DOE Bioenergy Technologies Office (BETO) 2023 Project Peer Review

SPERLU Selective Process for Efficient Removal of Lignin and Upgrading

April 6, 2023

Biochemical Conversion and Lignin Utilization

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Spero Renewables, LLC

Mission &
Value proposition

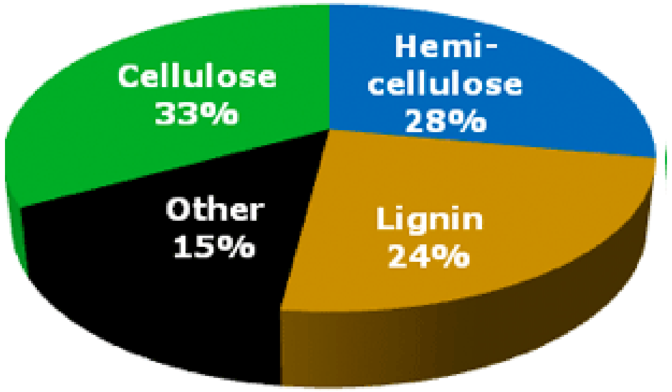
To provide renewable and cost-effective substitutes to petrochemicals – enhancing the quality of life and the environment

Spero Management team

Mahdi	Ian	Eric	Jasmine
CEO	*CTO*	*Eng.*	*BD*

- 20+ year of R&D management experience
- Technology translation & major JDA with multi-nationals
- Forbes 30 under 30
- Board/advisors include co-founder of a major oil exploration company, a former CTO of a major chemical company, and a world expert in polymers.

Moving towards a barrel of biomass will require making chemicals from lignin



$0.4 / kg
Technical lignin

SPERLU™

"Selective Process for Efficient Removal of Lignin and Upgrading"

- Feedstock: wood, non-food agricultural residues, technical lignin

- Replacement for Bisphenol A (BPA) in production of thermoset plastics

- Bio-based chemicals for consumer products

Project Overview

Project Goals:

- Convert 50% of lignin carbon into bio-phenols.
- Make polymers from lignin bio-phenols.
- Bioprocess lignin bio-phenols into chemicals.
- Establish technoeconomic analysis (TEA) and life cycle analysis (LCA) for SPERLU™.

Approach - Objectives

Task 1: Scale SPERLU^{TM} to produce ≥ 200 g of bio-phenols per day

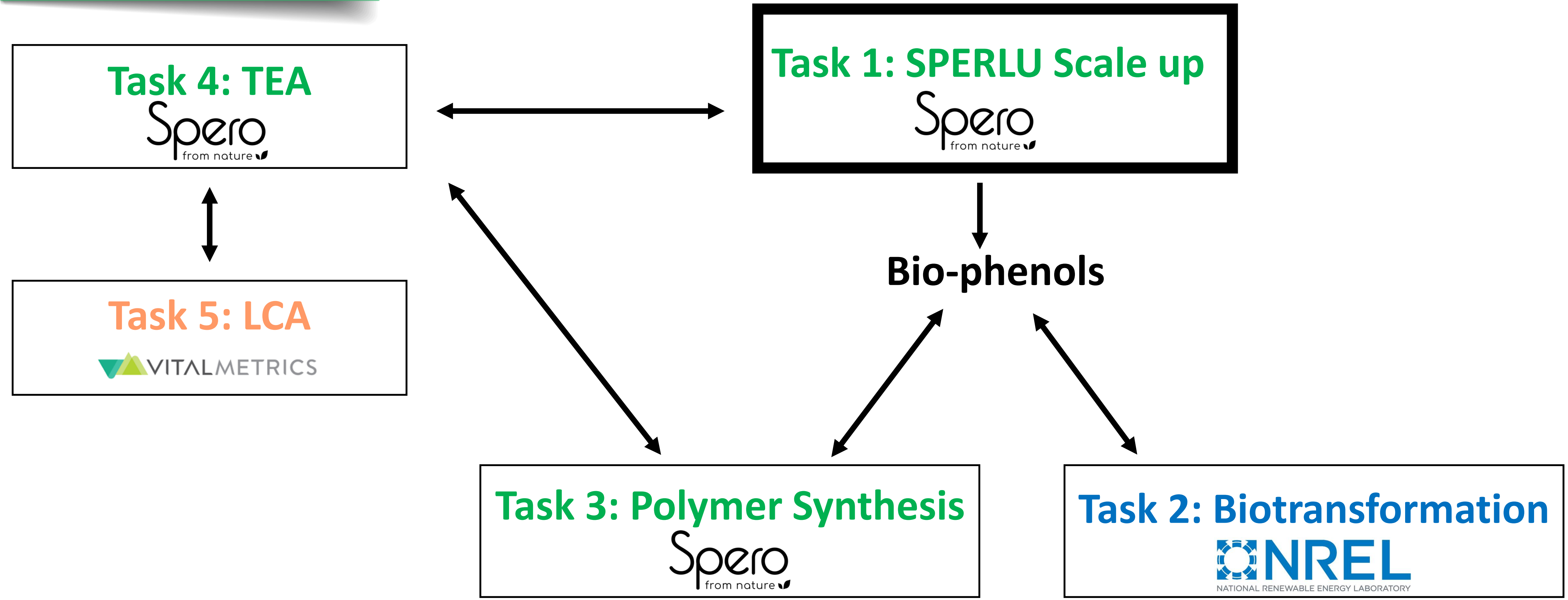
Task 2: Biotransformation optimization

Task 3: Bio-polymer synthesis and determine physical properties

Task 4: TEA of SPERLU^{TM}

Task 5: LCA of SPERLU^{TM}

Approach – Project Structure



Risk Management: (1) Monthly Project Meetings (PIs)

(2) TEA of all process changes

Technical Approach

Task	Technical Approach
1) Scale SPERLU™ to ≥ 200 g bio-phenols	• Extraction kinetics, Catalysis kinetics, Catalyst regen. • Design & Build reactor, produce 200 g bio-phenols (G/NG) • Purify Bio-phenols for Task 2 & 3
2) Biotransformation Optimization	• Strain optimization (delete known aldehyde dehydrogenase) • Test Spero bio-phenols of varying purity • 90% conversion, titer ≥ 10 g/L, productivity ≥ 0.2 g/L/h
3) Biopolymer synthesis & testing	• Polymers from (1) purified bio-phenols (2) bio-phenol mixtures • Test thermal and mechanical properties
4) TEA of SPERLU™	• ASPEN model based on experimental data • Evaluate process alternatives (lignin feed & purifications) • Sensitivity analysis to maximize NPV
5) LCA of SPERLU™	• Life-cycle GHG emissions & energy balance • Sensitivity analysis & process recommendations

Approach – Challenges

Top challenges:

(1) Scale and develop SPERLU™ process while meeting BPA cost targets

- Risk analysis: TEA (ASPEN modeling)
- Risk identified: Solvent volume requirements
- Risk mitigation: Feedstock & purification changes

Technical Lignin
(>95% lignin)

Solvent
5x
Lignin
Mass

Biomass
(25% lignin)

Solvent
20x
Biomass
Mass

(5x lignin
mass)

Technical lignin available commercially at >>20 kta

Approach – Challenges

Top challenges:

(2) Lignin-based polymers match specs of BPA based polymers (glass transition temperature, storage modulus)

- Risk analysis: Mechanical analysis of polymers
- Risk identified: Pre-polymer purity
- Risk mitigation: (1) Frequent lab scale polymer testing to assess impact of process changes
  (2) Material evaluation by customers

Lignin-based thermoset

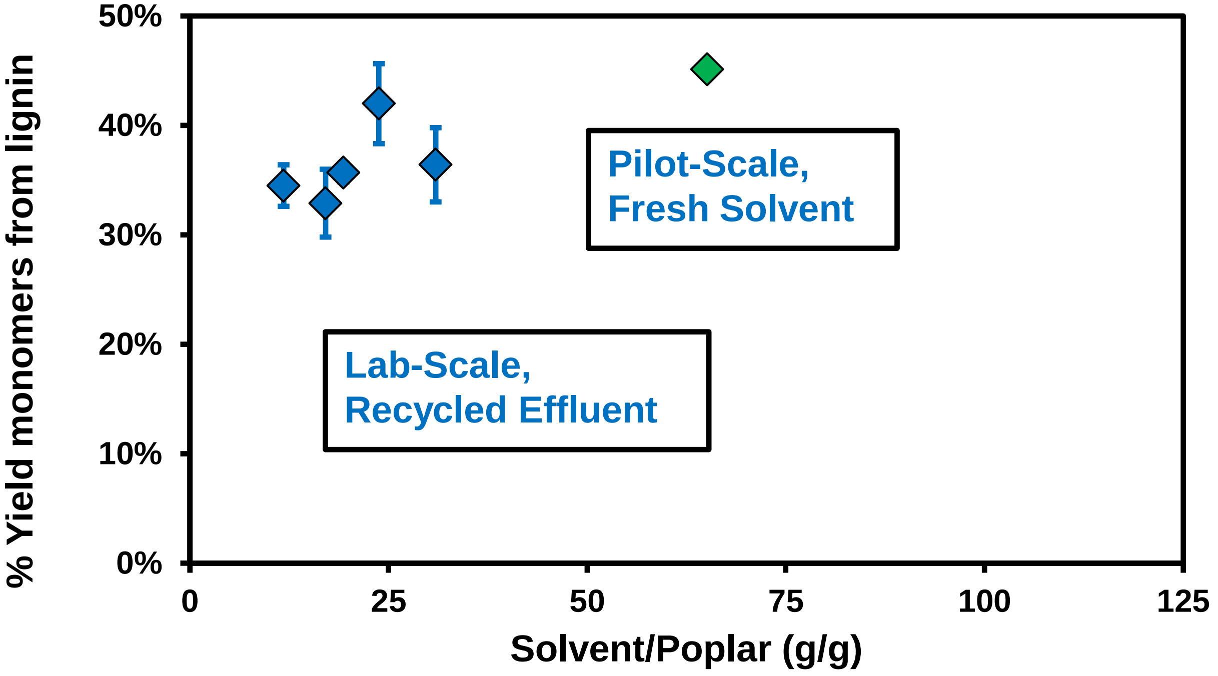
Progress and Outcomes

Task 1: Scaling SPERLU to 200 g bio-phenols

- ✓ M1.1 Lignin extraction kinetics
- ✓ M1.2 Catalyst selection
- ✓ M1.3 Reactor construction
- ✓ M1.4 Catalyst kinetics
- ✓ M1.5 Catalyst regeneration methodology
- ✓ **M1.6/ GNG 2:** 200 g bio-phenols at 40% yield (04/2021)
- ✓ **M1.7** Bio-phenol separations at 200 g scale (02/2023)

Progress and Outcomes: Task 1 Addressing Solvent Use

Monomer yield from poplar with solvent recycle

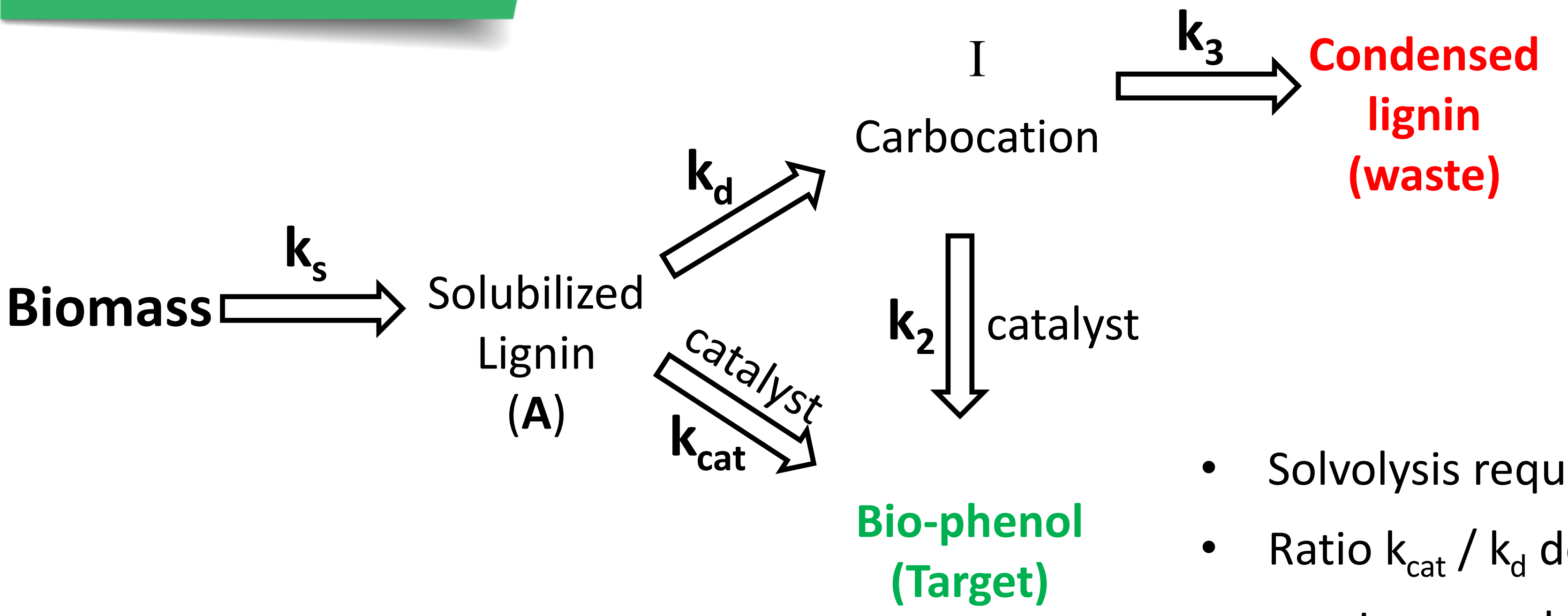


- Solvent : biomass = 62:1 (no recycle)
  - 40-50% monomer yield from poplar
- Solvent recycle reduces ratio to 10:1
  - 30-40% monomer yield from poplar
- Technical lignin reduces ratio to 5:1

<u>Reductive Catalytic Fractionation (RCF) Approach</u>

- Investigated in Plug-Flow Reactors
- Pilot scale: 200-300 g poplar, lab scale 10-20 g poplar

Mechanistic Understanding of Solvent Demand and Yield



Reductive Catalytic Fractionation (RCF) Approach

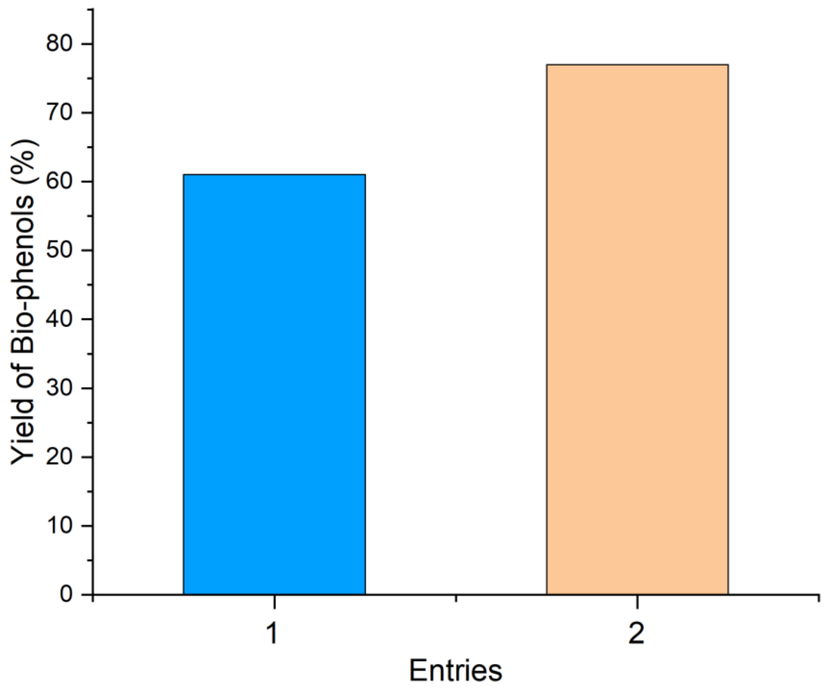
- Investigated in Batch and Plug-Flow Reactors

Key Learnings

- Solvolysis requires wetted biomass (solvent intensive)
- Ratio k_{cat} / k_d determines product yield
  - Increased catalyst loading or solvent volume favors k_{cat} over k_d
  - k_d is concentration dependent
- Technical lignin removes solvent demand for solvolysis

SPERLU™ Technology with Technical Lignin

Bio-phenol yield from technical lignin



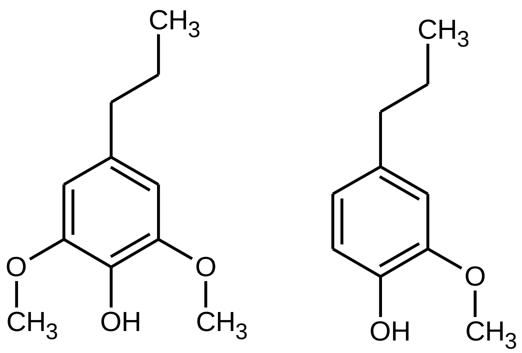
- 60-90% Yield soluble bio-phenols
- TEA mixed solvent systems under investigation
- Technical lignin commercially available

Conditions: 5 g lignin, 40 g solvent, catalyst, temperature > 100 °C, time 6 hrs
*Entry 1: uses single solvent system (basis of TEA), Entry 2 uses mixed solvent system (TEA under investigation)
**90% yield soluble bio-phenols achieved with longer reaction time

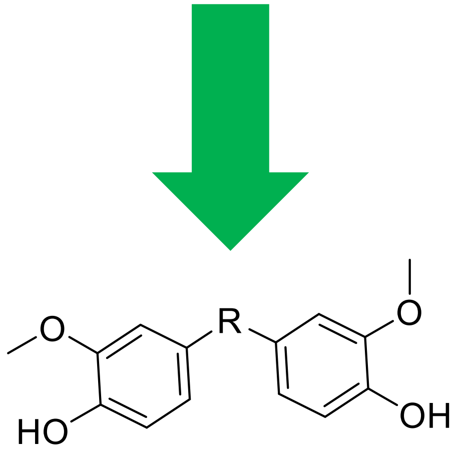
Milestone 1.7 – SPERLU™ Monomer Purification

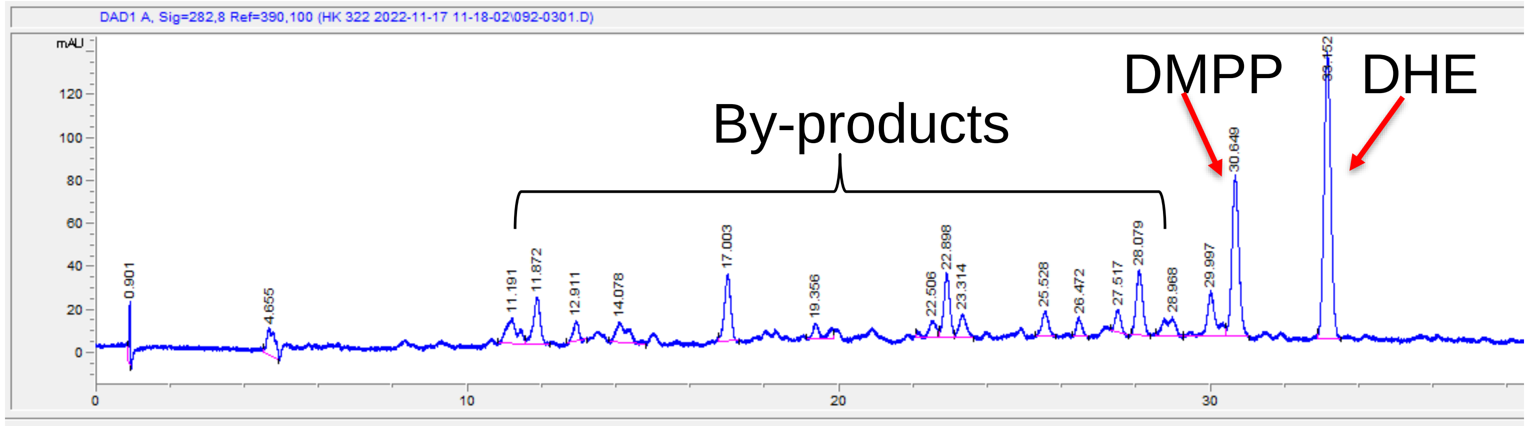
SPERLU™ with poplar produces 40 wt% DHE & DMPP from lignin

- 100 g poplar yields: 10 g monomer + 60 g carbohydrate
- Monomer purification & upgrading requirements:
  - High temperature vacuum distillation
  - Dimerize to bisphenol



DMPP DHE





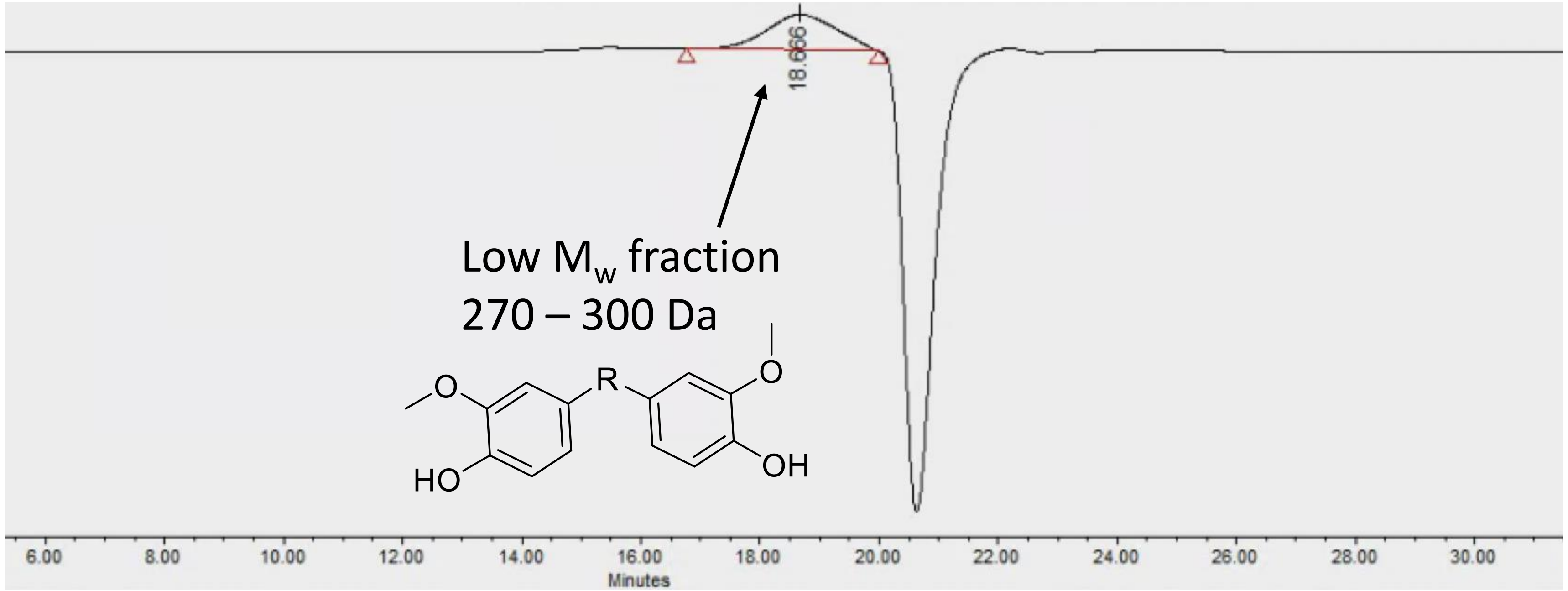
HPLC Chromatogram: SPERLU products from poplar

Milestone 1.7 – SPERLU^{TM} Monomer Purification

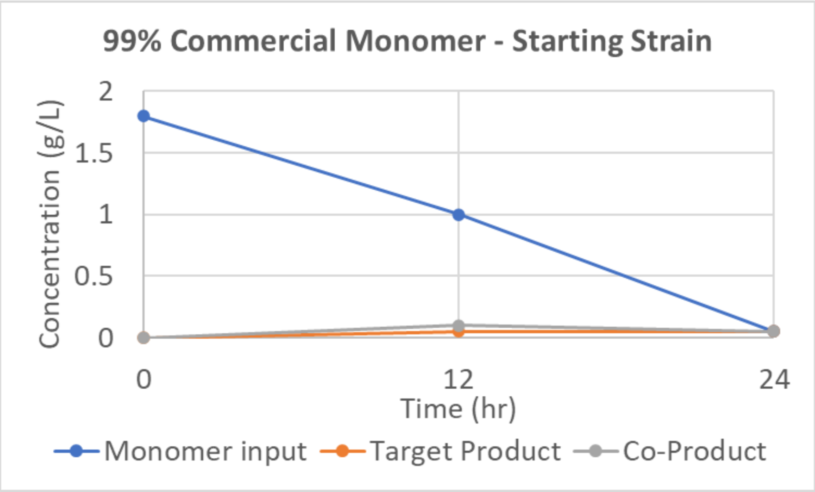
SPERLU^{TM} with technical lignin produces >60-90 wt% soluble bio-phenols

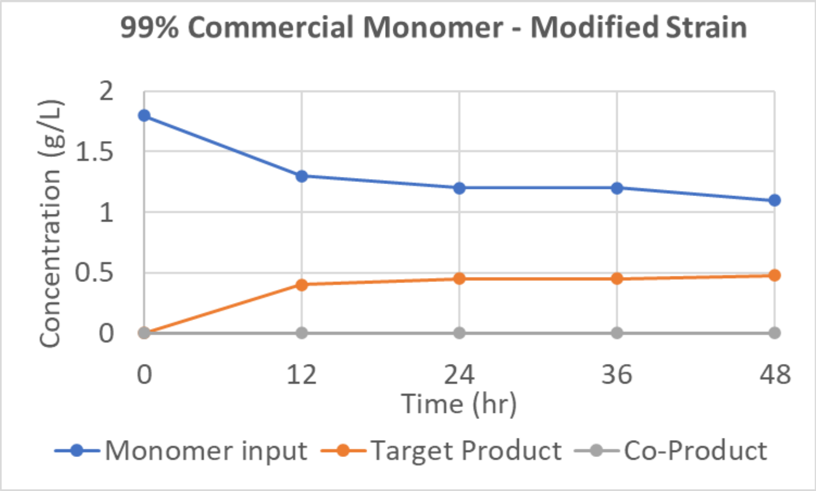
- 100 g lignin yields: 60-90 g bio-phenols
- Bio-phenols mainly dimeric
- Distillation & dimerization not required

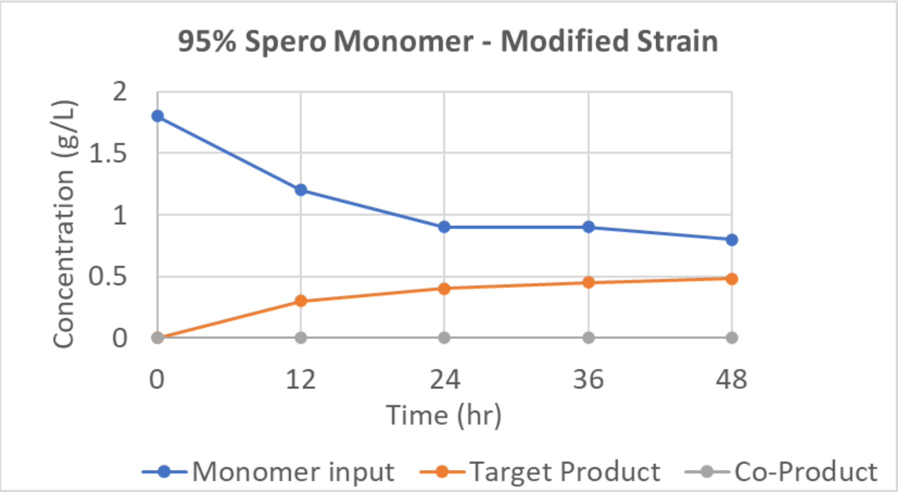
Gel Permeation Chromatography (GPC) of soluble bio-phenols (60% yield)



Task 2 Progress & Outcomes: Biotransformation Optimization







<u>Key findings</u>

- *Pseudomonas* putida genetically modified
  - Eliminating undesired co-products
  - Minimized target product metabolism
- Spero monomer performance similar to commercial standard
- Biotransformation not commercially viable

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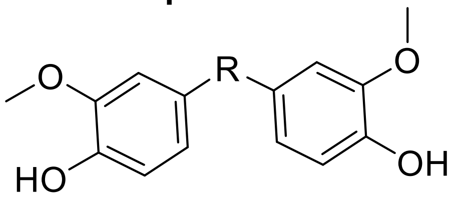
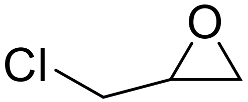
Task 3 Progress & Outcomes: Lignin to Thermoset Polymers

Technical lignin

SPERLU™

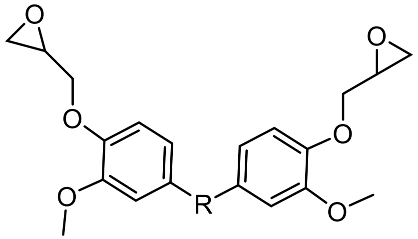
Bio-phenols





Epoxidation

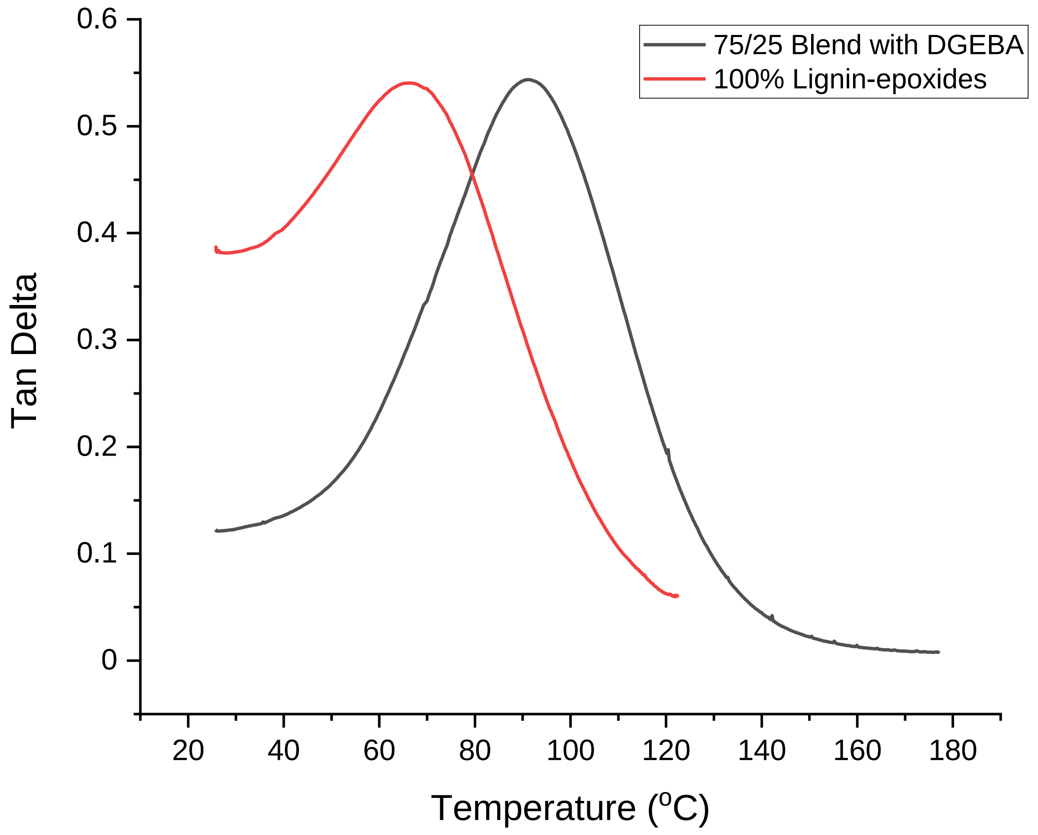
Bio-based epoxides

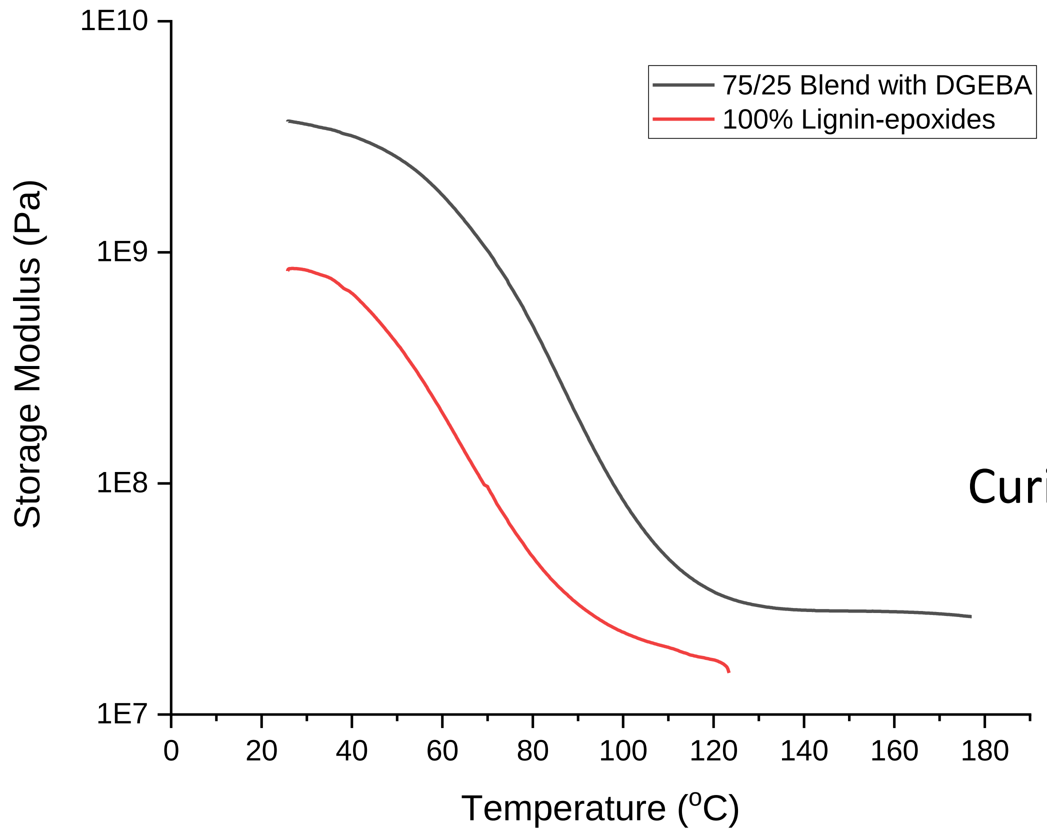


- Bio-phenols from Task 1 upgraded
- Blends with current formulations
- Matches petroleum-based specs. (glass transition temperature, storage modulus)

Task 3: Lignin based Thermosets meet Technical Targets

Dynamic mechanical analysis (DMA) of Lignin based Thermosets





Curing agent: DETA

❖ T_g between 65 - 90℃, Strong storage modulus at room temperature ≥ 1 GPa

Task 5: SPERLU TEA Progress and Outcomes– OPEX and Indicators

NPV	**$274,836,749**
NPV %	**12%**
ROI	**140%**

- Technical lignin feed
- 15 year plant lifespan
- 5 year catalyst replacement period
- Catalyst cost estimated using CatCost Tool at ChemCatBio.org

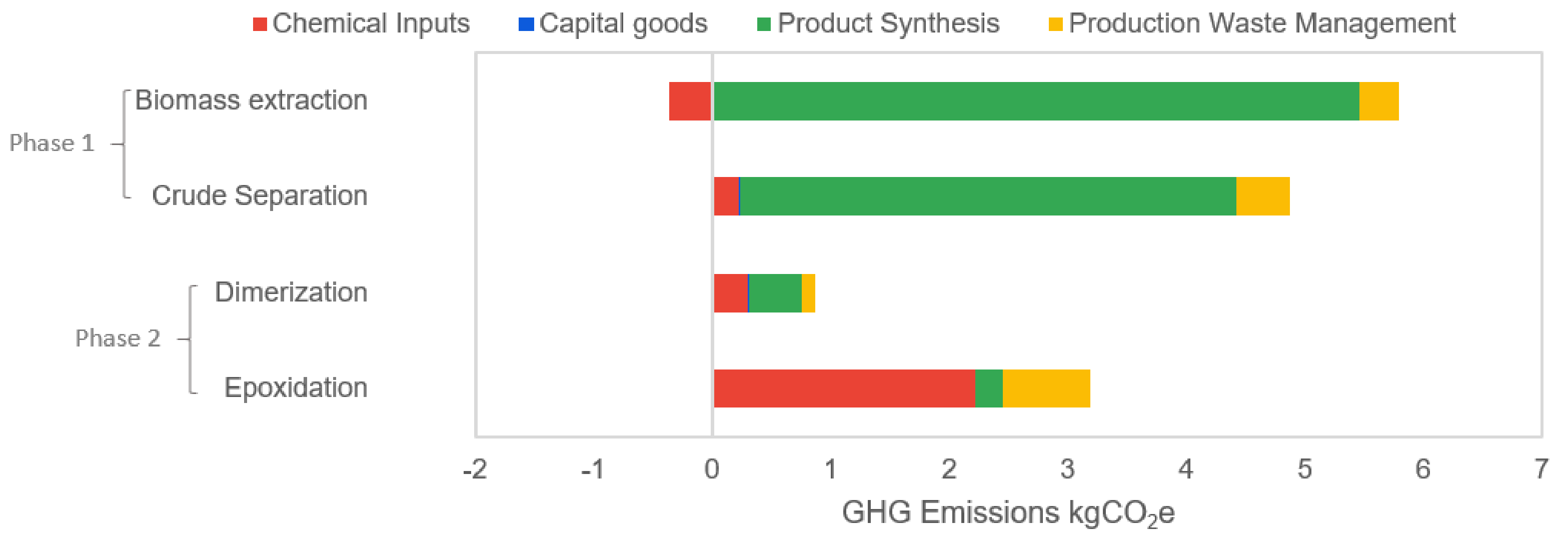
	Mass Flows		**Price**
	Name	**Mass Flow Rate [kta]**	**Annual [$MM/y]**
Feedstock	Lignin	21.0	-7.4
	Catalyst & Reagents	-	-43.9
	Solvents	-	-12.12
			-63.4
OPEX (waste + utilities)	Liquid Waste	-	-4.4
	Solid Waste	-	-0.2
	Cooling Water	17.1 MW	-0.11
	HP Steam	16.4 MW	-1.02
	Electricity	0.2 MW	-0.08
			-5.69
Product	Lignin Epoxide		147.0

Total Revenue	$147.0 MM/yr
Total Costs	-$69.1 MM/yr
Balance	$77.9 MM/yr

Task 5: SPERLU LCA Progress and Outcomes
Use of technical lignin vs wood removes unit operations and reduces GHG

Using Technical lignin vs wood eliminates unit Operations

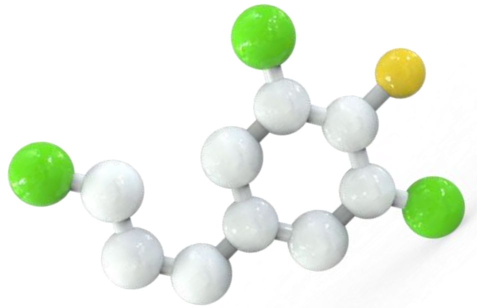
- Crude separation
- Dimerization



Resulting Impact on Green-House Gas (GHG) Emissions:

- GHG: 38% decrease

Impact & Summary



- SPERLU™ technology applicable to commercially available technical lignin
  - 60-75% yield bio-phenols from technical lignin
  - Scale-up to MT lignin in planning stages
  - Collaborating with leading paper company
- Bio-phenols upgraded to thermoset polymers
  - Drop in replacement for epoxidized bisphenol A (DGEBA)
- TEA: Positive NPV of 12%
- LCA: 38% GHG reduction when technical lignin used vs poplar completed
  - TEA & LCA used to inform process modifications (risk mitigation)

Spero from nature

Acknowledgements

Spero from nature

NREL NATIONAL RENEWABLE ENERGY LABORATORY

VITALMETRICS

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Milena Rangelov
Ilayda Dinc
Yirui Zhang
Sangwon Suh

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

Quad Chart Overview

Timeline
- Project start 01/2019
- Project end 03/2023

	FY22 Costed	Total Award
DOE Funding	$1,307,347	$1,613,457
Project Cost Share *	$376,353	$419,707

TRL at Project Start: 3
TRL at Project End: 5

Project Goal
Scale-up & validate SPERLU process, converting >50% lignin in non-food biomass to bio-phenols. Make polymers from lignin monomeric bio-phenols and bioprocess bio-phenols into chemicals.

End of Project Milestone
- Produce 1 kg bio-phenols
- ≥ 50% lignin conversion to bio-phenols
- Purify bio-phenols to ≥ 90% purity
- Synthesize bio-polymers matching properties of BPA thermosets
- Optimize biotransformation of select bio-phenols

Funding Mechanism
BEEPS DE-FOA-0001916
Lignin valorization
2018

Project Partners*
- NREL (Gregg Beckham, PhD)
- Vital Metrics (Sangwon Suh, PhD)

Additional Slides

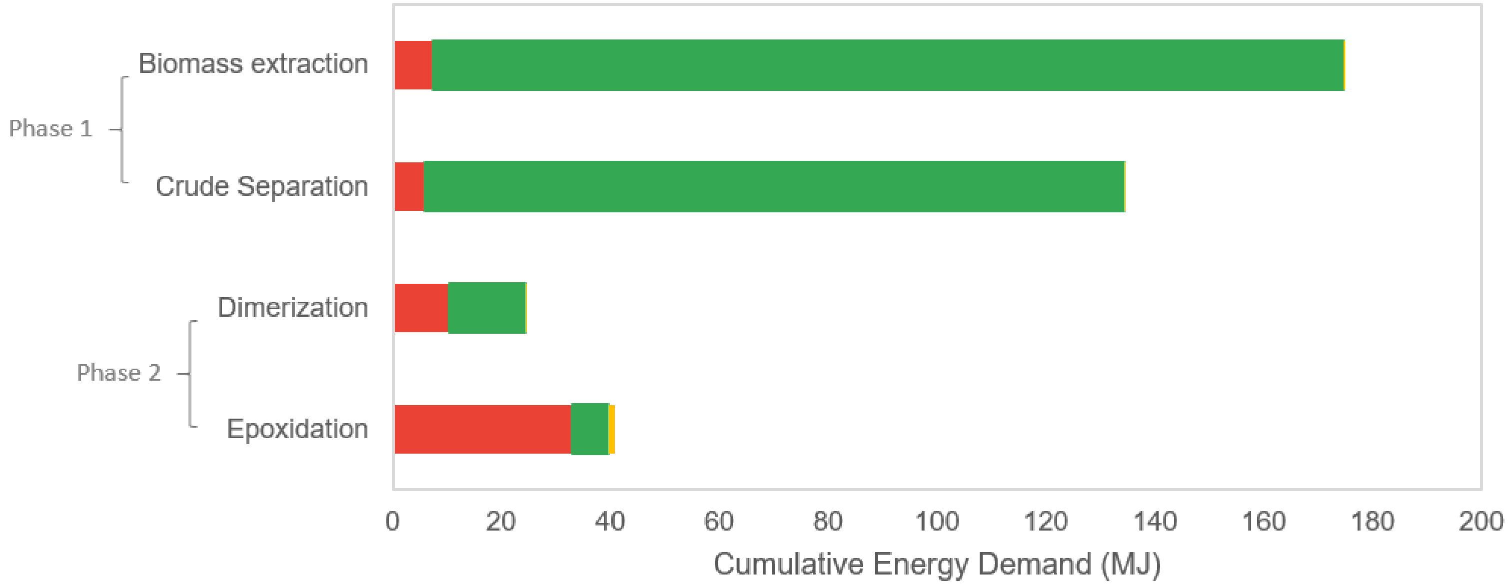
Task 5: SPERLU LCA Progress and Outcomes – Technical lignin vs wood

Using Technical lignin vs wood eliminates unit Operations

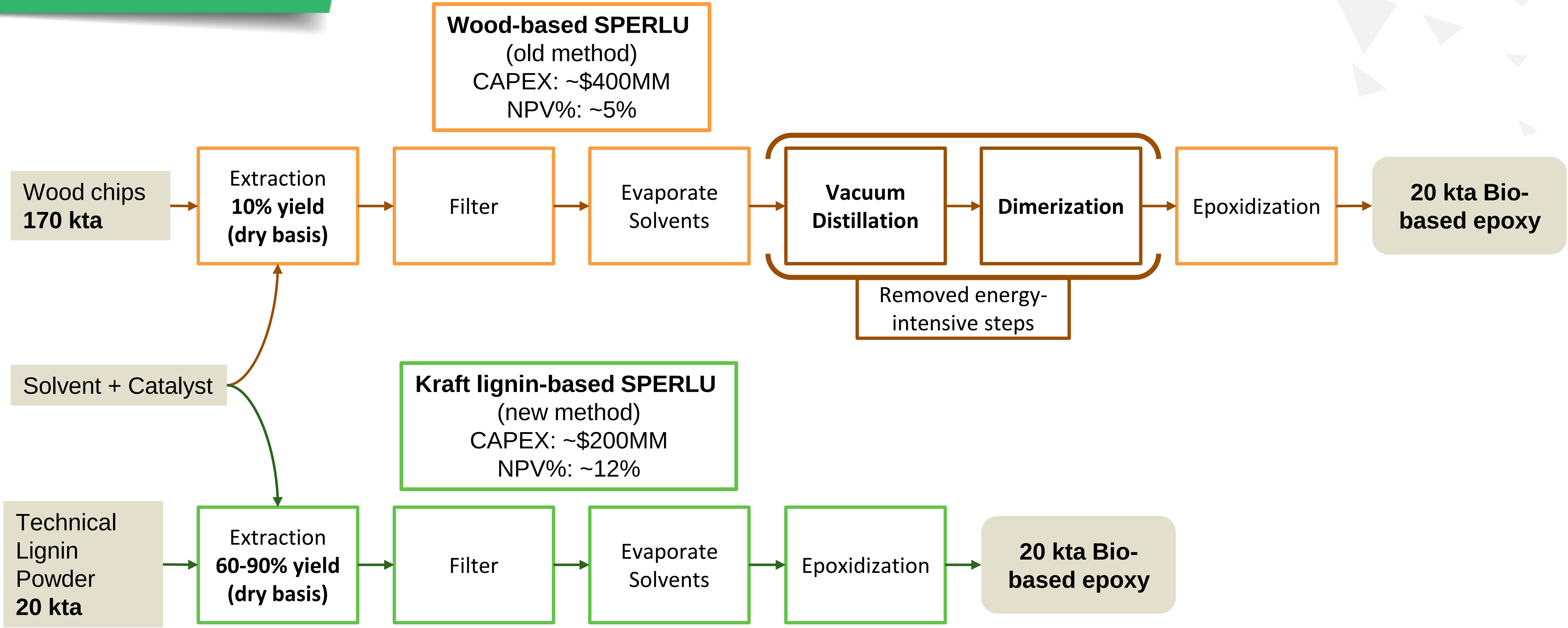
- Crude separation
- Dimerization

Resulting Impact on Cumulative Energy Demand (CED):

- CED: 42% decrease



SPERLU Biomass vs. Technical Lignin Feedstock



Products

<u>Presentations</u>:

Klein, I. Lignin Conversion and Upgrading to Materials. Presented at Advanced Biofuels Leadership Conference, Washington D.C., March 18, **2021**

Klein, I. Lignin Valorization for use in Bio-Based and Recyclable Materials. Presented at meeting of the American Chemical Society, San Diego, CA, March 23, **2021**

Klein, I. Innovations in Selective Lignin Upgrading to Create Profitable and Sustainable Chemical Businesses. Presented at meeting of the American Chemical Society, Chicago, IL, August 23, **2022**

Response to 2021 Peer Review Comments

- Regarding feedstock variability and selection: Spero has now identified a commercially available (and highly uniform) technical lignin source and is working directly with the lignin producer.