of the product) needed to achieve carbon neutrality.

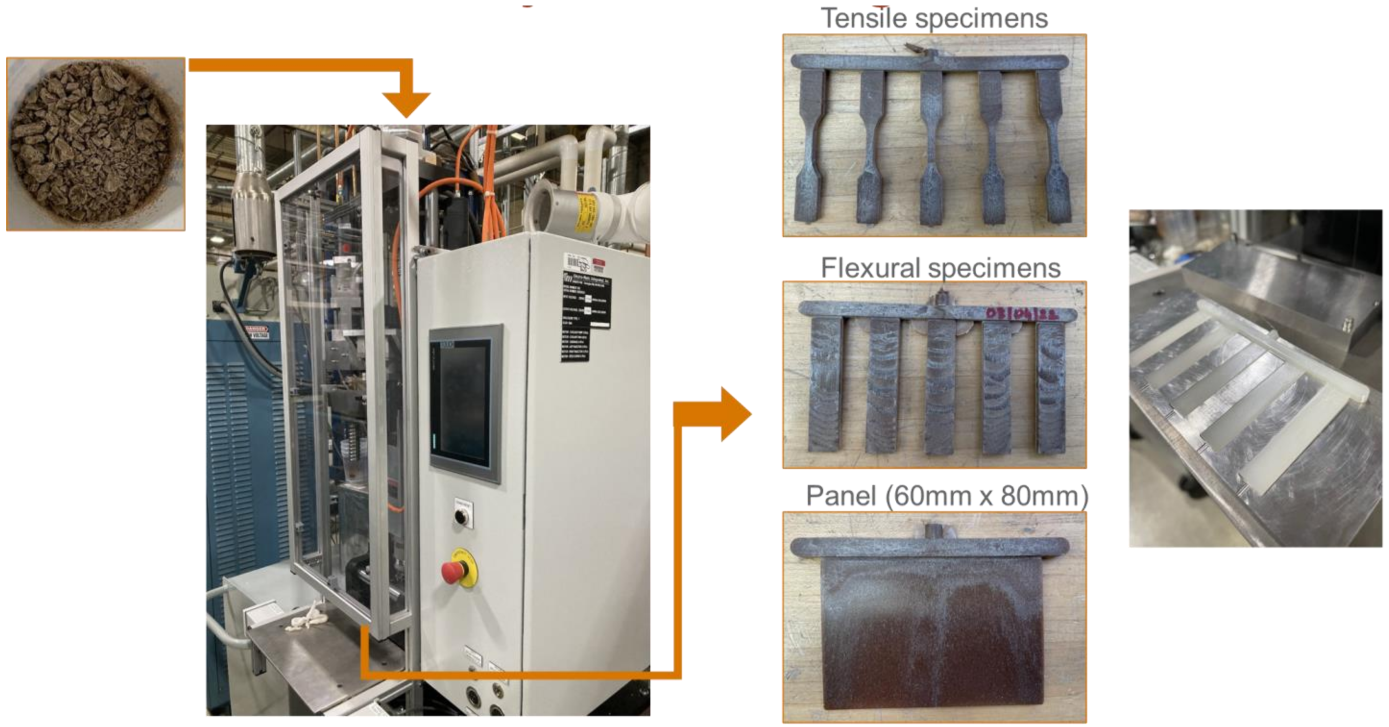
Total GWP: 0.95 $kgCO_{2e}/kg$
Total carbon storage: -3.45 $kgCO_{2e}/kg$



Using 100% renewable electricity, recycled HDPE with benefits from subsurface CO_2 storage

*Meets Milestone 7.1

Polymer Composites Future Testing and TEA



- Complete assessing composites strength, stability, and flammability.
- Down select composite that meets international building code (IBC) requirements for decking applications.
- Complete analysis to determine if the proposed process is CO_2-negative and
- Complete market analysis to access feasibility and impact.

Pacific Northwest
NATIONAL LABORATORY

Milestone Log

	No.	Budget Period	Task/ Subtask	Milestone Description	Planned Completion	Actual or Estimated Completion
	M1.1	1 & 2	1	Statement of Project Objectives	9/01/2021	8/24/2021
	M1.1.1	1 & 2	1	Updated Project Management Plan	10/1/2021	10/12/2021
Completed ✓	**M1.2**	1	1	Kickoff Meeting	10/21/2021	10/21/2021
	M1.3	1, 2 & 3	1	Quarterly progress reports	30 days after end of each reporting period	
Completed ✓	**M1.4**	1 & 2	1	Go-No-Go Presentation at NETL	9/30/2022, 9/30/2023	09/12/2022
	M1.5	3	1	Delivery of final report - Final technical and economic feasibility study with recommendations of continuation for slip stream testing and industry hand off	Report 30 days after end of project completion, presentation scheduled as convenient for DOE	10/31/2023
Completed ✓	**M2.1**	1	2	Synthesize 1-3 CO$_2$BOL solvents that can achieve 1-5 wt.% carboxylation of a model lignin or lignite molecule	2/28/2022	02/28/2022
Completed ✓	**M2.2**	1	2	Quantify the optimal density of carboxylic acids on lignin and lignite for composite manufacturing	5/31/2022	03/31/2023
Completed ✓	**M3.1**	1	3	Demonstrate 3-5 lignin/ lignite-polymer composites can be manufactured using injection and compression molding at varying temperatures (150-200 ºC).	9/30/2022	9/30/2022
Completed ✓	**M4.1**	1	4	Complete preliminary LCA/TEA using 1-5 Wt. % carboxylated to study the feasibility of producing carbon negative materials	9/30/2022	9/30/2022
Completed ✓	**M5.1**	1	2	Identify at least 1 viable CO$_2$BOL solvent that can achieve 1-5 wt.% carboxylation of lignin or lignite. (We have identified inexpensive inorganic bases such as sodium hydroxide (NaOH) and organic superbases such as 1,8-Diazobicylo[5,4,0]undec-7-ene (DBU) for achieving 1-5 wt.% carboxylation)	12/31/2022	12/31/2022
Completed ✓	**M5.2**	2	5	Produce 100 g of carboxylated lignin and lignite particles for fabrication into composites based on optimal filler concentration and structural thermal properties.	6/30/2023	6/30/2023
On Track ✓	**M6.1**	2	6	Produce decking composites meeting international building codes criteria such as distributed load > 100 lbs.ft^2.	9/30/2023	On track
On Track ✓	**M7.1**	2	7	Complete Preliminary LCA/TEA completed based on assumptions for at least one lignin/lignite candidate to study the feasibility of producing carbon-negative materials.	9/30/2023	On track

BP	Success Criteria	BP2 Results	Milestone
BP2	Down select 1 viable CO_2BOL solvent that can achieve 1-5 wt.% carboxylation of lignin or lignite at expected reboiler temperatures at rates commensurate with slipstream sizing and modeling.	Successfully identified an inexpensive base for achieving lignin carboxylation. Lignin carboxylation can be achieved at temperatures as low as 80 ^oC within 3 h.	**Met Milestone 5.1**
BP2	Produce 100 g of carboxylated lignin and lignite particles for fabrication into composites based on optimal filler concentration and structural thermal properties. Produce decking composites meeting international building codes criteria such as distributed load > 100 lbs.ft^2.	We successfully carboxylated alkaline lignin at 200 g scale at temperatures 130 ^oC within 3 h with an isolated product yield of 64 %. We successfully produced LPCs containing carboxylated alkaline lignin (CAL) that meet the IBC standards for both flooring and decking materials. These LPCs can withstand a uniform live load of 105 pounds per square foot (psf), which exceeds the minimum requirement of 100 psf necessary for qualifying as a flooring or decking material as per the IBC guidelines.	**Met Milestone 5.2** **Met Milestone 6.1**
BP2	Complete preliminary LCA/TEA completed based on assumptions for at least one lignin/lignite candidate to study the feasibility of producing carbon-negative materials	Initial TEA results suggests that the proposed technology can produce alternative CO_2 negative building material at a cost lower than the price of conventional building material (i.e., HDPE). Carbon neutrality can be attained after 20 years of carbon storage through the usage of 100% renewable electricity, recycled HDPE, sequestering CO_2 in CO_2LIG, and storing the CO_2 underground. Beyond this time frame, continuing to store carbon would yield a net decrease in Global Warming Potential (GWP), advancing carbon negativity.	**Met Milestone 7.1**

Project Outputs

Keynote Talk

1. **Satish K. Nune**, Conversion of CO_2 into High Value Materials: Producing CO_2-Negative Building Composites, June 4-5, 2023, Baker Hughes's Energy Frontier Summit, Energy Innovation Center, Oklahoma City, USA.

Presentations

2. **David J. Heldebrant**, CO2-Negative Building Composites; CO_2 Mineralization Workshop at University of Minnesota, May 2, 2023, USA.
3. **Jaelynne King,** Spectroscopic techniques for Carboxylic Acid Quantification, 2023 Spring ACS meeting at Indianapolis, USA.

Abstracts Submitted to Conferences

1. **Satish K. Nune,** Conversion of CO_2 into Materials: Producing CO_2-Negative Building Composites; **47th International Technical Conference on Clean Energy** (July 23-27, 2023) (session organized by DOE PM Aaron Fuller).
2. Satish K. Nune, Integrated Capture and Conversion of CO_2 into Materials (IC^3M): Pathways for Producing CO_2-Negative Building Composites, 2023 FECM / NETL Carbon Management Research Project Review Meeting, Pittsburgh, PA.
3. **Satish K. Nune,** Production of CO_2-Negative Building Composites; **2023 AICHE Annual Meeting, Orlando, FL.**
4. **Yuan Jiang** Process Modeling, Techno-economic and Life-cycle GHG Emission Assessments of Producing Carbon-Negative Building Material from CO_2 and Waste Lignin or Lignite; **2023 AICHE Annual Meeting, Orlando, FL.**

Project Outputs

Submitted U.S. Patent/ Provisional Patent Applications

1. INTEGRATED CAPTURE AND CONVERSION OF CO_2 INTO MATERIALS: METHODS AND PROCESSES FOR PRODUCING CO_2-NEGATIVE BUILDING COMPOSITES; **8/2022**
2. CONVERSION OF CO_2 INTO MATERIALS: PRODUCING CO_2-NEGATIVE BUILDING COMPOSITES; **3/2023**

Invention Disclosures

1. Friction extrusion of polymer composites with high filler content (**5/10/2023**)

Publications

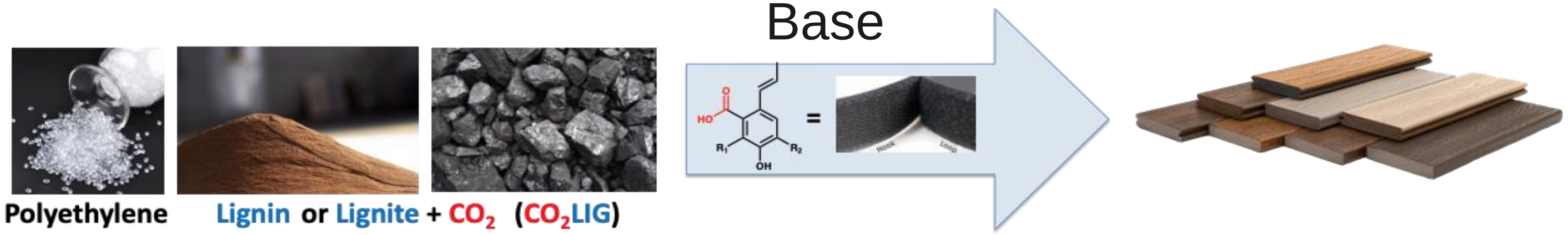
1. Quantification of carboxylic acid groups in Kolbe-Schmidt type reaction of lignin
   Manuscript in preparation **7/2023**.

2. Manufacturing and characterization of lignin-polymer composites (LPC)
   Manuscript in preparation **7/2023**.

3. Process parameter optimization, manufacturing, and performance evaluation of lignin polymer composites (LPC) with high lignin filler concentrations
   Manuscript in Preparation **9/2023.**

Key Findings of FWP 78606 in BP2

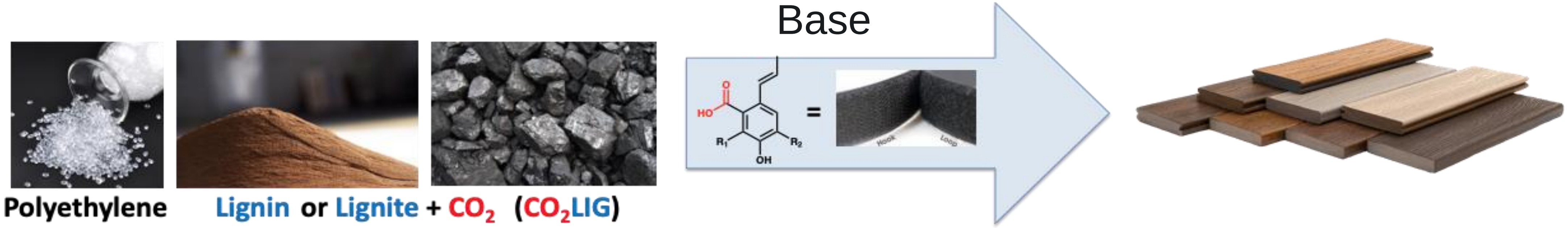
CO_2 can provide the “Velcro” to help lignin and lignite bind strongly to polymers.



- Demonstrated lignin carboxylation (~2-5 wt. % CO_2 fixation) at temperatures as low as 80 °C in 100g batches
  - **Meets Milestone 5.1 and BP2 success criteria #1**
- Developed separation methods to separate pure carboxylated lignin (up to 94 % Yield)
- Manufactured lignin plastic composites (LPC; 50-70 wt.% lignin filler) with improved mechanical performance over wood plastic composites (WPCs)
- Manufactured lignite plastic composites (LtPC, 80 wt.% lignite filler) using shear assisted processing and extrusion (ShAPE^{TM})
  - **IP filed , manuscript #1 in preparation**
- Manufactured LPCs with carboxylated alkaline lignin to meet IBC metrics to withstand a uniform live load of up to 105.2 psf (>100 psf minimum requirement) for qualifying as a flooring or decking material.
  - **Meets Milestone 5.2 and BP2 success criteria #2**
- 100 % renewable electricity, recycled HDPE and sequestered CO_2 in CO_2LIG, and the CO_2 stored in the ground, the carbon neutrality is achieved after 20 years of carbon storage, and > 20 years results in a net negative GWP.
  - **Meets Milestone 7.1 and BP2 success criteria #3, manuscript #2 in preparation**

Summary

CO_2 can provide the “Velcro” to help lignin and lignite bind strongly to polymers.



- Successfully demonstrated lignin carboxylation (~2-5 wt. % CO_2 fixation) at temperatures as low as 80 ^oC (~94 % Yield).
- Produced lignin composites (LPC; 70 % lignin filler) with elastic modulus and tensile strength higher than Wood Plastic Composite (WPC’s)
- Carboxylated lignin polymer composites met IBC standard for flooring and decking material (>100 psf); carboxylic acids (captured CO_2) serves as a chemical binder that upgrades lignin or lignite particle polymer composites
- Produced uniform lignite-HDPE composite (CPC) wires (up to 135 cm in length) using PNNL proprietary method (Shape^{TM}).
- When 100 % renewable electricity, recycled HDPE was considered along with sequestered CO_2 in CO_2LIG, and the CO_2 stored in the ground, the carbon neutrality is achieved after 20 years of carbon storage. Storing carbon for more than 20 years would result in a net negative GWP.

Pacific
Northwest
NATIONAL LABORATORY

U.S. DEPARTMENT OF
ENERGY
Office of Fossil Energy

SoCalGas

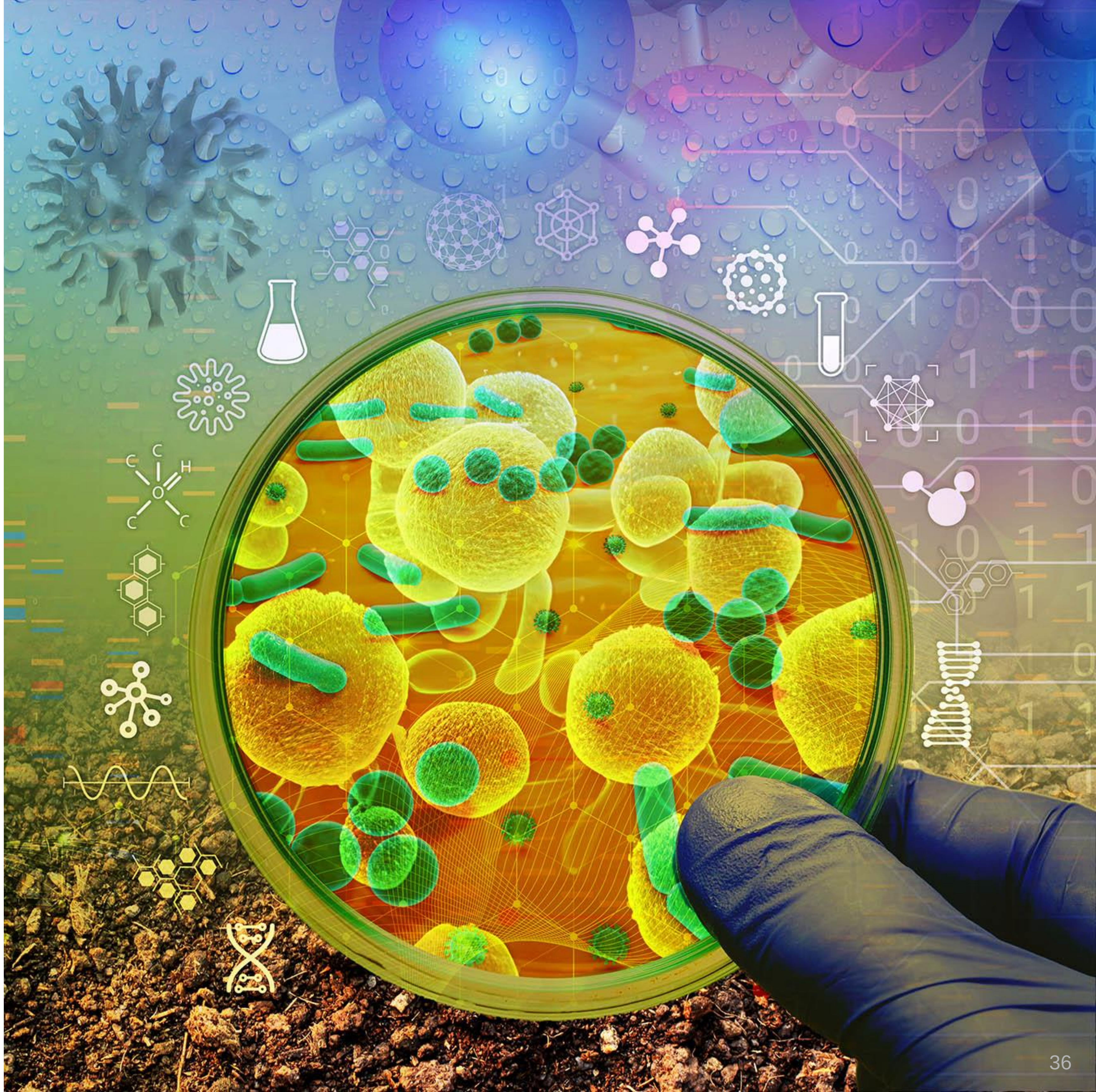
Thank you

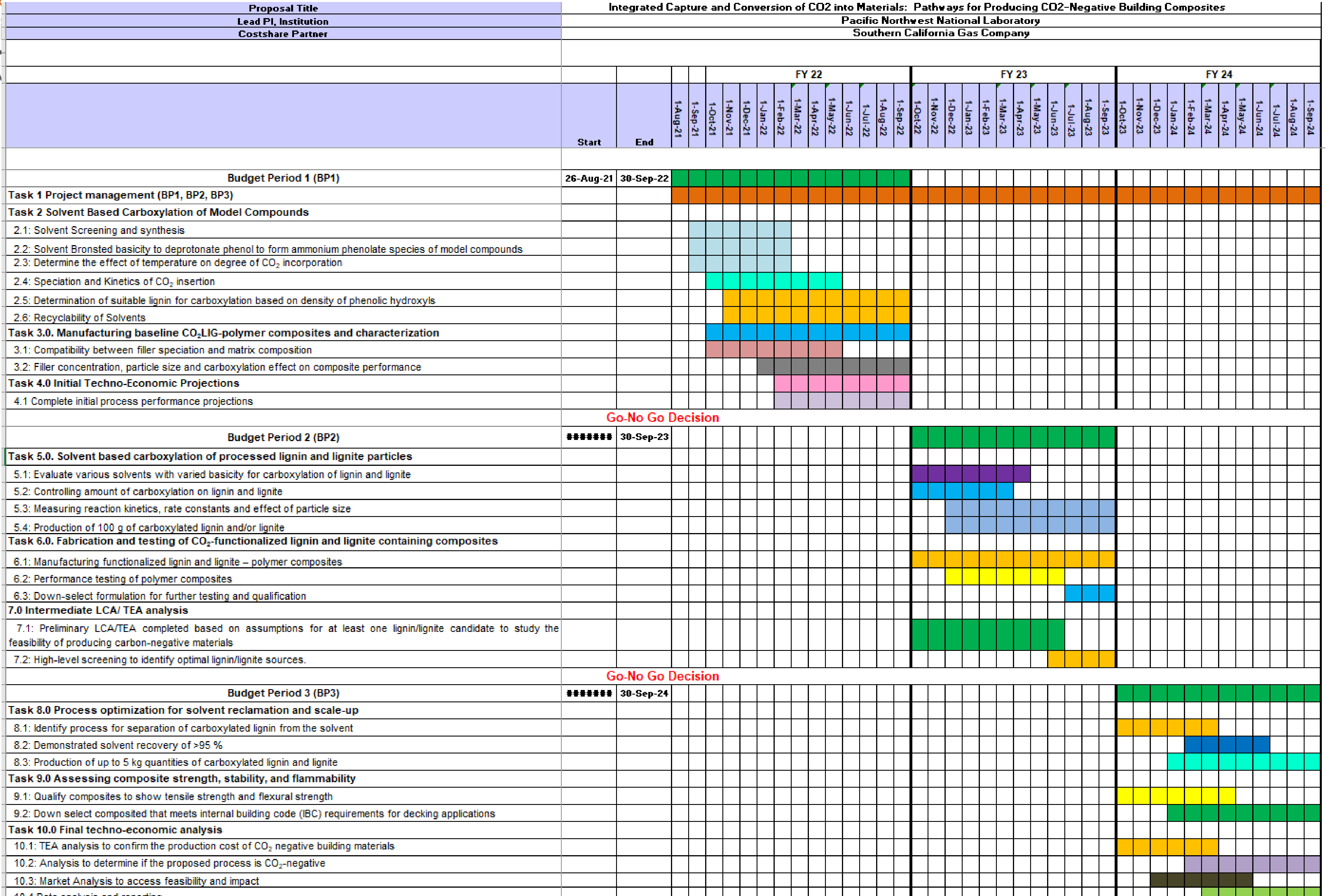
Satish K. Nune
SENIOR RESEARCH SCIENTIST

Phone: 509-371-6632
Satish.nune@pnnl.gov

902 Battelle Boulevard
P.O. Box 999
Richland, WA 99352

www.pnnl.gov





Proposal Title: Integrated Capture and Conversion of CO2 into Materials: Pathways for Producing CO2-Negative Building Composites
Lead PI, Institution: Pacific Northwest National Laboratory
Costshare Partner: Southern California Gas Company

					FY 22												FY 23												FY 24											
	Start	End	1-Aug-21	1-Sep-21	1-Oct-21	1-Nov-21	1-Dec-21	1-Jan-22	1-Feb-22	1-Mar-22	1-Apr-22	1-May-22	1-Jun-22	1-Jul-22	1-Aug-22	1-Sep-22	1-Oct-22	1-Nov-22	1-Dec-22	1-Jan-23	1-Feb-23	1-Mar-23	1-Apr-23	1-May-23	1-Jun-23	1-Jul-23	1-Aug-23	1-Sep-23	1-Oct-23	1-Nov-23	1-Dec-23	1-Jan-24	1-Feb-24	1-Mar-24	1-Apr-24	1-May-24	1-Jun-24	1-Jul-24	1-Aug-24	1-Sep-24
Budget Period 1 (BP1)	26-Aug-21	30-Sep-22	■	■	■	■	■	■	■	■	■	■	■	■	■	■																								
Task 1 Project management (BP1, BP2, BP3)			■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■
Task 2 Solvent Based Carboxylation of Model Compounds																																								
2.1: Solvent Screening and synthesis				■	■	■	■	■																																
2.2: Solvent Bronsted basicity to deprotonate phenol to form ammonium phenolate species of model compounds				■	■	■	■	■																																
2.3: Determine the effect of temperature on degree of CO_2 incorporation				■	■	■	■	■																																
2.4: Speciation and Kinetics of CO_2 insertion					■	■	■	■	■	■	■	■																												
2.5: Determination of suitable lignin for carboxylation based on density of phenolic hydroxyls						■	■	■	■	■	■	■	■	■	■	■																								
2.6: Recyclability of Solvents						■	■	■	■	■	■	■	■	■	■	■																								
Task 3.0. Manufacturing baseline CO_2LIG-polymer composites and characterization					■	■	■	■	■	■	■	■	■	■	■	■																								
3.1: Compatibility between filler speciation and matrix composition					■	■	■	■	■	■	■	■																												
3.2: Filler concentration, particle size and carboxylation effect on composite performance								■	■	■	■	■	■	■	■	■																								
Task 4.0 Initial Techno-Economic Projections									■	■	■	■	■	■	■	■																								
4.1 Complete initial process performance projections									■	■	■	■	■	■	■	■																								
	Go-No Go Decision																																							
Budget Period 2 (BP2)	#######	30-Sep-23															■	■	■	■	■	■	■	■	■	■	■	■												
Task 5.0. Solvent based carboxylation of processed lignin and lignite particles																																								
5.1: Evaluate various solvents with varied basicity for carboxylation of lignin and lignite																	■	■	■	■	■	■	■																	
5.2: Controlling amount of carboxylation on lignin and lignite																	■	■	■	■	■	■																		
5.3: Measuring reaction kinetics, rate constants and effect of particle size																			■	■	■	■	■	■	■	■	■	■												
5.4: Production of 100 g of carboxylated lignin and/or lignite																			■	■	■	■	■	■	■	■	■	■												
Task 6.0. Fabrication and testing of CO_2-functionalized lignin and lignite containing composites																																								
6.1: Manufacturing functionalized lignin and lignite – polymer composites																	■	■	■	■	■	■	■	■	■	■	■	■												
6.2: Performance testing of polymer composites																			■	■	■	■	■	■	■															
6.3: Down-select formulation for further testing and qualification																										■	■	■												
7.0 Intermediate LCA/ TEA analysis																																								
7.1: Preliminary LCA/TEA completed based on assumptions for at least one lignin/lignite candidate to study the feasibility of producing carbon-negative materials																	■	■	■	■	■	■	■	■	■															
7.2: High-level screening to identify optimal lignin/lignite sources.																									■	■	■	■												
	Go-No Go Decision																																							
Budget Period 3 (BP3)	#######	30-Sep-24																											■	■	■	■	■	■	■	■	■	■	■	■
Task 8.0 Process optimization for solvent reclamation and scale-up																																								
8.1: Identify process for separation of carboxylated lignin from the solvent																													■	■	■	■	■	■						
8.2: Demonstrated solvent recovery of >95 %																																	■	■	■	■	■			
8.3: Production of up to 5 kg quantities of carboxylated lignin and lignite																																■	■	■	■	■	■	■	■	■
Task 9.0 Assessing composite strength, stability, and flammability																																								
9.1: Qualify composites to show tensile strength and flexural strength																													■	■	■	■	■	■	■					
9.2: Down select composited that meets internal building code (IBC) requirements for decking applications																																■	■	■	■	■	■	■	■	■
Task 10.0 Final techno-economic analysis																																								
10.1: TEA analysis to confirm the production cost of CO_2 negative building materials																													■	■	■	■	■	■						
10.2: Analysis to determine if the proposed process is CO_2-negative																																	■	■	■	■	■	■	■	■
10.3: Market Analysis to access feasibility and impact																															■	■	■	■	■	■				
10.4 Data analysis and reporting																																		■	■	■	■	■	■	■

Patents/ Publications

Submitted U.S. Patent/ Provisional Patent Applications

1. INTEGRATED CAPTURE AND CONVERSION OF CO_2 INTO MATERIALS: METHODS AND PROCESSES FOR PRODUCING CO_2-NEGATIVE BUILDING COMPOSITES; **8/2022**
2. CONVERSION OF CO_2 INTO MATERIALS: PRODUCING CO_2-NEGATIVE BUILDING COMPOSITES; **3/2023**

Invention Disclosures

1. Friction extrusion of polymer composites with high filler content (**5/10/2023**)

Publications

1. Quantification of carboxylic acid groups in Kolbe-Schmidt type reaction of lignin
   Manuscript in preparation **9/2023**.

2. Manufacturing and characterization of lignin-polymer composites (LPC)
   Manuscript in preparation **9/2023**.

3. Process parameter optimization, manufacturing, and performance evaluation of lignin polymer composites (LPC) with high lignin filler concentrations
   Manuscript in Preparation **12/2023.**