DOE Bioenergy Technologies Office (BETO) 2023 Project Peer Review

Enabling Lignin Valorization with Liquid Liquid Chromatography

Performance-Advantaged Bioproducts and Bioprocessing Separations

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DynaMic Extractions
World-class High Performance CCC

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Project Overview

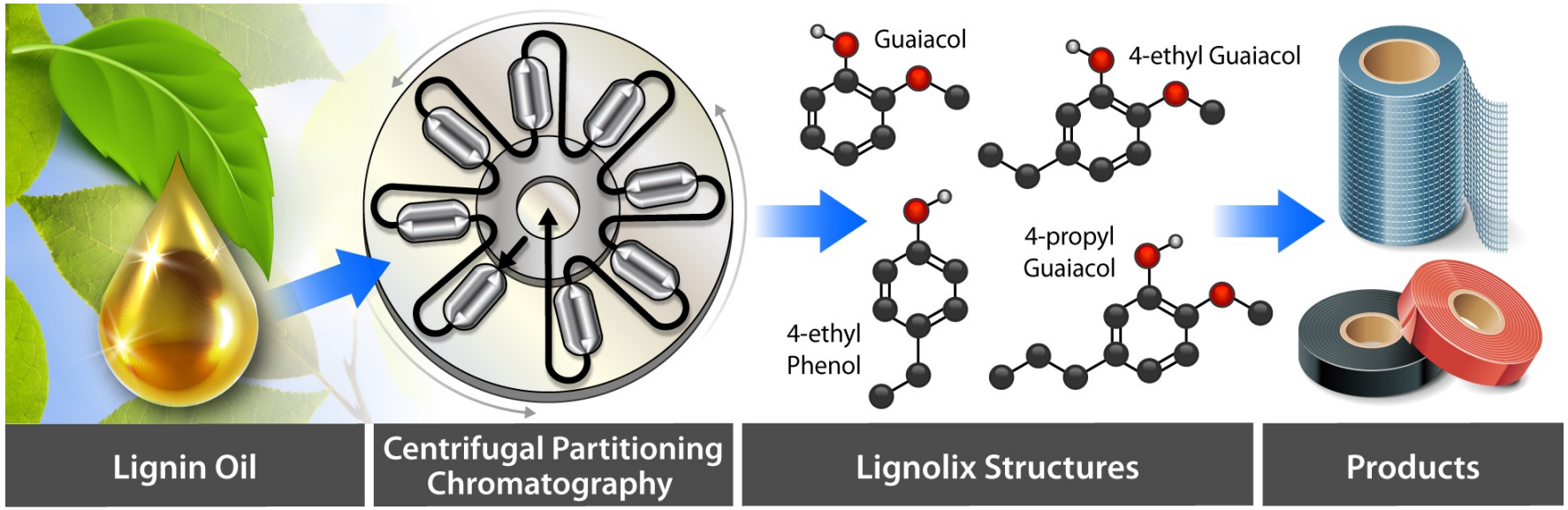
Goal:
Scalable separations for complex lignin-derived product mixtures

Current limitations:
Distillation is challenging/energy intensive for lignin deconstruction. Liquid liquid chromatography is not proven for high-volume commercial use

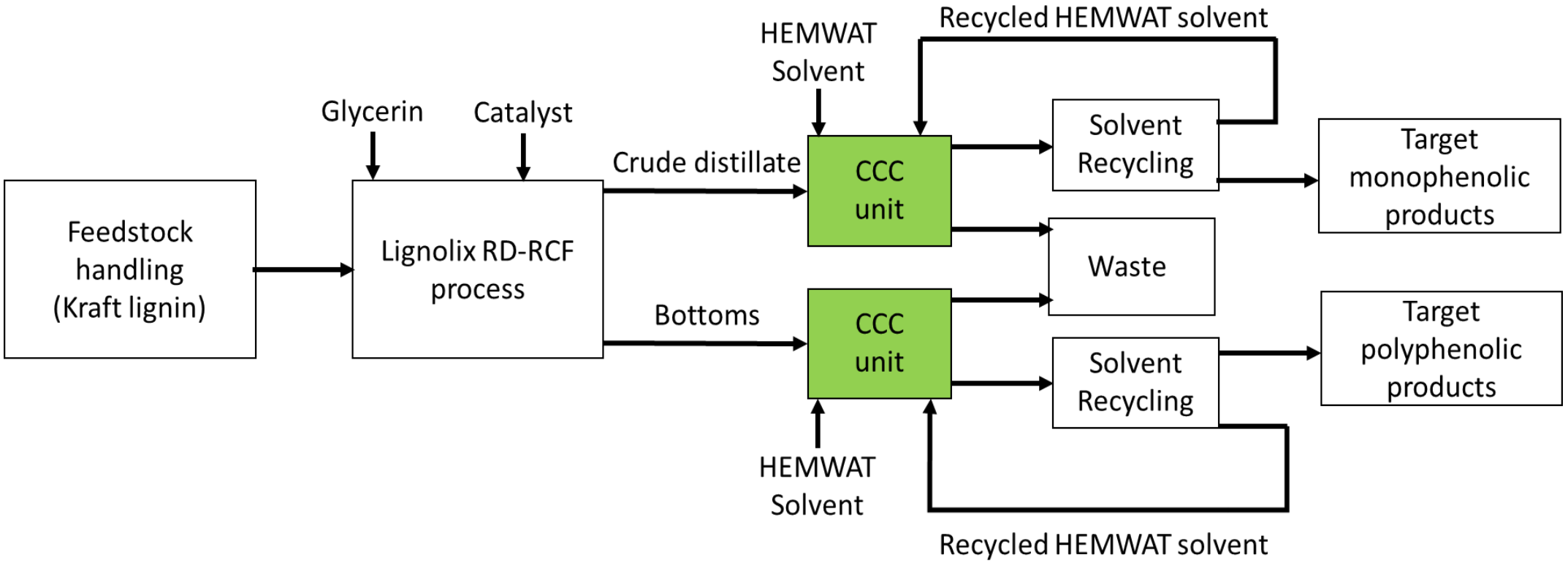
Approach:
Scale Counter Current Chromatography (CCC) and develop continuous CCC for lignin deconstruction product streams

Relevance:
Enable renewable chemical co-products to achieve low-carbon drop-in biofuels at $2.50 per GGE with >70% reduction in greenhouse gas emissions vs petroleum

Risks:
Complexity of controls to enable continuous operation; upfront capital cost; unproven at scale



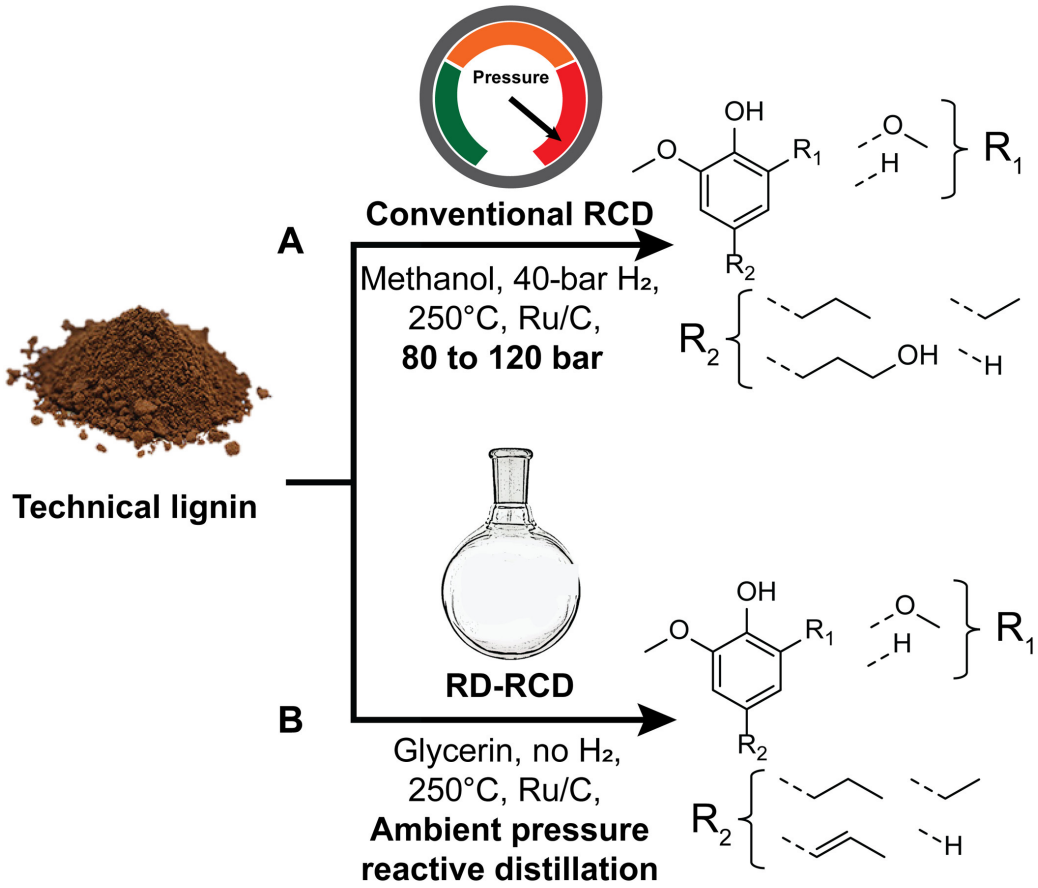
Project Overview



Approach

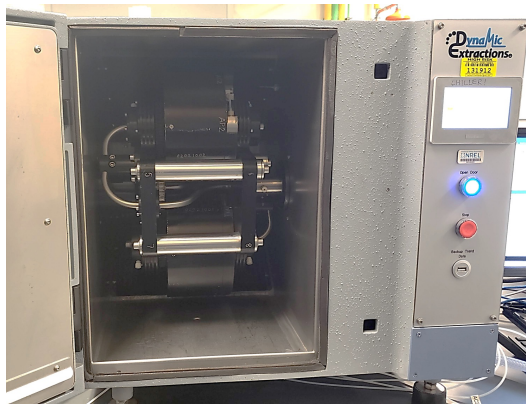
Reactive Distillation Reductive Catalytic Deconstruction (RD-RCD)

- **Benefits of RD-RCF**
  - Easier/less expensive to scale
  - Improved process economics
  - Reduced energy requirements
  - Effective separations are critical to commercial feasibility with reduced environmental footprint
- **Approach for this project**
  - Use commercially available sources as feedstock
  - Optimize reaction conditions
  - Develop continuous process
- **Risks/Mitigation Strategies:**
  - Scale-up hindered by mass transfer limitations – reactor design with better mixing and/or improve catalyst contact
  - Catalyst lifetime – explore catalyst regeneration strategies and/or switching to less expensive catalysts

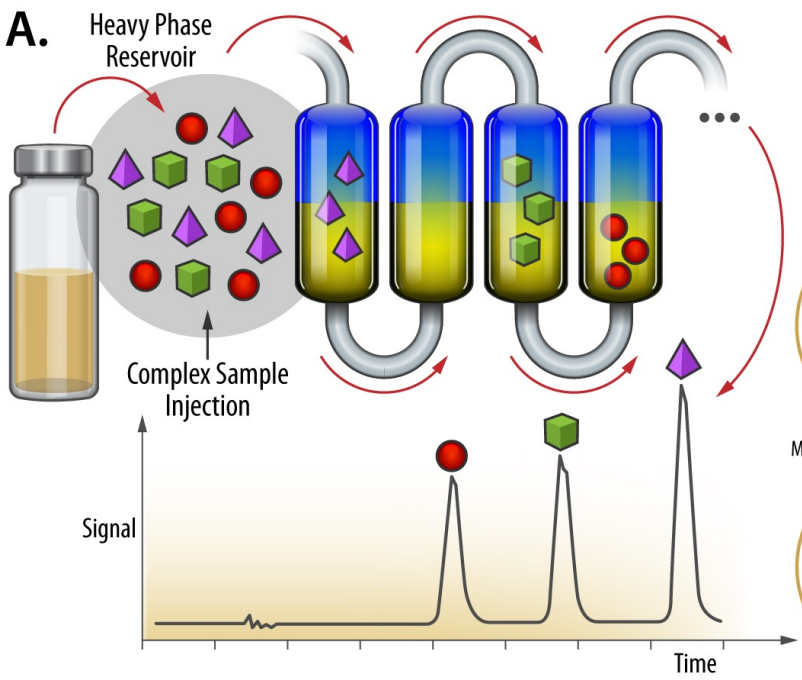


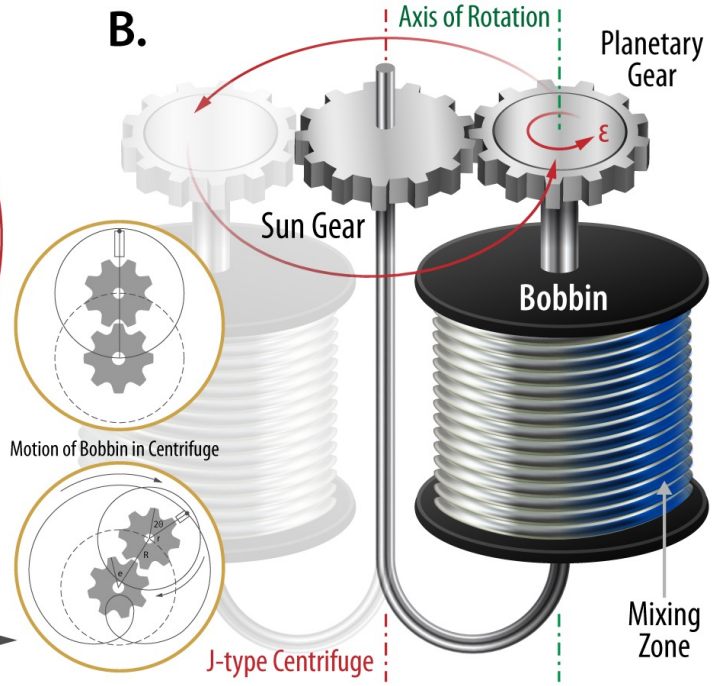
Approach

Counter Current Chromatography (CCC)



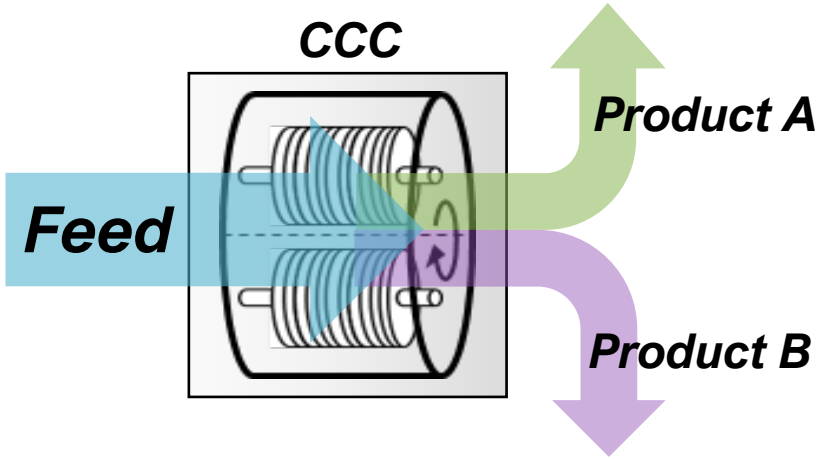
- Uses an immobilized liquid phase for separations
  - More efficient than other condensed phase separations
  - Not subject to fouling
  - Does not need upfront filtration
  - Enable full product recovery (no irreversible adsorption)
- <u>Uniquely well-suited to challenges of lignin coproducts</u>





Approach

- **Continuous CCC**
  - Conceptually analogous to True/Simulated Moving Bed
  - Periodic valve switching for continuous separation
  - Extend NREL predictive CCC model for continuous
- **Risks/Mitigation Strategies:**
  - Solubility of monomers in solvent system limits productivity at scale – investigate solvent systems other than HEMWAT solvent systems
  - Complexity of switching sequence – use valve block system from a traditional Simulated Moving Bed (SMB) for switching



Setup Continuous CCC system

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Develop Mathematical Model for Continuous CCC Process Design

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Demonstrate Model-based Design at Lab Scale

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Perform Scale up at Pilot Scale Process

Project Management - Team

Task	Activity
1	Verification
2	TEA/LCA
3	Lignin processing
4	Batch chromatography
5	Intermediate verification
6	Continuous chromatography

Project Organization

- Monthly team meetings
- Quarterly team meetings with DOE project managers
- Shared Dropbox for file management

lignolix

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Project Management

- Project start date: 10/1/2021
- BP2 conditions lifted: 12/15/2022
- Project end date: 4/30/2025

Task	Activity	Status
1	Verification	Complete
2	**TEA/LCA**	
2.1	Batch mode TEA/LCA	In progress
2.2	Continuous mode TEA/LCA	BP3
3	**Lignin processing**	
3.1	50 g scale lignin processing	Complete
3.2	500 g scale lignin processing	BP3
4	**Batch chromatography**	
4.1	Solvent screening	In progress
4.2	Parameter optimization	
5	Intermediate verification	
6	Continuous chromatography	BP3

Go/No-Go and End Goals

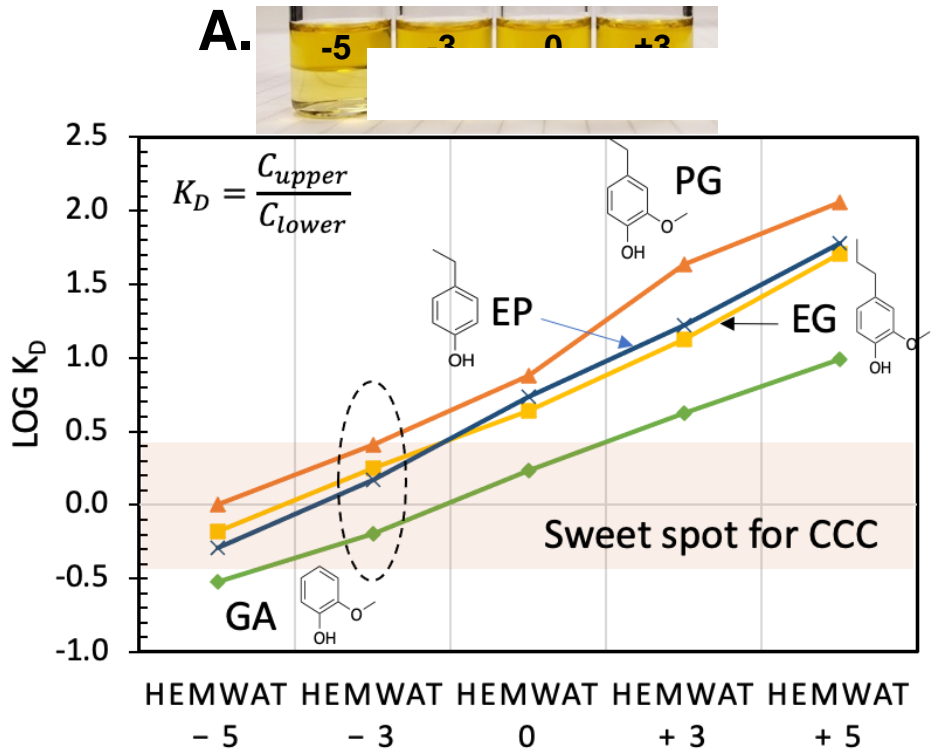
Go/No-Go

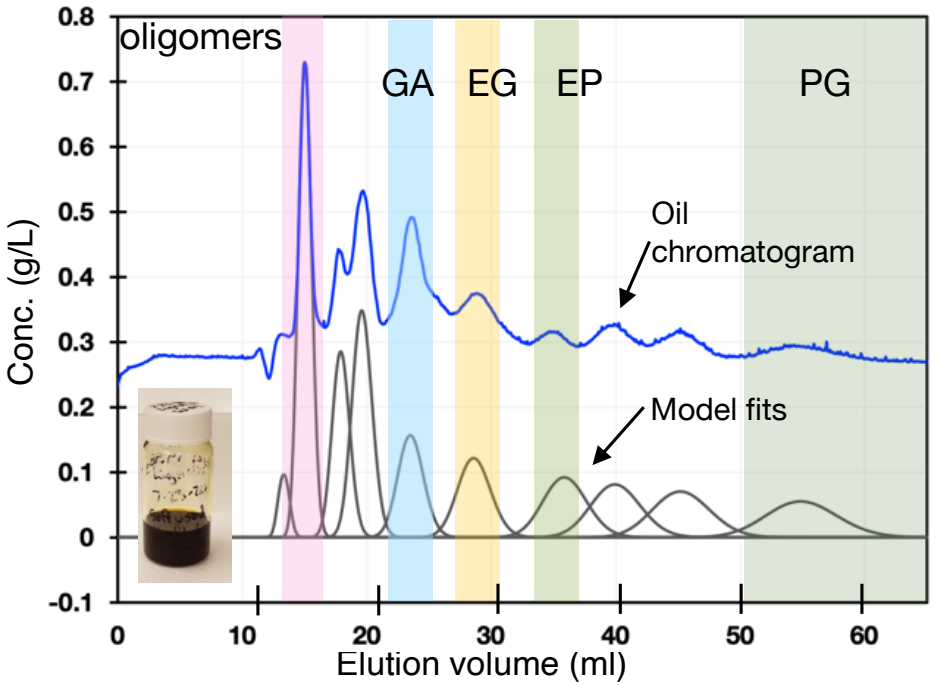
- Demonstrate that > 30% energy saving compared to batch CCC is achievable
- Demonstrate feasibility to produce:
  - > 500 g of raw distillate
  - > 1 kg of reaction bottoms
  - > 1 kg of isolated products
  - > 1 kg of isolated oligomers
- Deliver > 25 g of isolated monomer mixtures and oligomer mixtures (> 90% purity) from raw RCD products

End Goals

- 100 continuous hours of stable operation for CCC
- Recover > 1kg of isolated products above duration
- Scale up RD-RCD for continuous operation to match CCC

Progress and Outcomes: Task 4.1 (in progress)

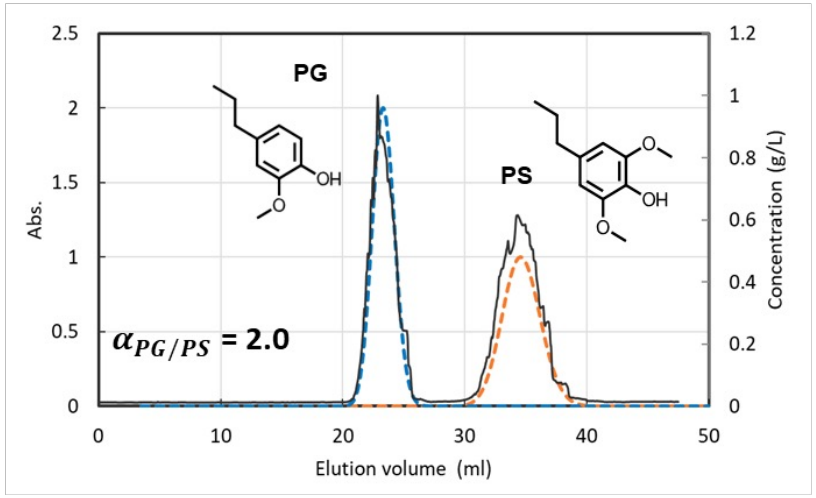




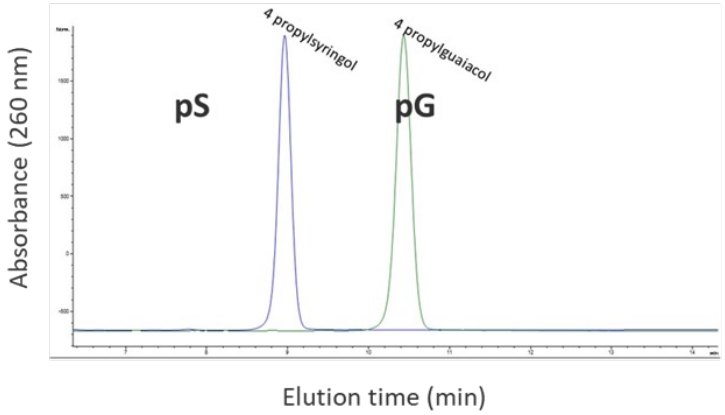
- **Solvent screening for batch CCC**
  - (A) Partition coefficient measurements for HEMWAT solvent systems
  - (B) Chromatogram of biooil after CCC. Good fit with predictive model
  - Demonstrates >90% yield and >97% purity are achievable for individual compounds

Progress and Outcomes: Task 2.1(in progress)

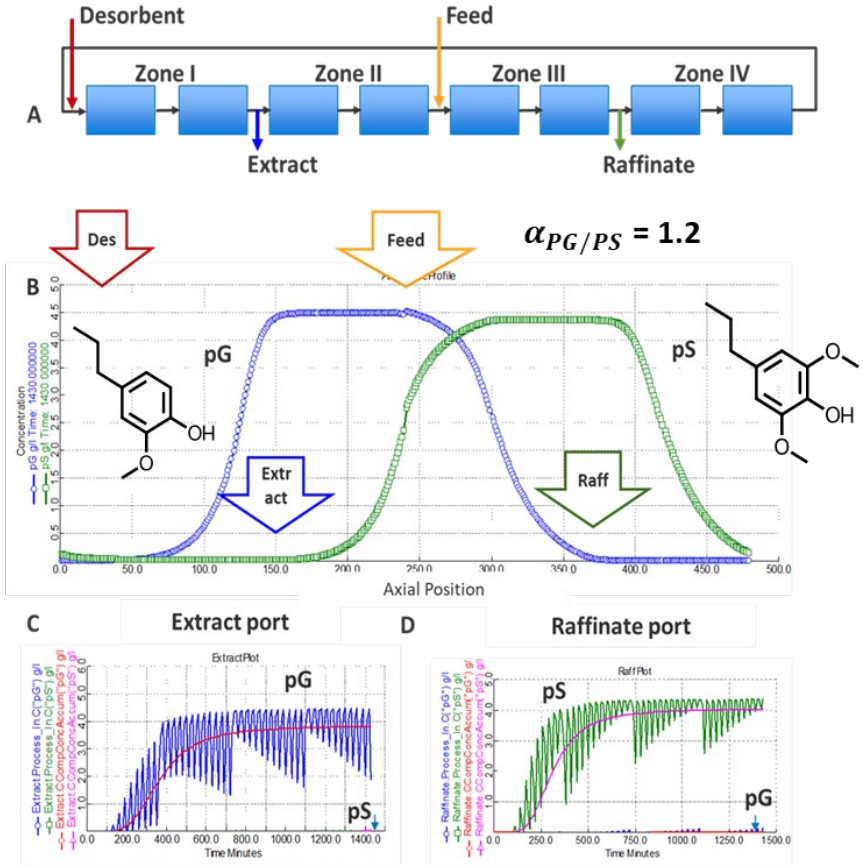
CCC Exp & Simulations for PG & PS Separation



Flash (Batch LC) Exp for PG & PS Separation

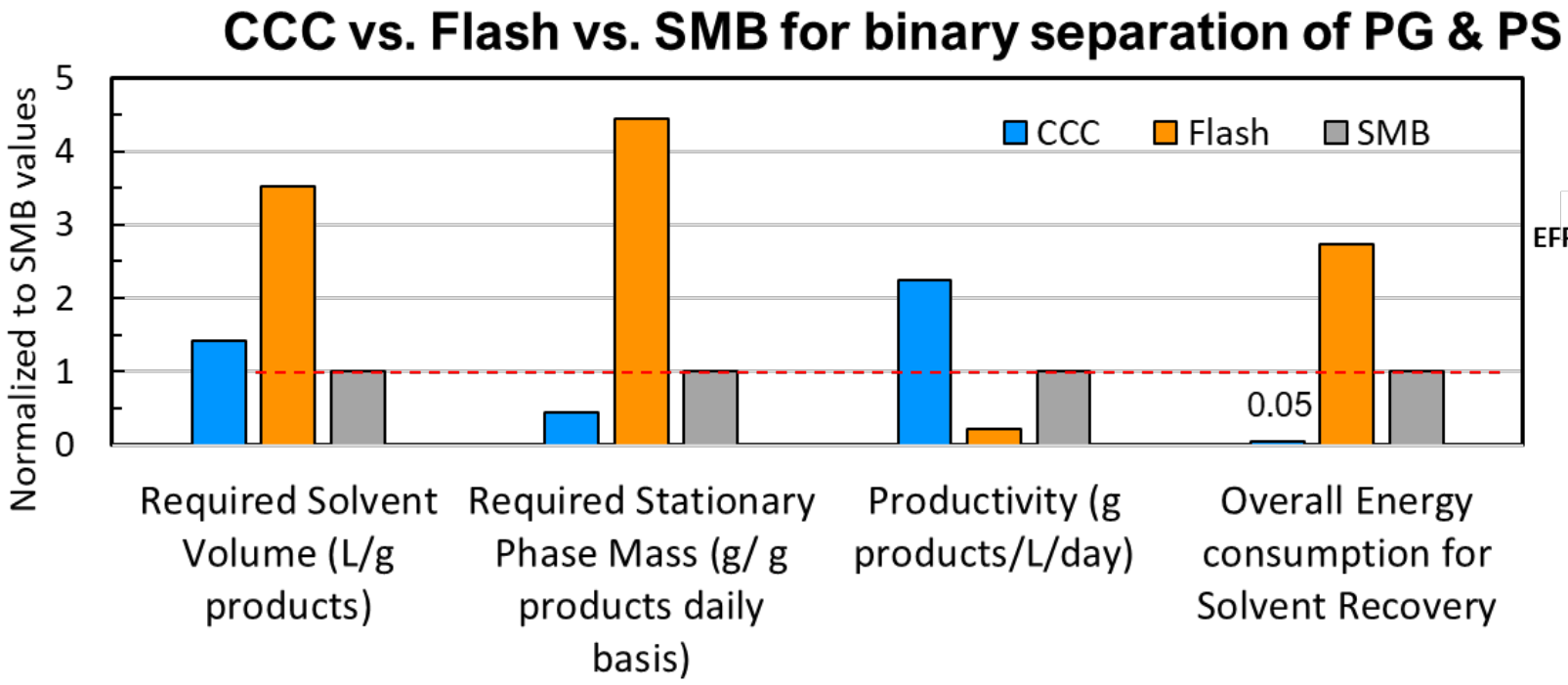


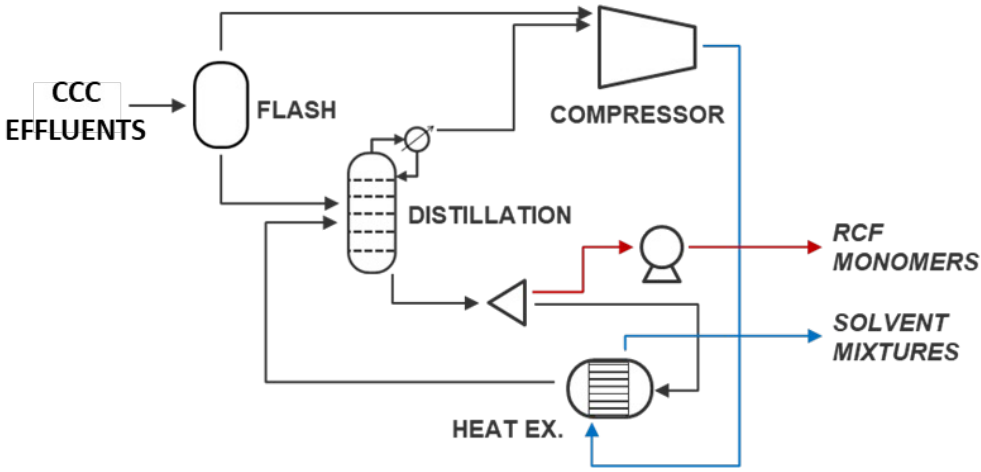
SMB Simulation for PG & PS Separation



- **Comparison between batch CCC, flash chromatography, and SMB assessing:**
  - Solvent volume
  - Productivity
  - Energy consumption

Progress and Outcomes: Task 2.1 (in progress)





- **Preliminary comparative TEA/LCA for batch CCC**
  - Energy consumption reduced by ~95% vs SMB
  - > 100% increase in productivity vs SMB
  - Further improvements expected in moving to continuous CCC

Impact

- **Impact for Biorefineries**
  - Creation of co-product streams could reduce fuel selling prices by up to ~ $3/GGE MFSP
  - Focus on product development/commercialization of adhesives with industry partners
- **Improvements vs SMB**
  - Energy consumption reduced by ~95%
  - > 100% increase in productivity
  - Ability to handle solids can increase yields by 50%

- **Impact for Separations Consortium**
  - Develop public continuous CCC model tool
  - Extend predictive tools for continuous CCC
  - Scale CCC towards pilot

Summary

- CCC is a uniquely suited for downstream separations of lignin-derived products
- Batch CCC vs SMB:
  - Energy consumption reduced by ~95%
  - > 100% increase in productivity
- Continuous CCC is anticipated to improve the above metrics
- Goal of integrating a continuous lignin deconstruction process with continuous CCC

- **Completed tasks 1 and 3.1:**
  - Initial verification
  - Lignin deconstruction scale up and optimization for processing for 50 g
- **Tasks 2.1 and 4.1 in progress:**
  - TEA/LCA for optimized batch CCC
  - Shake flask experiments for solvent system selection

- **Future work**
  - Develop and scale up continuous RD-RCD
  - Develop continuous CCC columns, switching and predictive models

Quad Chart Overview

Timeline

10/1/2021 – 3/21/25

	FY22 Costed	Total Award
DOE Funding	$79,194	$2,499,196
Project Cost Share	$44,517	$627,500

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

Project Goal

Design and develop scalable liquid liquid chromatography for commercial-scale lignin valorization

End of Project Milestone

Produce >1 kg of products from raw distillate and oligomers using the continuous CCC operation. Target > 80% yield at > 90% purity and an energy savings > 50% over the incumbent SMB technology.

Funding Mechanism

FY21 BETO Scale-up and Conversion
FOA DE-FOA-0002396

Project Partners

NREL – Gregg Beckham
Dynamic Extractions – Colin Bright