NREL
Transforming ENERGY

BETO 2023 Peer Review

Solid Lignin Recovery (SLR)
WBS#: 3.3.4.601

April 5, 2023
Systems Development and Integration

Dan Schell
NREL

Project Overview

Need to convert all the biomass (carbohydrates and lignin) to achieve cost effective SAF production meeting BETO's GHG emission and LCA goals.

Problem: Solid lignin derived from the Deacetylation and Mechanical Refining (DMR) process or caustic-based pretreatment processes will not filter using conventional vacuum or pressure filtration techniques.

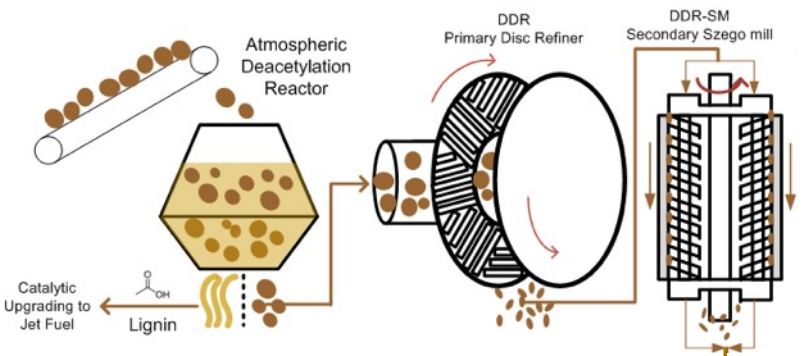
Project Goal: Find a promising solution for recovering this material using existing commercial, large-scale separation technology.

Project History: Initially a two-year (~$600,000) BETO seed project starting in FY20

- Mid-project (Go/No-Go decision point) reach in Dec. 2020 (one quarter behind)
- Targeted a mid-FY22 project end point (two quarters behind), but no funds received in FY22
- Work now finishing at end of FY23

DMR and Enzymatic Hydrolysis Process Background

Deacetylation and Mechanical Refining (DMR)



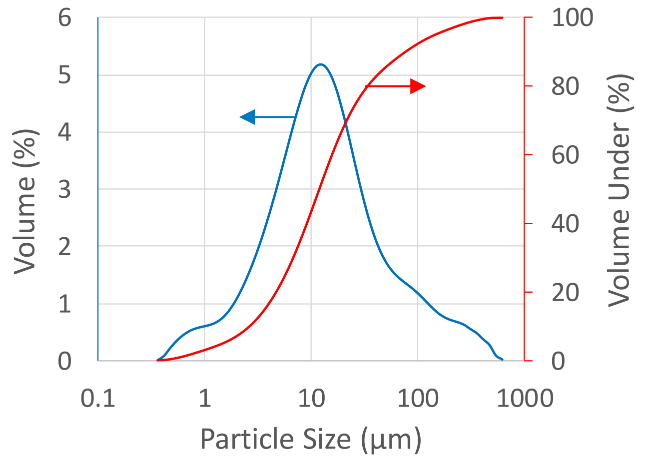
Mild caustic treatment followed by a two-stage refining process (high and low-consistency grinding)

New joint projects between NREL and industry have been awarded to further develop DMR technology and other BETO projects are using this material in various sugar-to-SAF pathways.

Enzymatic Hydrolysis → Solid-Liquid Separation (Water in) → Washed Lignin (Lignin Valorization)

Solid-Liquid Separation → Sugars (Biological Conversion)

Lignin Particle Characteristics



Approach

Problem: Need a cost-effective technique for washing (sugar recovery) and separating solid lignin from enzymatic hydrolysates.

State-of-the-Art: Flocculation followed by vacuum filtration

- Experimental performance data and TEA available* (baseline)
- Why an issue: High cost of flocculants; poor dewatering of the lignin sludge; high wash water usage; unknown impacts on downstream processes, e.g., fermentation, lignin upgrading

Process Parameter	Value
Sugar recovery (%)	95
Wash ratio (L/kg IS†)	17.5
Flocculant‡ loading (g/kg IS)	20
Equipment capacity (kg IS/m^2-h)	12
Insoluble solids (IS) recovery (%)	99.5
Recovered IS cake total solids content (%)	27

†IS: Insoluble solids

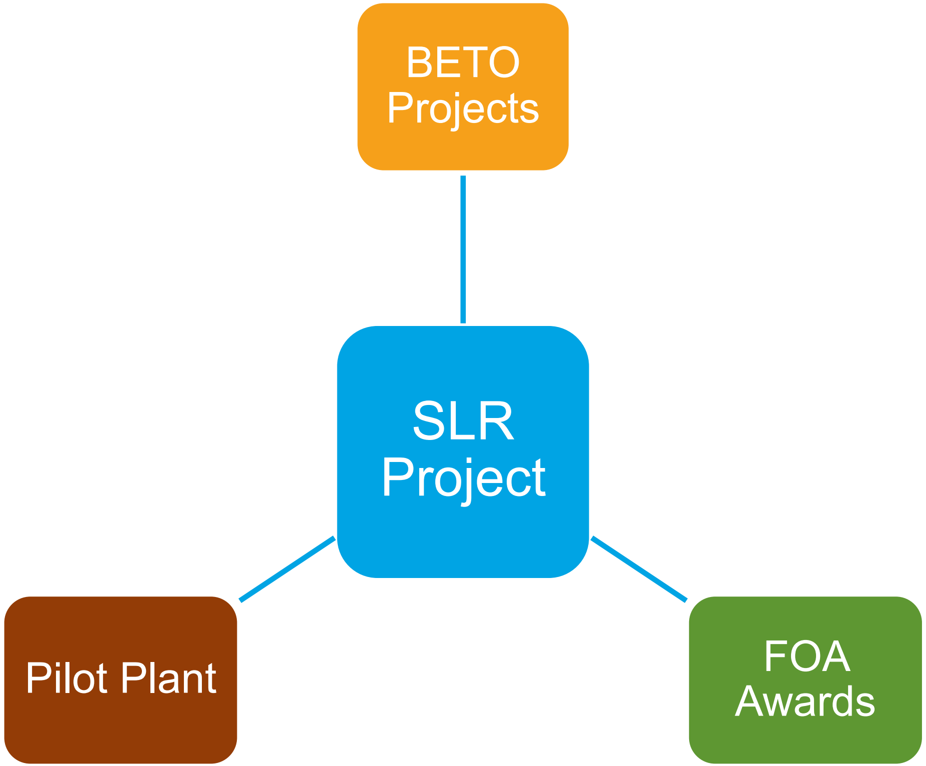
*Sievers, D. et. al. 2015. *Bioresourc. Technol*. 187, 37-42.

Approach

- No direct collaborations, but some interactions with BioProcessing Separations Consortium (BPSC)
- Risks
  - Changes in technical scope
  - Project delays

Project Team

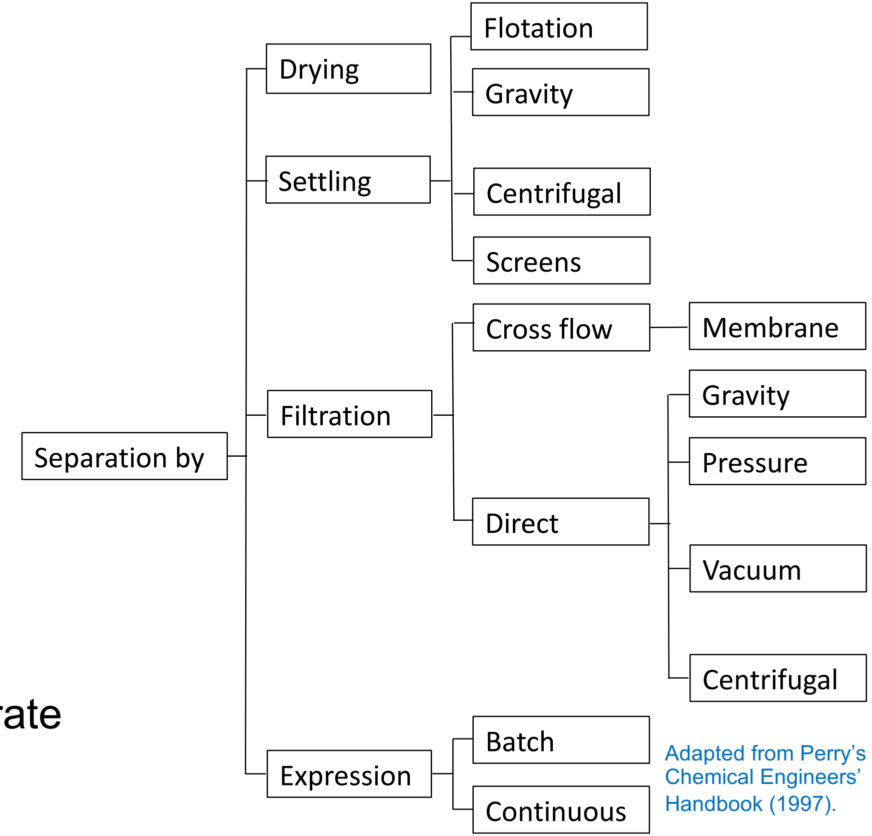
- PI/Management: Schell
  - Separations Science: Sievers, Saboe, Gunther
  - Plant Operations: Jennings, Baker Sievers, Gunther, Operating Staff
  - TEA: Davis, McNamara, Dempsey



Approach

Approach: Perform TEA using experimental data to identify the best (most cost-effective) separation technology.

- **First Half of Project** - Initial evaluation of non-flocculated options to compare to a flocculated process (Go/No-Go decision for future direction)
- **Second Half of Project** - Based on TEA (with associated Go/No-Go decision), continue developing most promising processes and generate pilot scale data for final TEA

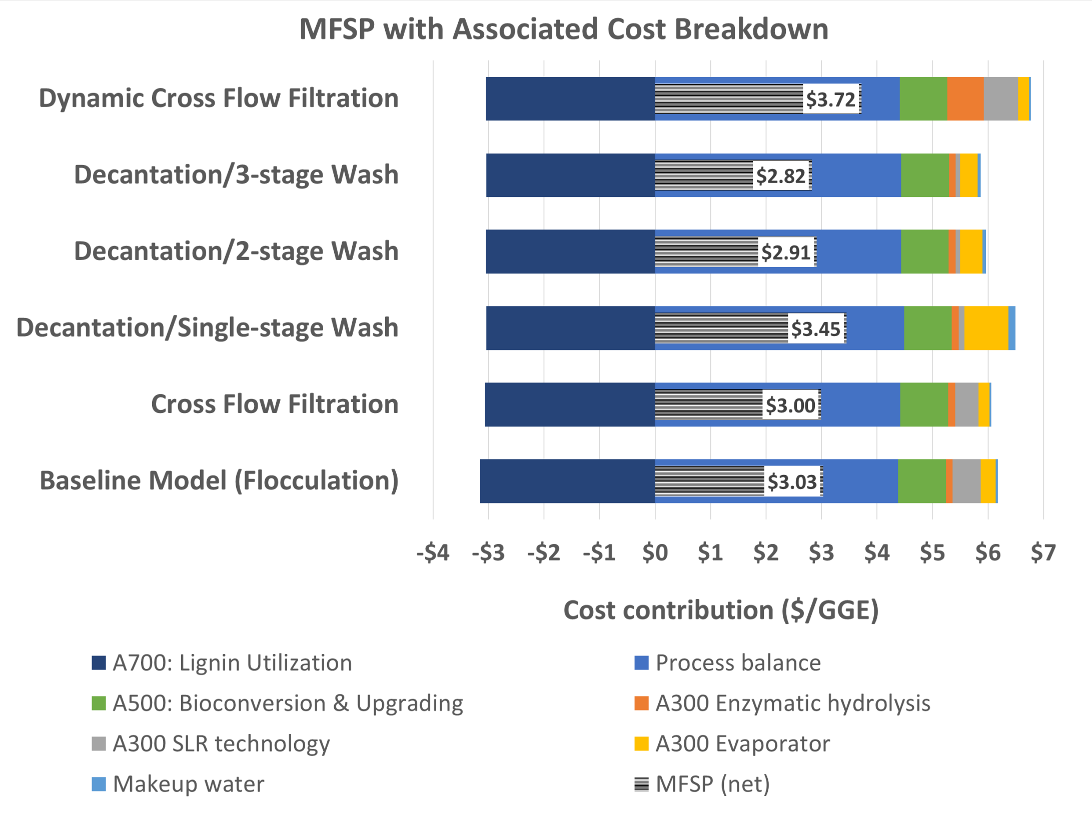


Adapted from Perry's Chemical Engineers' Handbook (1997).

Approach

Go/No-Go Decision Criteria: Go if MFSP for non-flocculated process < MFSP for flocculated process (baseline); otherwise, No-Go

- **No-Go**: Further evaluate flocculation-based processing
- **Go**: Continue more rigorous investigation of non-flocculated processing (pilot scale)



MFSP-minimum fuel selling price

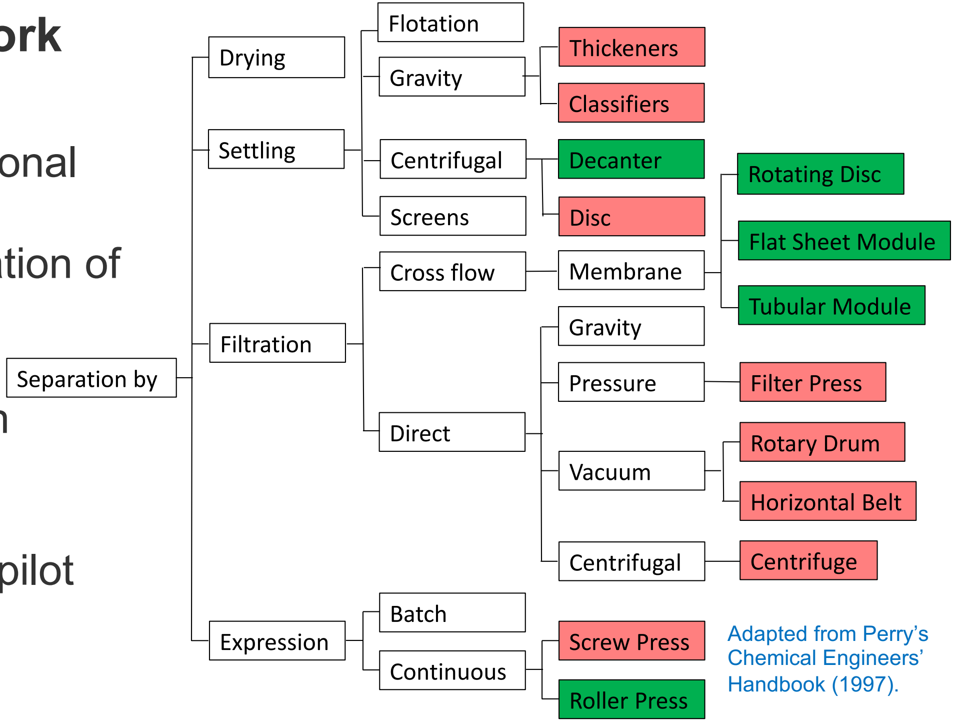
Approach

Based on the Go decision, remaining work elements included:

- Dynamic Cross Flow Filtration (DCFF), additional testing with bench unit completed, FY21 Q3
- Cross Flow Filtration (CCF), pilot scale validation of simulation results completed, FY21 Q4
- Expression evaluation completed, FY22 Q1
- Flocculant performance/cost review based on literature data completed, FY22 Q2
- Electrocoagulation (vendor study completed)
- Centrifugation (decantation) testing in NREL pilot plant ongoing
- Final TEA assessment (ongoing)

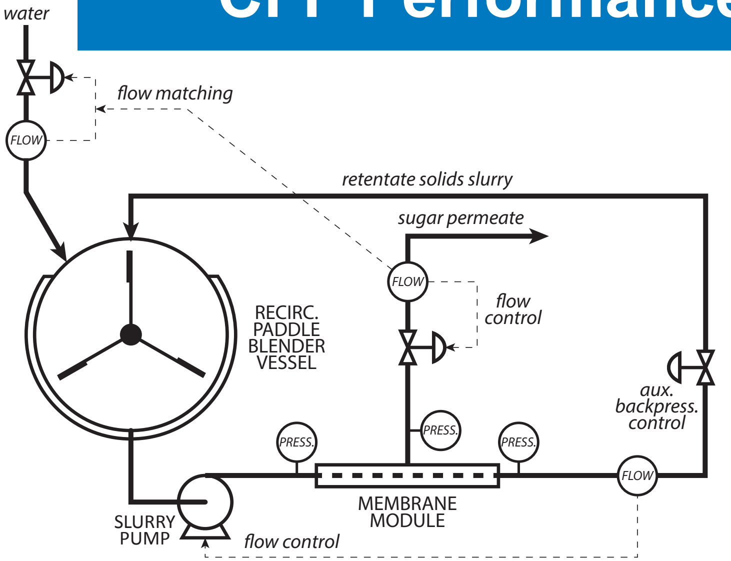
Limited risk with remaining project work

- Equipment failure (decanter) – Old equipment
- Resort to bench-scale testing using comparable g force-time criteria



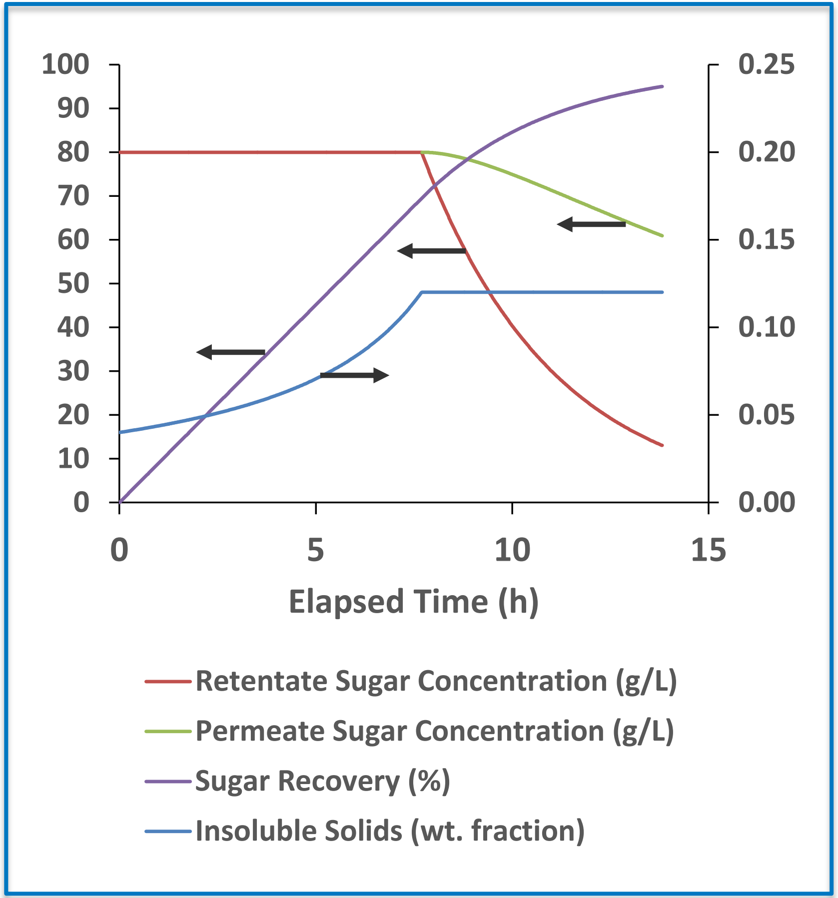
Adapted from Perry's Chemical Engineers' Handbook (1997).

CFF Performance Model Validated at Pilot Scale



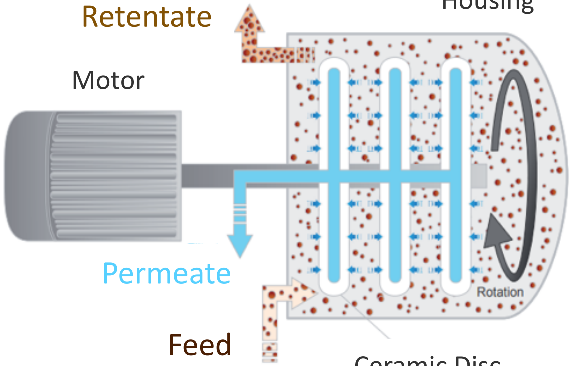
Cross Flow Filtration (CFF) Schematic with Diafiltration

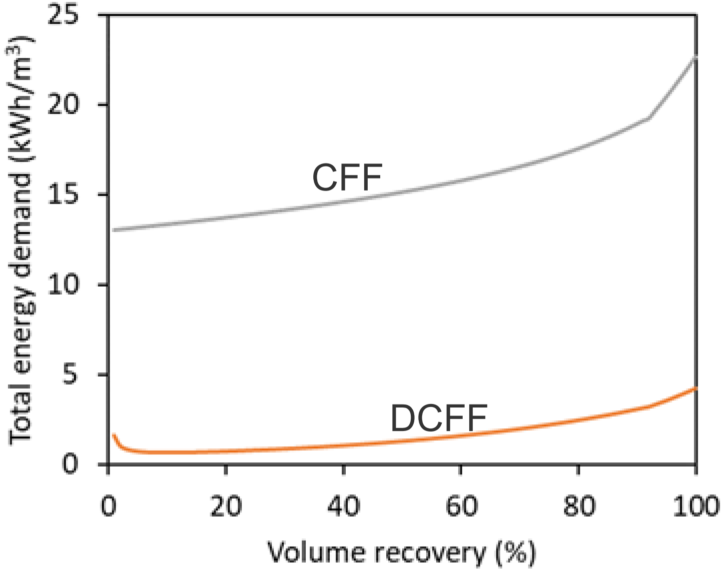
Pilot CFF Unit

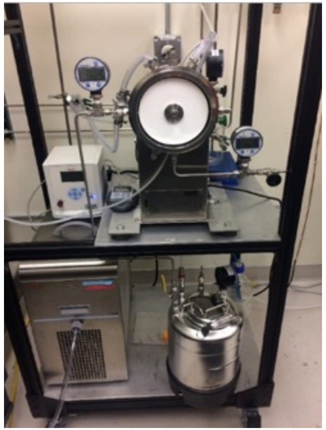


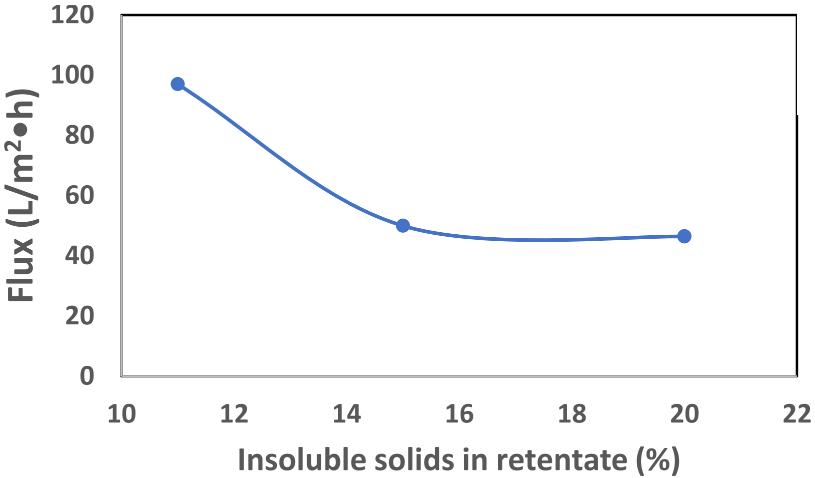
Improved DCFF Performance Data and Modeling Results

Dynamic Cross Flow Filtration (DCFF)



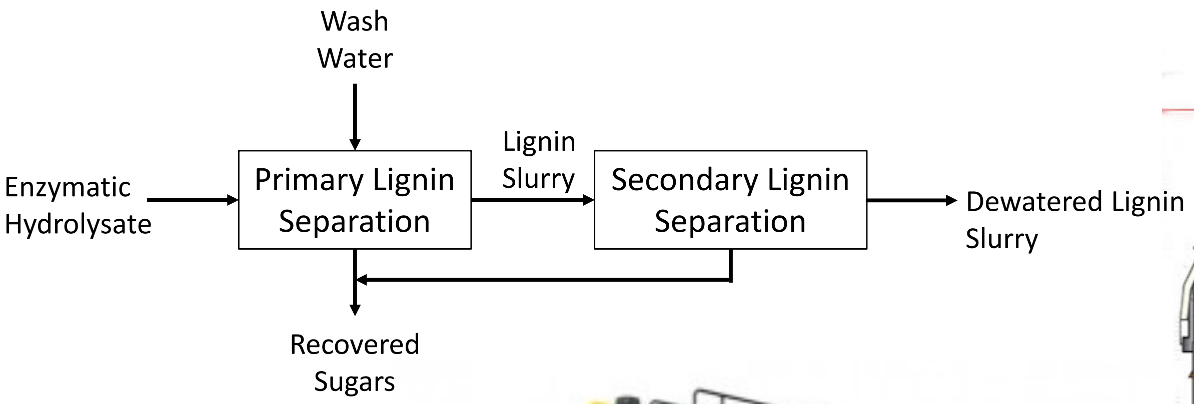


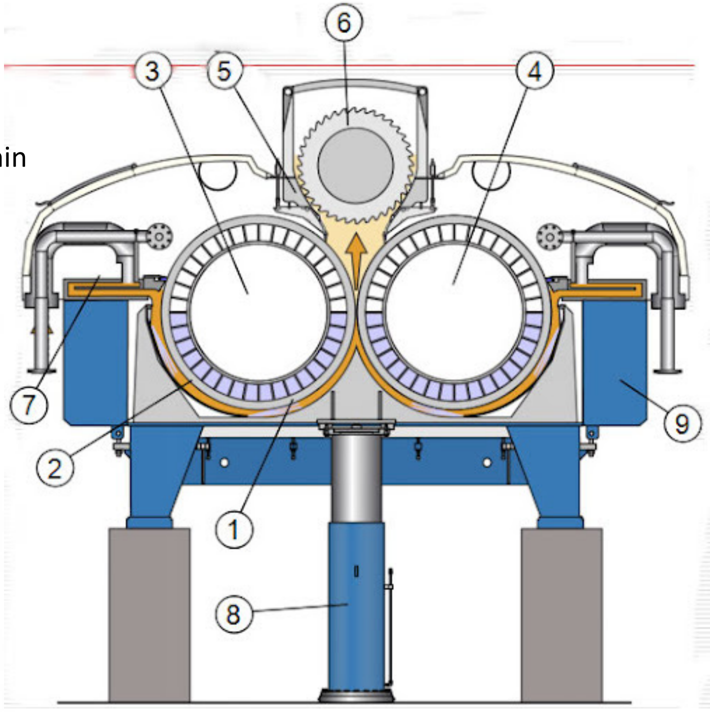




$$Volume\ recovery = \frac{Permeate\ Volume}{Total\ Feed\ Volume}$$

Expression Processes Not Workable





Conclusion:
Expression is not a viable option due to need for filter media.

https://www.valmet.com/pulp/mechanical-pulping/washing/twinroll-press/

Lower Flocculant Cost Possible

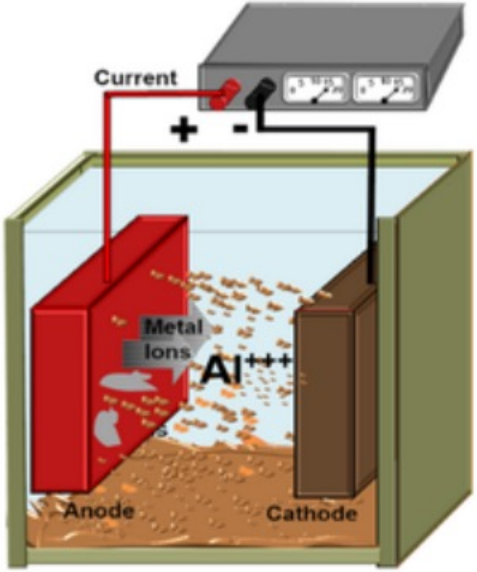
Flocculant Source	Compound	Dosage Data		Feed Type	IS Content	Cost	Estimated Cost
		mg/L	g/kg IS		(mg/L)	($/kg)	($/GGE)
KemSep C-7105	Cationic polyacrylamide	800	20	DMR corn stover EH	40,000	1.32	0.12
KemSep C-7107	Cationic polyacrylamide	718	10	DDA corn stover EH	67,000		
KemSep C-7107	Cationic polyacrylamide	421	8	DA corn stover EH	50,000		
KemSep C-1592	Cationic polyacrylamide	100	1.7	Pine wood EH	50,000		
Organopol 5415	Cationic polyacrylamide	5	1	Pulp/paper mill wastewater	5,240		
Self-synthesized	Polydiallyldimethylammonium chloride (polyDADMAC)	1.2	0.2	Pulp/paper mill wastewater	6,000	2.00	0.18
Self-synthesized	Chitosan	7	1.1	Cardboard wastewater	6,500	3.00	0.27
Novillars	Poly-aluminium chloride	0.4	0.5	Cardboard wastewater	6,500	0.40	0.04
Self-synthesized	Fenugreek mucilage (solid powder)	0.08		Tannery waste	350		
Self-synthesized	Polyacrylamide-oatmeal (graphed)	1.25	8.3	Sewage wastewater	150		
Self-synthesized	Polyacrylamide-agar (graphed)	0.75	3.4	Sewage wastewater	222		
Self-synthesized	Chitosan-based			n/a			
Self-synthesized	Xylan-trimethyl ammonium chloride (DMC)	500	1400	Pulp mill wastewater	356		

The cost for a KemSep C-7105 was half of the cost assumed in the previous TEA, but vendor noted price is extremely variable.

IS-Insoluble Solids

Electrocoagulation (EC) Test Results

Performed by Avivid Water Technologies



https://www.avividwater.com/

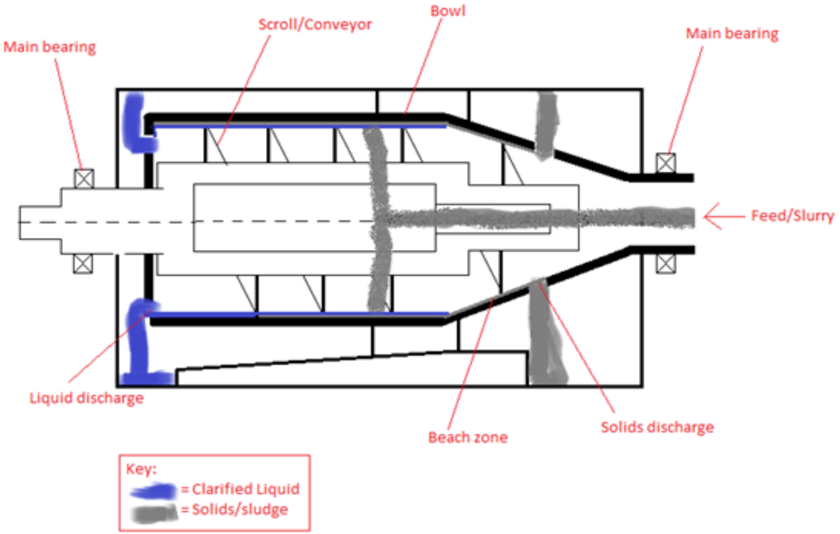
EC-Treated DMR-Derived Enzymatic Hydrolysate

Centrifugation of EC Treated Material

- Coagulation successful and likely achieves the same performance as flocculants
- TEA still to be done

Next Steps

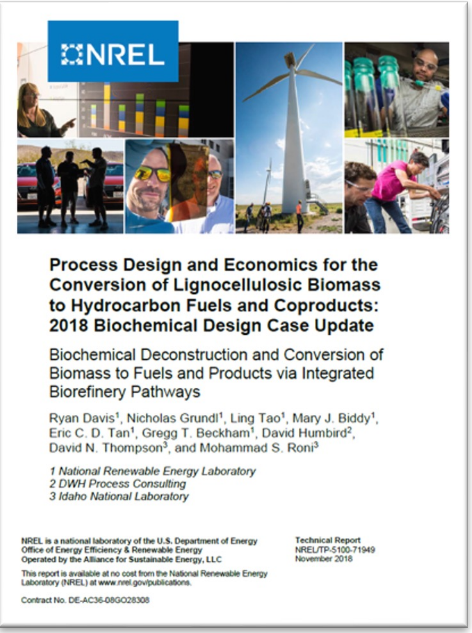
- Completed pilot scale evaluation of decantation
- Validate decantation washing performance/models
- Finalize TEA for non-flocculated processes
- Generate recommendations



Pilot Plant Decanter

How TEA Modeling Is Being Done

2018 TEA Model



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Process Design and Economics for the Conversion of Lignocellulosic Biomass to Hydrocarbon Fuels and Coproducts: 2018 Biochemical Design Case Update

Biochemical Deconstruction and Conversion of Biomass to Fuels and Products via Integrated Biorefinery Pathways

Ryan Davis[1], Nicholas Grundl[1], Ling Tao[1], Mary J. Biddy[1], Eric C. D. Tan[1], Gregg T. Beckham[1], David Humbird[2], David N. Thompson[3], and Mohammad S. Roni[3]

1 National Renewable Energy Laboratory
2 DWH Process Consulting
3 Idaho National Laboratory

NREL is a national laboratory of the U.S. Department of Energy
Office of Energy Efficiency & Renewable Energy
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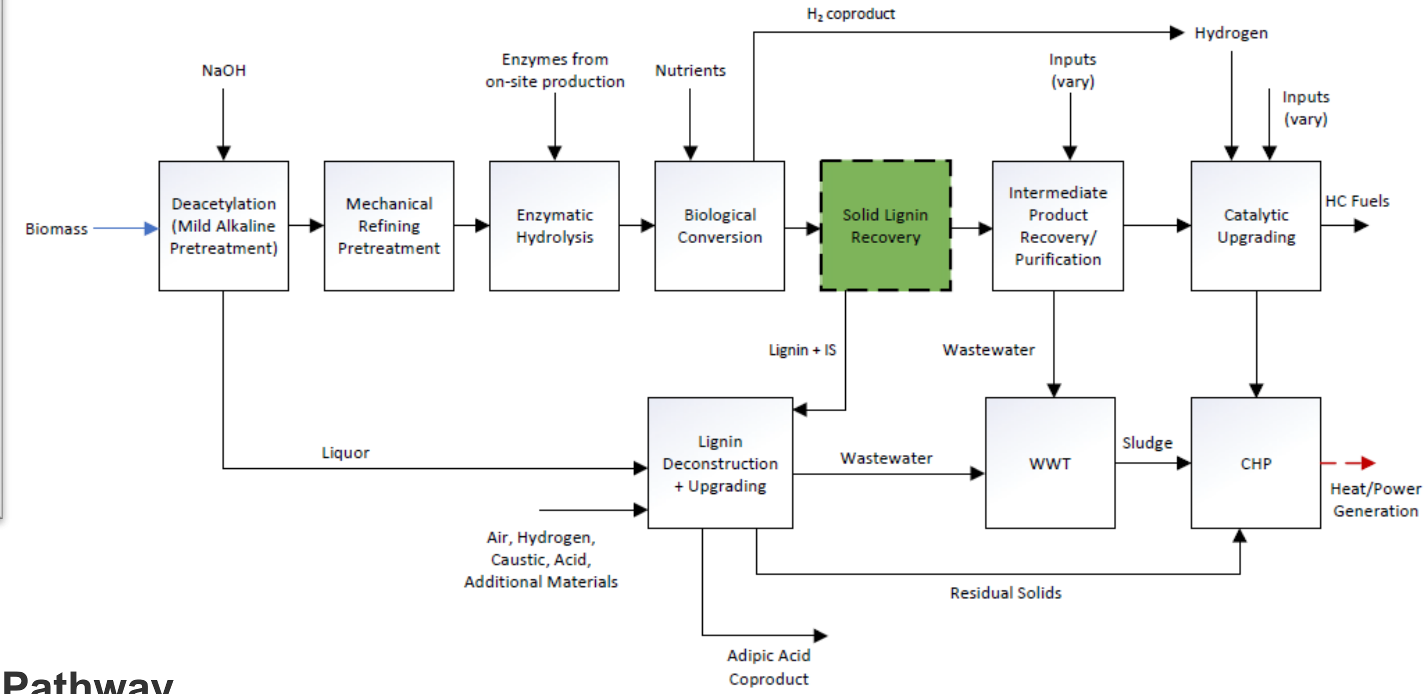
Contract No. DE-AC36-08GO28308

Technical Report
NREL/TP-5100-71949
November 2018

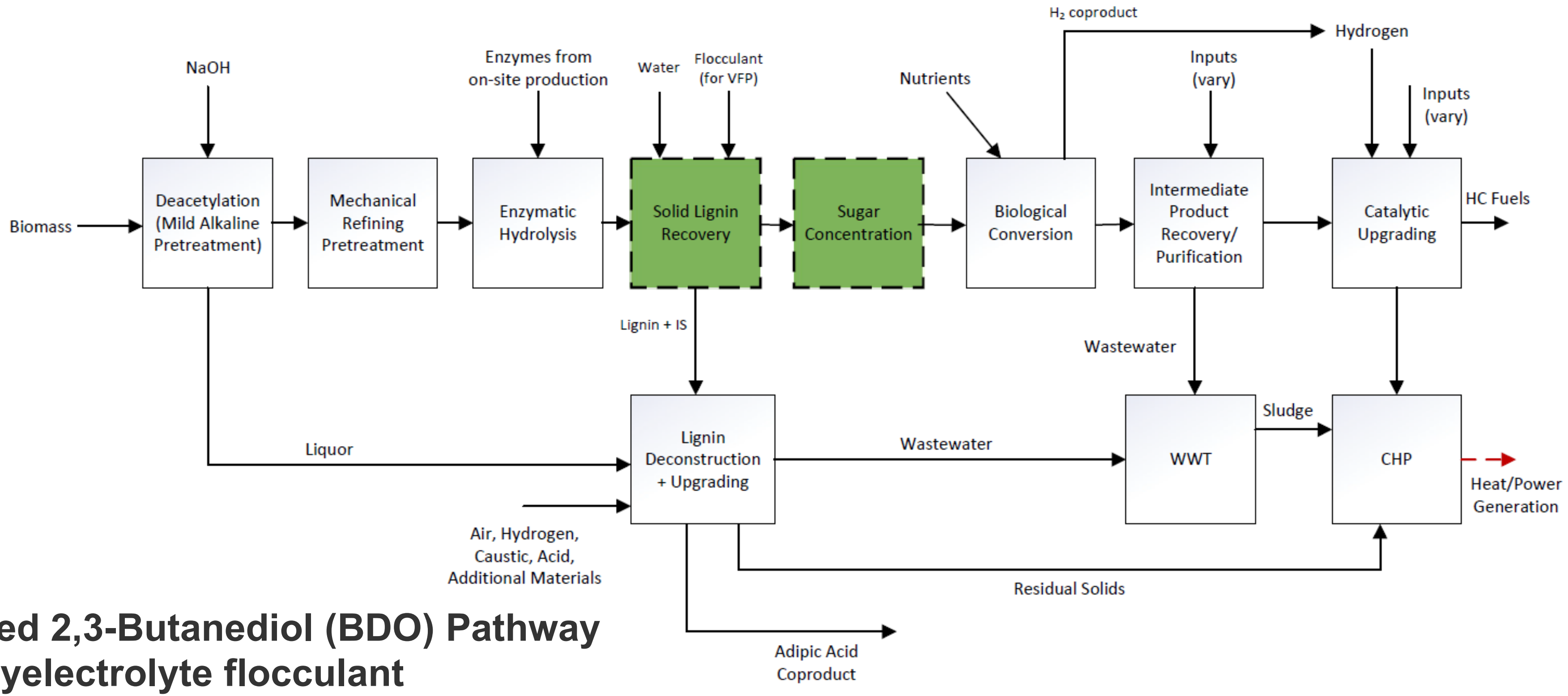


2,3-Butanediol (BDO) Pathway

- **No flocculation**
- **Lignin separation after bioconversion**

New TEA Model for This Work

Modified TEA Model Incorporating Flocculation - Baseline



Updated 2,3-Butanediol (BDO) Pathway

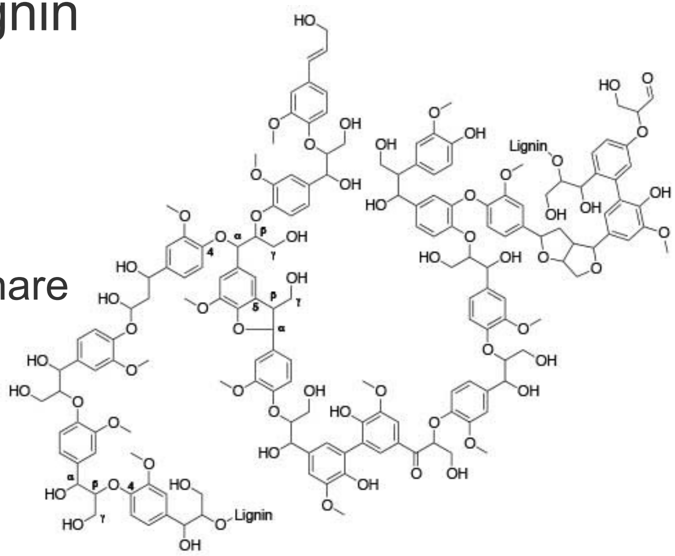
- **Polyelectrolyte flocculant**
- **Lignin separation before bioconversion**
- **Feb-batch conversion process**

Impact

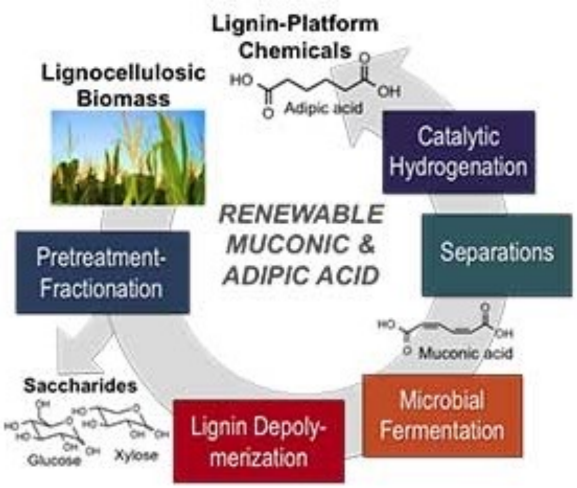
Effective solid lignin recovery and subsequent valorization lowers biofuels cost and enabling multiple biomass to biofuel conversion processes currently be explored by BETO and industry.

This work strives for a better understanding of the performance and cost drivers for solid lignin separation processes.

- Already identified a more cost-effective separation process compared to the baseline model
- Generate new separation ideas or suggestions to share with equipment manufacturers
- Provide guidance to bioeconomy/biofuels industry
- Work will be disseminated via publications



Summary



Approach:

- Initial evaluation of non-flocculated separations led to the decision to further investigate these processes
- New work continues to evaluate economic performance of various separation processes with pilot scale data, if possible

Accomplishments:

- Pilot scale testing completed for cross flow filtration including washing performance results
- More testing completed on dynamic cross flow filtration at higher insoluble solids loadings and electricity use modeling
- Assessment of expression, alternative flocculants and electrocoagulation completed, but no outcome changing goals

Impact:

- Solid lignin separation processes identified that lowered the cost of SAF compared to the baseline process
- Provides foundation for more process development and optimization if needed

Acknowledgments

Team Members

- Mike Baker
- Ryan Davis
- Jacob Dempsey
- Matt Fowler
- Casey Gunther
- Wes Hjelm
- Luke Klin
- Bob Lyons
- Ian McNamera
- Patrick Saboe
- Dave Sievers

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- US Department of Energy Office of Energy Efficiency and Renewable Energy Bioenergy Technologies Office
- Josh Messner – BETO Technology Manager
- Jim Spaeth – SDI Program Manager

Questions?

www.nrel.gov

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Quad Chart Overview

Timeline

- Project start date: Oct. 1, 2019
- Project end date: Sept. 30, 2023

	FY22 Costed	Total Award
DOE Funding	$51,100	$600,000
Project Cost Share		

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

Project Goal

Valorizing lignin residue remaining after enzymatic hydrolysis of pretreated biomass is necessary to achieve BETO's $2.50/GGE biofuel cost target for a biochemical pathway. But no clear options exist for recovery of washed/dewatered lignin residue for valorization—particularly for a DMR-process-derived lignin. The goal of this work is to develop an effective washing and recovery strategy for DMR-derived solid lignin that is more cost effective than current state-of-the-art.

End of Project Milestone

Generate pilot scale data for TEA still meeting year one economic results while retiring previously assumed performance assumptions.

Funding Mechanism - FY19 BETO Seed Project Lab Call

Project Partners

- None

Additional Slides

Responses to Previous Reviewers' Comments

Reviewer Comments: Although the DMR process is used internally and the team members shared their experience with the excellent fermentability of the substrate, it is unclear if the DMR process is supported by industry and will be scaled up. Immense funding has been given specifically to the DMR process, but none of that funding appears to be going toward promoting this process within industry.

- **Response:** Industry is becoming interested in this technology as within the last year, two industry cost-shared projects further developing this technology are now active. Also, while the focus has been on DMR-derived biomass, the results should generally be applicable to any aqueous-phase pretreatment process; however, we have found that DMR-derived lignin is the most difficult to separate, and for this reason, this material was used in this work.

Reviewer Comment: The approach (slide 8) shows the desire for investigating more non-flocculating options, but the TEA shows only a flocculating path. It is unclear if the TEA was developed within this group or by another team. The concern is that the team may make the TEA lean toward the direction that they prefer to investigate. It is unclear what the price point of lignin or its application in the TEA is based on. The baseline model really should be burning the lignin or perhaps discarding the lignin without any additional processing. In this way, the value of any additional processing is made clear.

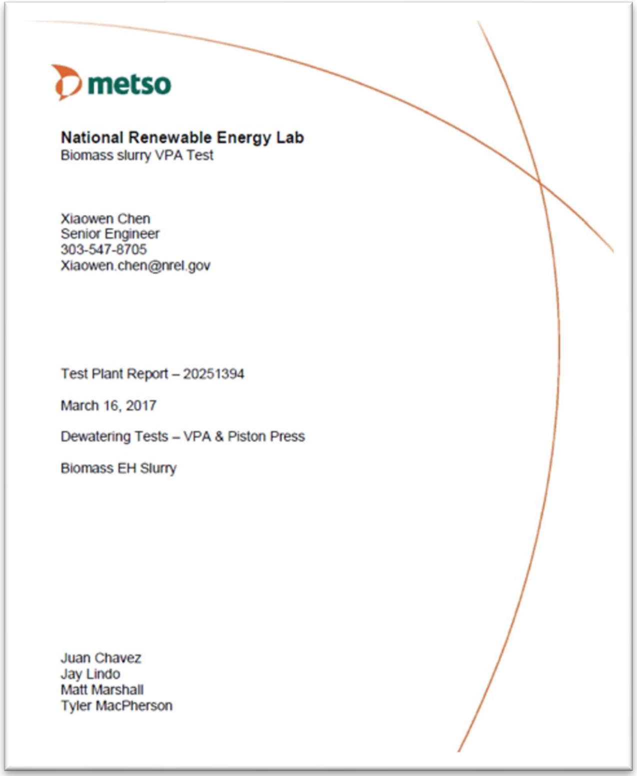
- **Response:** TEA was performed by the NREL process analysis team using previously established models. These models include DMR and lignin utilization process design and economics as documented in NREL's 2018 design report (https://www.nrel.gov/docs/fy19osti/71949.pdf), which has been reviewed and vetted by industry and other external reviewers. The analysis here already established that lignin conversion to biofuels is more cost effective than burning lignin.

Publications, Patents, Presentations, Awards, and Commercialization

- No output yet

2. Approach

Technology History/Background – Vendor Testing


metso

National Renewable Energy Lab
Biomass slurry VPA Test

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Test Plant Report – 20251394

March 16, 2017

Dewatering Tests – VPA & Piston Press

Biomass EH Slurry

Juan Chavez
Jay Lindo
Matt Marshall
Tyler MacPherson

Major Findings (Report Quotes):

- "During the VPA (pressure filtration) test a very thin filter cake formed on both sides of the chamber. **At this time, filtrate was unable to escape through the filter cloth.** This was under low filter pressures, 3-4 bar. **Filtrate was very brown and cloudy."**

- "During the Piston Press Test, a very thin filter cake formed against the filter cloth. **At this point, filtrate ceased to penetrate through the filter cake.** The remainder of the feed slurry was stuck in the chamber. This occurred both under high pressures, 100 bar, and low pressure, 10 bar. **Filtrate was very brown and cloudy."**