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

U.S. Department of Energy (DOE) Bioenergy Technologies Office (BETO) 2017 Project Peer Review

BIOPROCESSING SEPARATIONS CONSORTIUM

March 6, 2017

Biochemical Conversion

Jennifer Dunn

Consortium Steering Committee Lead

Argonne National Laboratory

Goal Statement

Goal: Move cost-effective, high-performing separations technology to market faster through coordinated separations research at the national laboratories that targets challenges relevant to BETO's priority pathways and industry.

Outcome: Biofuels and bioproducts industries will have new, high-performing, low-cost separations technologies available to them.

Relevance to Industry: BETO industrial stakeholders have long raised separations challenges as a major barrier to cost competitive biofuels and bioproducts. BETO analyses indicate that separations steps can constitute up to 50% of processing costs.

Quad Chart Overview

Timeline

- Project start date: 10/1/2016
- Project end date: 9/30/2019
- Percent complete: 8%

Barriers

- Ct-G: Efficient Intermediate Clean Up and Conditioning
- Ct-I: Product finishing, acceptability, and performance.
- Ct-J: Process integration

Budget ($thousand)

<table>
<tr><th>Costs</th><th>FY 16</th><th>FY 17*</th><th>Total Planned Funding
(FY 17- End)</th></tr>
<tr><td>Consortium (DOE Funded)</td><td>$500</td><td>$3,000/
$2,500</td><td>$9,000/$7,500</td></tr>
<tr><td>Biochemical Separations</td><td>0</td><td>$800/$885</td><td rowspan="7">Funding for individual teams dependent upon shifts in project portfolio based on impact of R&D on process economics and achievement of technical targets.

*Black text indicates originally planned funding levels. Red text indicates revised funding levels to account for budget uncertainty.</td></tr>
<tr><td>Thermochemical Separations</td><td>0</td><td>$800</td></tr>
<tr><td>Ionic Liquid Separations</td><td>0</td><td>$400/$0</td></tr>
<tr><td>Algal Separations: Seed</td><td>0</td><td>$200</td></tr>
<tr><td>Biochemical Catalyst Preservation: Seed</td><td>0</td><td>$200</td></tr>
<tr><td>Analysis</td><td>0</td><td>$400/$315</td></tr>
<tr><td>Steering Committee</td><td>0</td><td>$200/$100</td></tr>
</table>

Partners

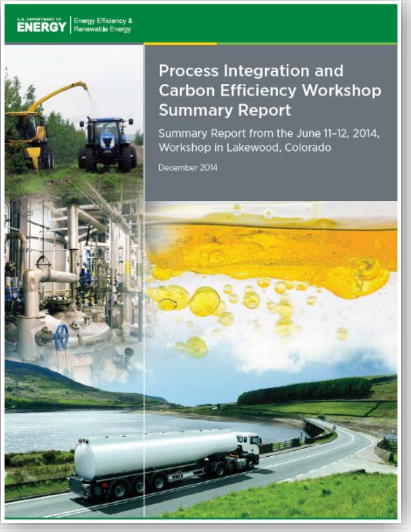
- ANL
- INL
- LANL
- LBL
- ORNL
- NREL
- PNNL
- Sandia

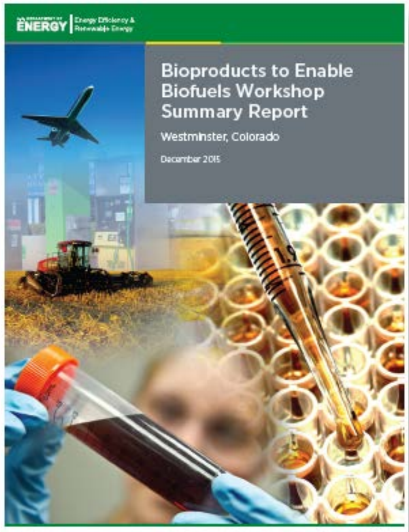
1. Project Overview

Industrial stakeholder input to BETO[1,2] has indicated that separations challenges impede the cost-competitive production of biofuels and bioproducts. Stakeholders identified upstream and downstream separations and purification challenges as near term barriers that could benefit from immediate attention. For example:

- Separations steps are a barrier to efficient unit operation integration
- Improved solid-liquid and liquid-liquid separations technologies are needed for bioprocessing
- Residual solids influence downstream processes and need to be removed
- Membrane fouling and poorly-understood material properties limit their effective application in bioprocessing

The Consortium addresses this stakeholder feedback, bringing together expertise and capabilities across eight national laboratories to develop new separations technologies and apply existing technologies to new bioprocessing challenges.

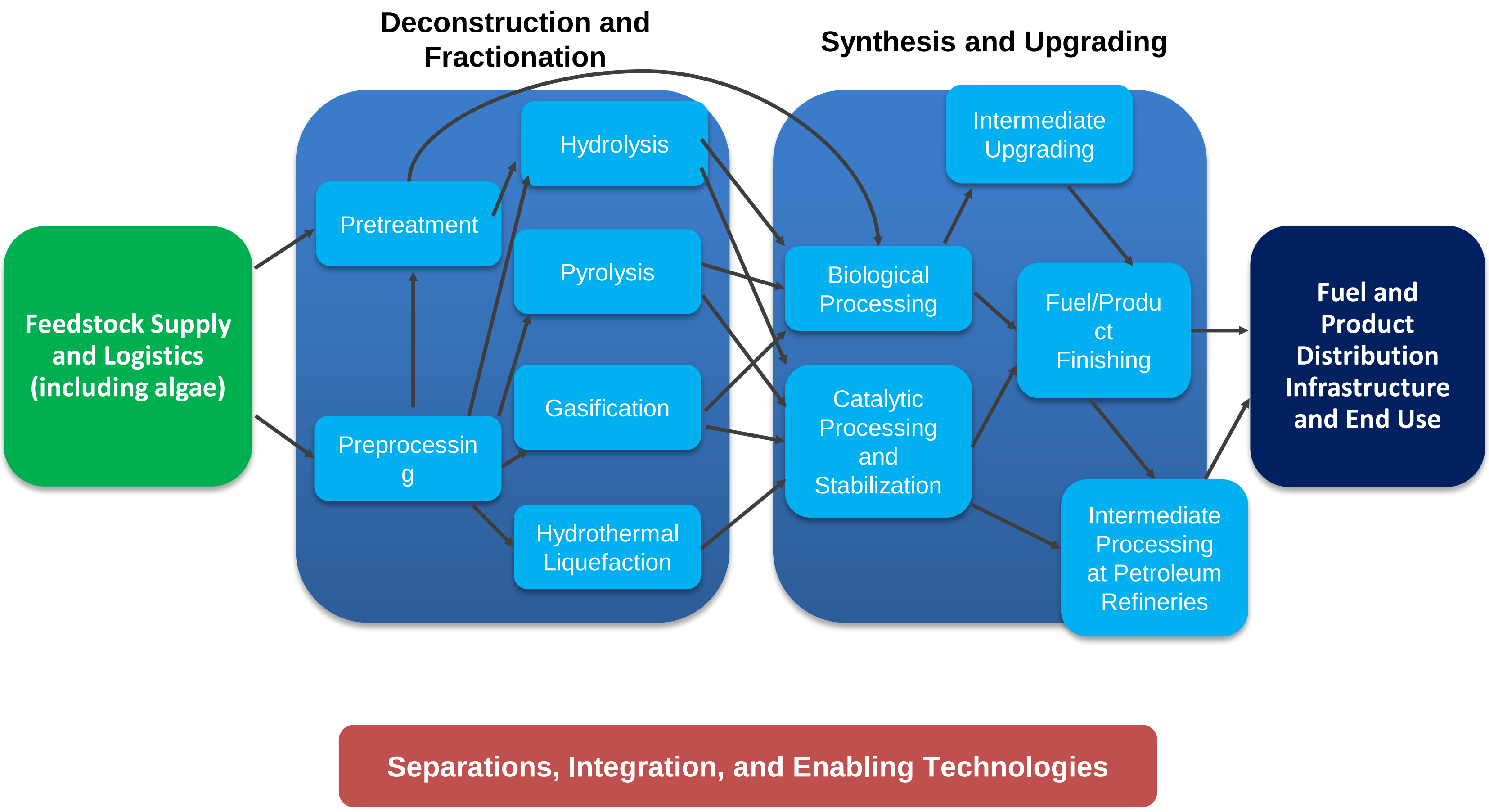




1. EERE. 2015. Bioproducts to Enable Biofuels Workshop Summary Report. Workshop held in Westminster, Colorado.
2. EERE. 2014. Process Integration and Carbon Efficiency Workshop Summary Report. Workshop held in Lakewood, Colorado.

1. Project Overview

Separations are crucial to all biomass conversion pathways



1 - Project Overview: Consortium Formation

- Teams surveyed separations challenges in BETO priority pathways
- 54 separation challenges identified from seven separate pathways across the BETO algae and conversion platforms and formed the “technology map”

Mark with a Y technologies/applications that are IN BETO portfolio		Applicable Separation Technologies (Must designate which are currently in R&D Portfolio and which are gaps, can build in links to one-pagers ab					
Mark with an N technologies/applications that are OUTSIDE BETO portfolio		Membrane		Liquid Liquid Extraction		Ultrasonic	
		Membrane Type 1 (Liquid Phase)	Membrane Type 2 (Vapor Phase)	LLE 1 (Liquid/liquid equilibrium separations, no solvent used)	LLE 2 (liquids extraction into a solvent immiscible with process liquid)	Low Frequency Sonication	High Frequency Separation
Pathway	**Pyrolysis and Upgrading (In-Stitu/Ex-Situ) Fuel Production**						
Challenges	Remove char particles from liquid or vapor	N	N	N		N	Y - temp dependent
	Separate liquid organic and aqueous phases	YY	YY	Y	Y - possible to extract some	N	Y
	Separate oxygenates from hydrocarbon in intermediate upgraded bio-oils; separation of aqueous phase becomes easier with more deoxygennation; removal residual hyrdrocarbons from aqueous phase	YY	Y	Y - separation easier with more deoxygenation		N	Y

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

1 - Project Overview: Consortium Formation

Many possible projects identified; the following questions used to select first portfolio and organize the consortium into teams.

Indicator	Notes
Does the project address a technical challenge facing one of the priority pathways?	• Can check technology map or with the author(s) of the relevant design reports
Does the challenge have a large impact on process economics for the relevant pathway(s)? (e.g., potential to reduce modeled costs)	• Can check technology map or with the author(s) of the relevant design reports
Is this project similar to or a continuation of a project within BETO's portfolio?	• Check technology map and with the steering committee
Is there any indication that this project has been addressed by external work?	• Brief literature/patent search
Do the labs have existing capital equipment and personnel with the necessary technical knowledge to carry out the project?	• Check with team members/LRM/steering committee
Potential annual cost of project (($ - <$250k, $$ - $250-$500k, $$$ - $500k-$1MM, $$$$ - > $1MM)	• Estimate based on staff needs, operating costs, etc. No major capital costs should be expected
What other projects would benefit if this project were successful?	• For example, maybe a catalysis project will no longer be impacted by a certain inhibitor or perhaps a fermentation will have a higher yield because an inhibitor will no longer be present • Synergistic projects list is here.
Who (specifically) are potential industrial and academic partners	

2- Approach (Management)

Bioenergy Technologies Office:
Nichole Fitzgerald

Crosscutting Analysis:
Mary Biddy (NREL)
Sue Jones (PNNL)
Jennifer Dunn (Argonne)

Steering Committee:
Jennifer Dunn (Argonne)
Taraka Dale (LANL)
Todd Pray (LBL)

Advisory Board

Teams

Biochemical:
Gregg Beckham (NREL)
YuPo Lin (Argonne)

Thermochemical:
Jae-Soon Choi (ORNL)
Bob Baldwin (NREL)

Ionic Liquid:
Ning Sun (LBL)
Aaron Wilson (INL)
Seema Singh (Sandia)

Algae (Seed):
Jim Coons (LANL)
Brian Bischoff (ORNL)

Biocatalyst Preservation (Seed):
Phil Laible (ANL)

Investigator Roster

Biochemical Separations:
Gregg Beckham (NREL)
David Brandner (NREL)
Jim Coons (LANL)
Robin Cywar (NREL)
Eric Karp (NREL)
YuPo Lin (ANL)
Lorenz Manker (NREL)
Michelle Reed (NREL)
Patrick Saboe (NREL)

Thermochemical Separations:
Robert Baldwin (NREL)
Justin Billing (PNNL)
Jae-Soon Choi (ORNL)
Chai Engkatrul (NREL)
Michael Hu (ORNL)
Mark Jarvis (NREL)
Sue Jones (PNNL)
YuPo Lin (ANL)
Aimee Lu (ORNL)
Kim Magrini (NREL)
Asange Padmaperuma (PNNL)
Andrew Schmidt (PNNL)
Tim Theiss (ORNL)
Huamin Wang (PNNL)

Algae Processing Separations:
Brian Bischoff (ORNL)
Jim Coons (LANL)
Tao Dong (NREL)
Phil Pienkos (NREL)
Nicholas Sweeney (NREL)
Ben Yap (LANL)

Biochemical Catalyst Preservation:
Seth Darling (ANL)
Deborah Hanson (ANL)
Patricia Ignacio de Leon (ANL)
Phil Laible (ANL)

Cross-Cutting Analysis:
Mary Biddy (NREL)
Jennifer Dunn (ANL)
Sue Jones (PNNL)

Ionic Liquid Separations:
Seema Singh (SNL)
Ning Sun (LBL)
Aaron Wilson (INL)

Steering Committee:
Taraka Dale (LANL)
Jennifer Dunn (ANL)
Todd Pray (LBL)

2- Approach (Management)

Consortium Management

- Monthly full-consortium calls to update on business and technical accomplishments; each team has regularly scheduled calls
- **Cross-team interactions** leverage results and information
- **Cross-cutting analysis team** engages with each R&D team to inform technical targets, economic impact of projects
- Stream stewards produce commonly-used, **exemplary streams** for separations experiments within different teams
- Internal sharepoint for file management
- **External website** established to disseminate information about the consortium and provide stakeholders information about current project portfolio and how to interact with the consortium.

http://bioesep.org/

2- Approach (management)

- Industrial Advisory Board Interactions
  - Held first face-to-face with IAB on December 1, 2016
  - Preparing document to address comments from IAB at face-to-face
  - IAB interactions will be quarterly, two face-to-face meetings per year

ALFA LAVAL
AJINOMOTO
arpa·e Advanced Research Projects Agency • ENERGY
AMYRIS
DUPONT®
novasep
VIRENT®

- Industrial Advisory Board Charter
  - Help the consortium maintain an industry-relevant focus and knowledge of recent technology advances and challenges.
  - Provide advice, review results and progress in comparison with work plans, provide feedback regarding prioritization of research projects (experimental and analytical), and inform development of the consortium's strategy for out years.

Cross Cutting Analysis Team

Analysis Team plays a critical role in the consortium

- **Completed Assessment of BETO Priority Conversion Routes**
  - Identified separations challenges with potential to reduce biofuel production costs
  - Quantified $/gge that these separation challenges are posing
- **Analysis Team Consortium Goals**
  - **Work closely with each technical team**
    - Estimate potential cost reductions for proposed projects
    - Enable teams to generate appropriate data for TEA analyses
    - Help set technical targets and direct research towards greatest impact
    - Track technical progress towards targets
  - **Compare proposed technologies to baseline technologies**
    - Understand potential for improving costs,
    - Assess sustainability footprint (such as fossil/water/electricity usage)
    - Evaluate reliability of proposed strategies
  - **Integrate life-cycle analysis into portfolio for projects that achieve a certain threshold (e.g., pass go/no-go)**

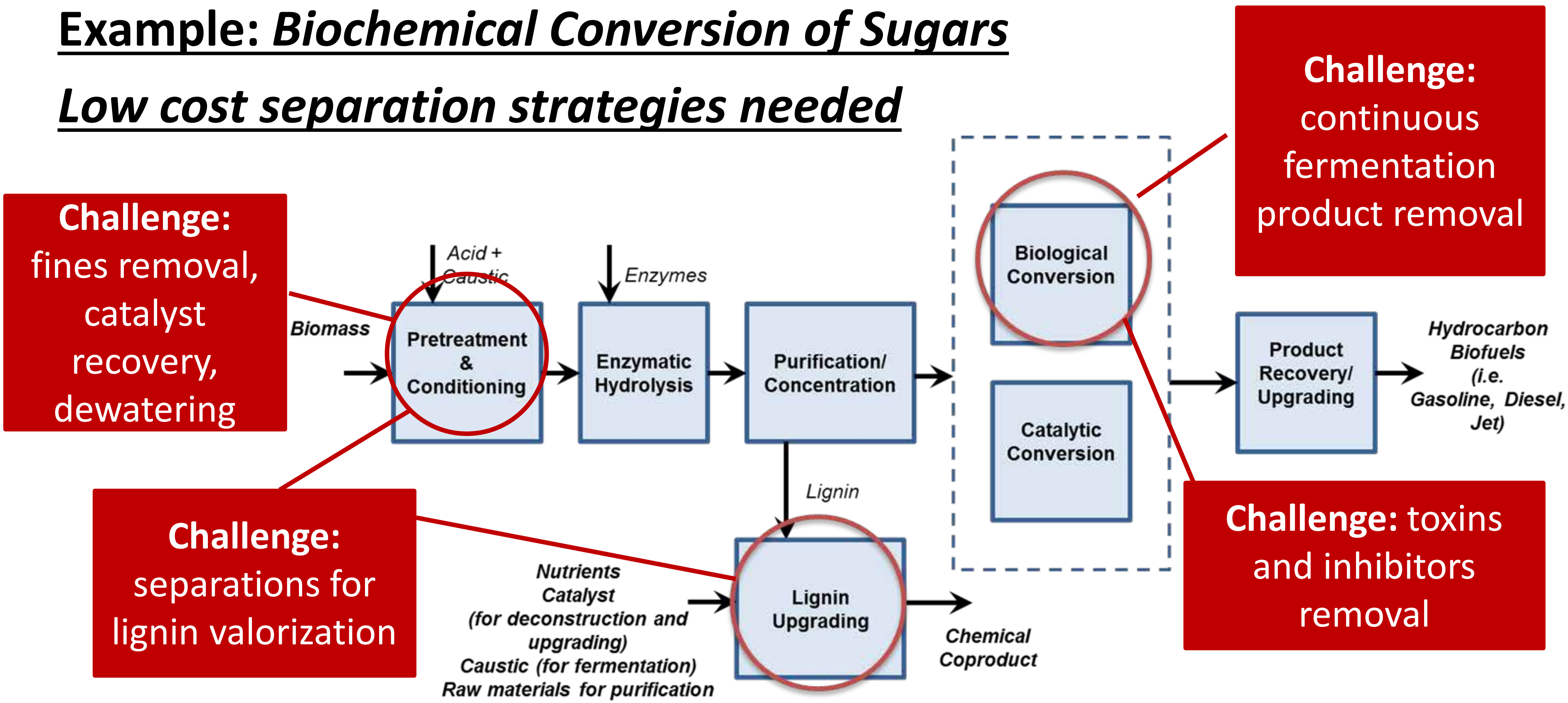
Cross Cutting Analysis Team

Assessed BETO Priority Conversion Routes:

- Fast pyrolysis and upgrading
- *Ex Situ* catalytic fast pyrolysis
- Indirect liquefaction
- Algae production
- Combined algal processing
- Hydrothermal liquefaction
- Biological conversion of sugars
- Catalytic conversion of sugars

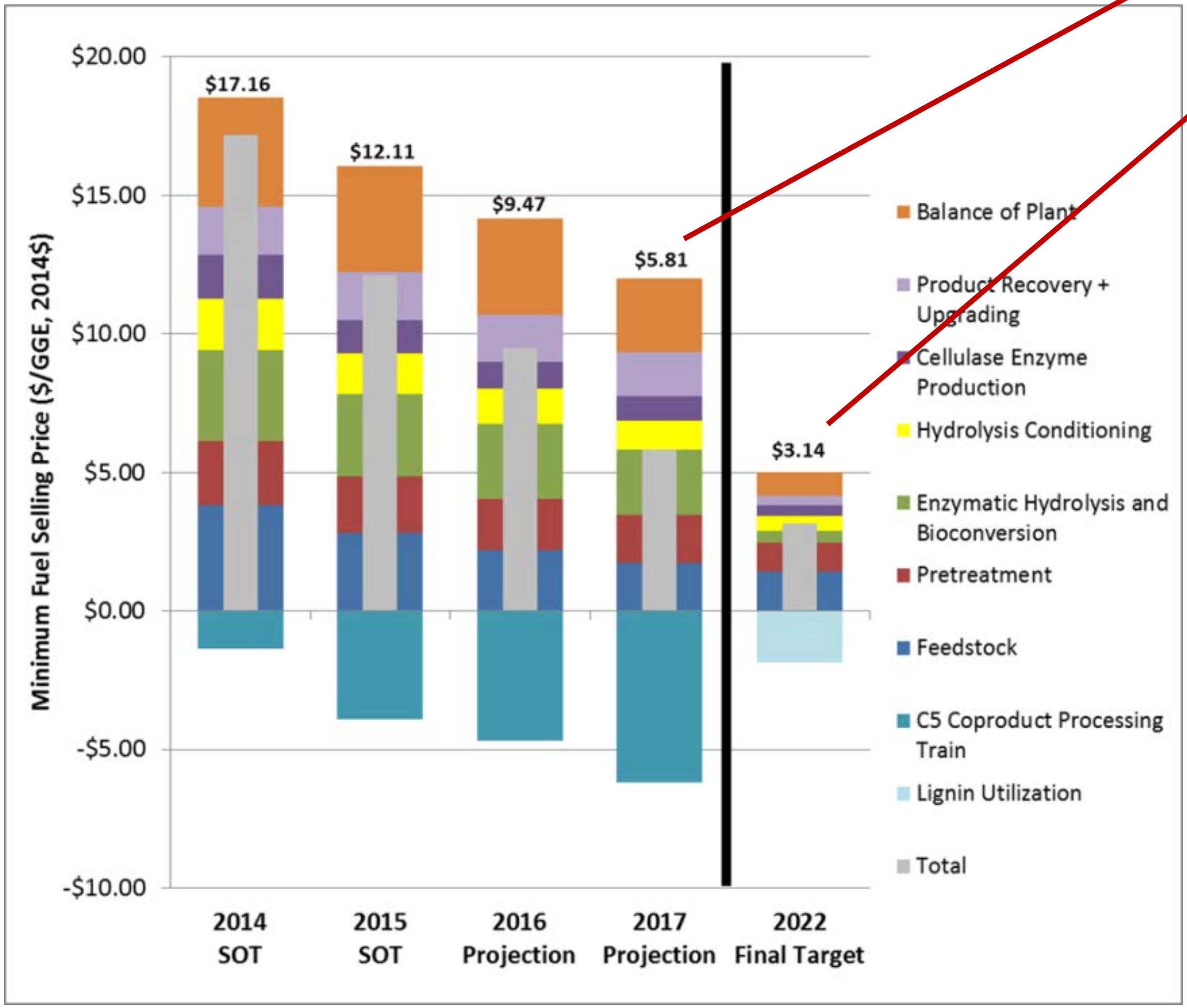
<u>Example: *Biochemical Conversion of Sugars*</u>

<u>*Low cost separation strategies needed*</u>



Cross Cutting Analysis Team

Example: Biological Conversion of Sugars Assessing Costs and Strategies



Significant Cost Reductions needed between now and 2022 – mostly related to lignin usage

R&D Strategies

– Develop on-stream data with recyclable materials to mitigate toxins and inhibitors for fermentation

– Electrochemical membrane separation technology to extract/concentrate lignin depolymerization catalysts and low molecular weight aromatic acids from alkaline pretreated liquor (APL)

3- Accomplishments

- Establishment of industrial advisory board and charter, first face-to-face meeting
- Launched website
- Early technical accomplishments within the Consortium's first quarter of work summarized by team in future work section

Photos: LBL

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

4-Relevance to Biofuels and Bioproducts Industries

- BETO stakeholders in the biofuels and bioproducts industries have flagged separtaions challenges as ripe for near- and mid-term research. The consortium addresses this input.
- The consortium is actively engaging with industry through its advisory board, seeking input regarding current and future research portfolios.
- Further biofuel and bioproduct industry input will be sought in a listending day scheduled for May 2017.

Photo: NREL

4-Relevance to BETO's Strategic Plan and the MYPP

- One strategy to enhance bioenergy's value proposition is to reduce cost and improve performance. This strategy has a substrategy of Improving Process Performance through Improved Catalysts and Separations, which states:
  - Currently, separations costs can represent up to 50% of processing costs for biofuels and bioproducts. Carbon not contained in the biofuel or bioproduct is usually used for power generation—a low-value use. It is imperative to move cost-effective, high-performing separations technologies to market faster to enable cost-competitive biofuels production. BETO will … optimize separations technologies to use biomass feedstocks efficiently.
- The Separations Consortium directly addresses this substrategy.
- The Consortium addresses the following MYPP goals and barriers
  - Separations and cleanup are overarching issues in conversion
  - Cost-effective routes to eliminating impurities in intermediates that inhibit chemical and biological catalysts
  - BETO is targeting improved separations processes to increase yields and purity including solid-gas (e.g., hot gas filtration), solid-liquid, gas-liquid, and liquid-liquid separtaions.

4-Relevance to other BETO projects

- The Separations Consortium is integrated with the overall BETO conversion platform
- Project metrics, technical targets, and newly proposed projects driven by TEA and LCA
- Through regular Conversion Team conference calls, the Consortium provides updates regarding project progress and learns about separations challenges faced in other conversion projects or ways in which consortium learnings could be applied to other projects.
- Addressing challenges associated with biochemical (e.g., enzymes, microorganisms) and thermochemical (e.g., hydrotreating) catalysts.
- Separations Consortium projects are largely designed around challenges faced in BETO’s priority pathways and are therefore directly applicable to the overall work of the Conversion platform.

Section 5: Future Work

Bioenergy Technologies Office:
Nichole Fitzgerald

Crosscutting Analysis:
Mary Biddy (NREL)
Sue Jones (PNNL)
Jennifer Dunn (Argonne)

Steering Committee:
Jennifer Dunn (Argonne)
Taraka Dale (LANL)
Todd Pray (LBL)

Advisory Board.

Teams

Biochemical:
Gregg Beckham (NREL)
YuPo Lin (Argonne)

Thermochemical:
Jae-Soon Choi (ORNL)
Bob Baldwin (NREL)

Ionic Liquid:
Ning Sun (LBL)
Aaron Wilson (INL)
Seema Singh (Sandia)

Algae (Seed):
Jim Coons (LANL)
Brian Bischoff (ORNL)

Biocatalyst Preservation (Seed):
Phil Laible (ANL)

Using TEA and LCA to support FY18 Go/No-Go decisions

On-going integration of TEA, LCA and R&D:

- **Example template** shown below
  - Outlines data needs for R&D to develop TEAs
  - At least 3 separations strategies to be assessed at the Go/No-Go

Data Used for Modeling				
Key Process Step	Brief Design Description	Based on Biomass Feedstock/Model Compounds/Other?	Experimental Scale (lab/bench/pilot /literature)	Experiment Consistent with Design (yes/no), explanation

				Key process parameters			Key results		
Mass Closure (%)	Carbon Balance (%)	Number of times experiment repeated	Time on stream (hrs)	Temp (°C)	Press (psi)	Residence Time (hr)	Efficiency(wt%)	Selectivity (wt%)	Yield (g/g)

Goal Statement – Biochem. Conversion Separations

Two aims and associated outcomes:

- Enable cost effective **separations relevant to lignin valorization**
- Enable cost effective **recovery of target products from fermentation**

Relevance to Conversion Platform, BETO, and industry:

- Lignin valorization is essential for BETO cost targets of a $3/gge MFSP – many separations challenges exist
- Recovery of intermediates from fermentation often accounts for ≥ 50% of MSP of a fuel or chemical produced via biological cultivation

Team from 4 national labs:

Argonne NATIONAL LABORATORY

BERKELEY LAB Lawrence Berkeley National Laboratory

Los Alamos NATIONAL LABORATORY EST. 1943

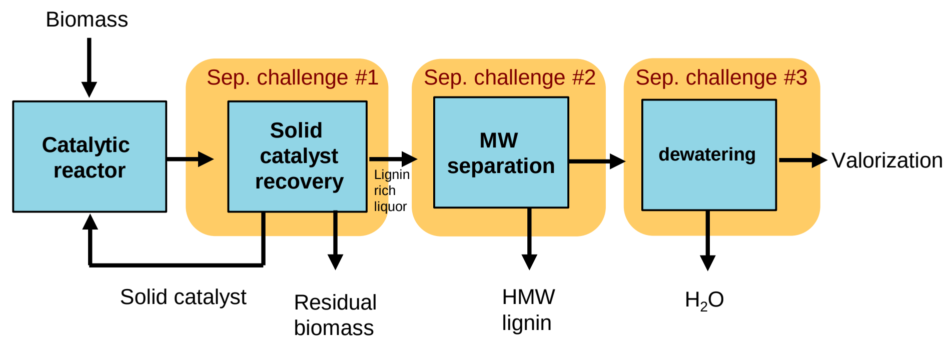
NREL NATIONAL RENEWABLE ENERGY LABORATORY

Technical Approach (and Future Work) – Lignin Separations

Approach: examine homogenous & heterogeneous catalytic lignin depolym.

- S/L separation (fines or catalysts)
- MW fractionation
- Dewatering
- Catalyst recovery

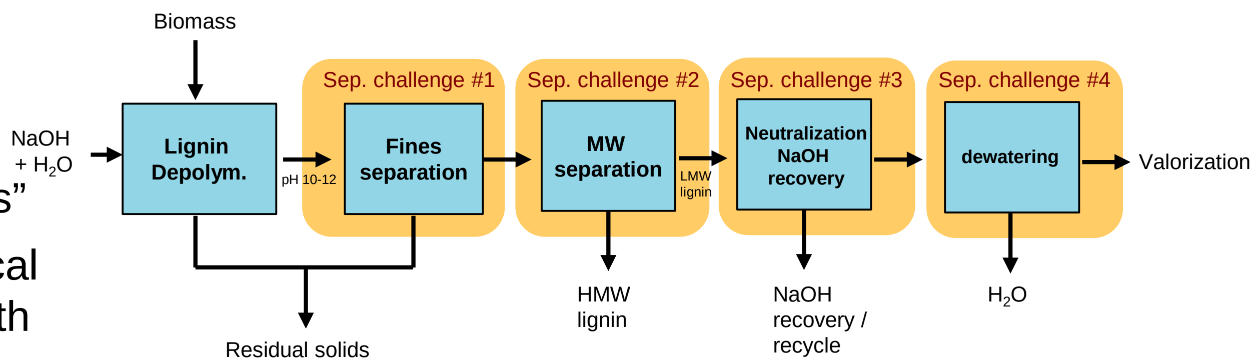
HETEROGENEOUS LIGNIN DEPOLYMERIZATION



Challenges and Critical Success Factors

- Developing <u>scalable</u> separations processes
- “Too many pots and pans”
- Catalyst recovery is critical for lignin valorization (both NaOH or solid catalyst)

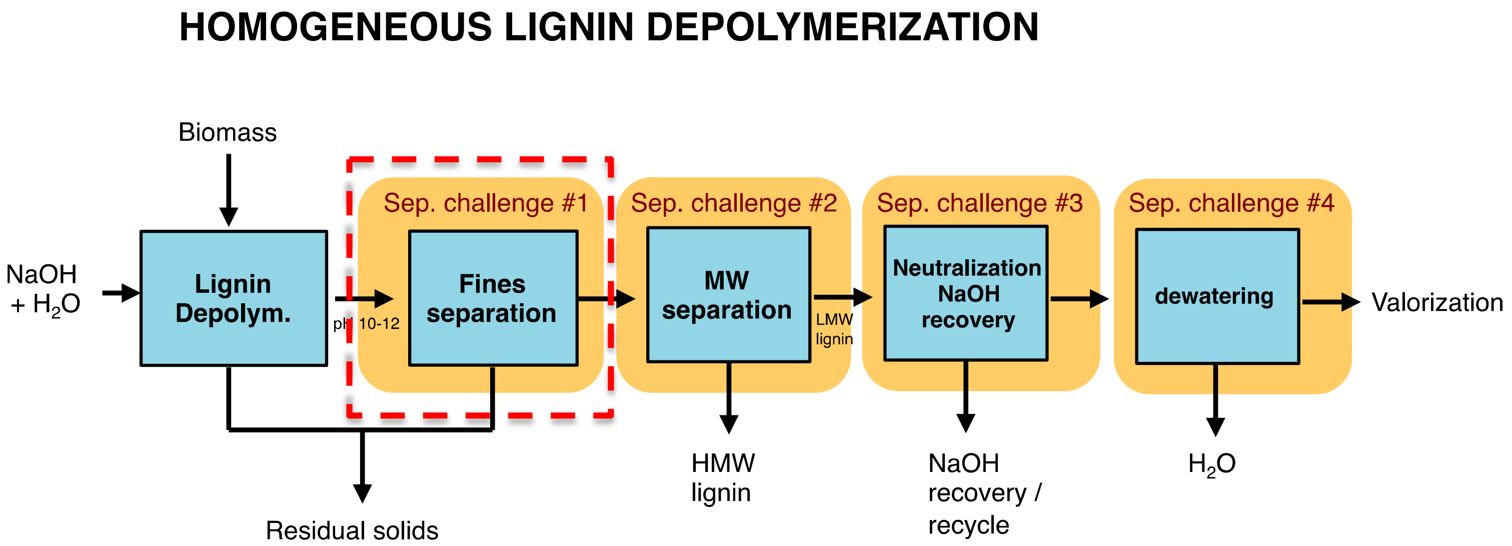
HOMOGENEOUS LIGNIN DEPOLYMERIZATION



Working towards **quantitative separations targets** based on TEA

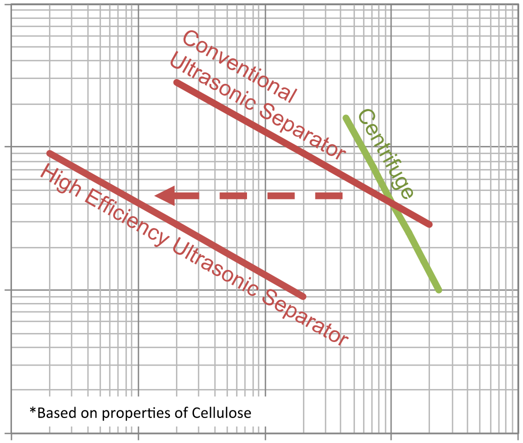
- Applying technical approaches to <u>exemplary</u> lignin streams mainly from NREL pilot plant
- Common problems for lignin valorization: results will be applicable to many lignin streams

Approach: Homogeneous depolymerization



Fines separations

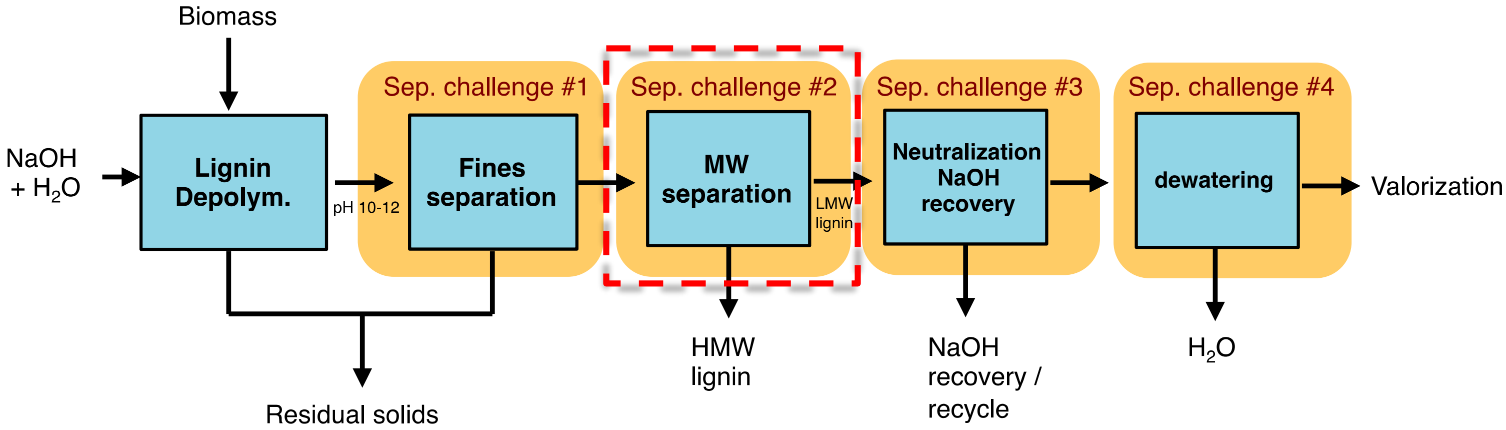
- **Alkaline pretreated liquor** from stover – a **BETO process-relevant stream** for lignin processing
- APL contains significant amount of fines from biomass processing
- **Ultrasonic separation:** can result in particle aggregation at low energy inputs



Karp *et al. ACS SusChemEng* 2014; Karp *et al. ACS SusChemEng* 2015
Karp *et al. Green Chem.* 2016; Munson, Karp *et al. ACS SusChemEng* 2016

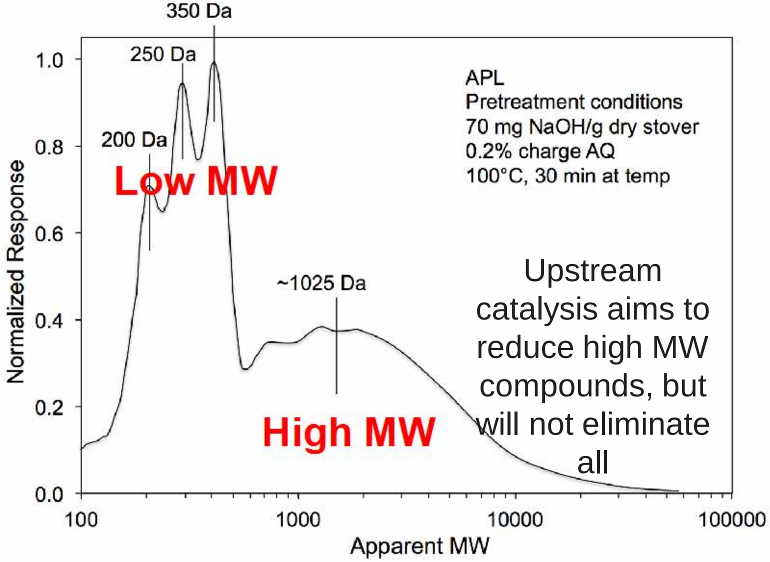
Approach: Homogeneous depolymerization

HOMOGENEOUS LIGNIN DEPOLYMERIZATION



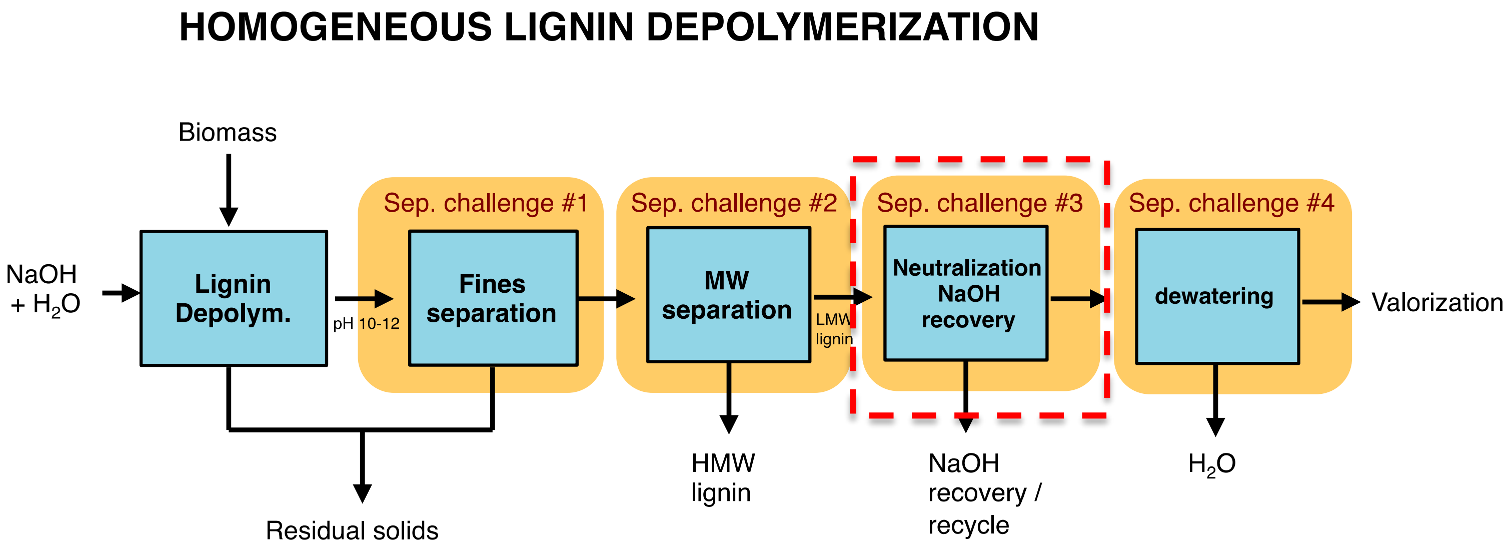
MW separations

- Membrane filtration
- Chromatographic approaches (simulated moving bed)
- Membranes with targeted MW sizes
- APL contains significant amount of high MW species from biomass processing
- **Very common, general problem** in lignin processing



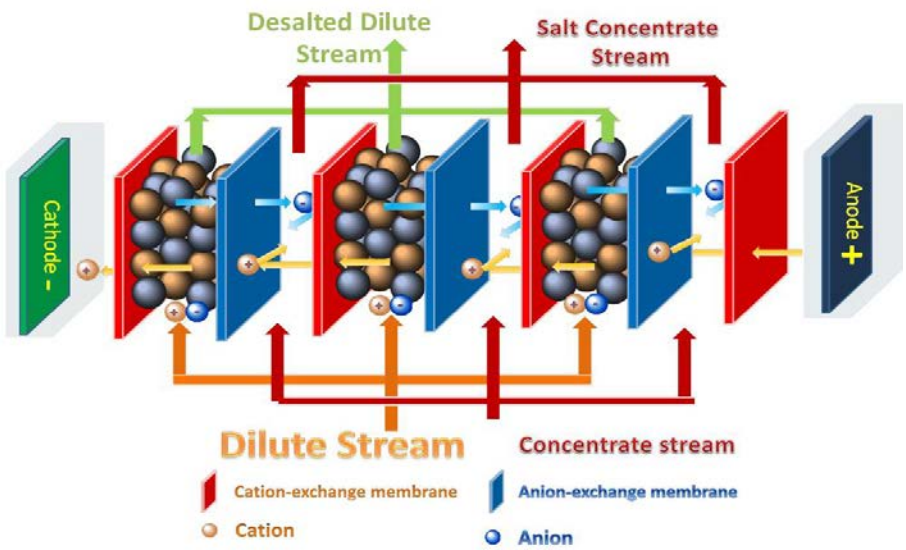
Linger, Vardon, Guarnieri, Karp *et al. PNAS* 2014

Approach: Homogeneous depolymerization



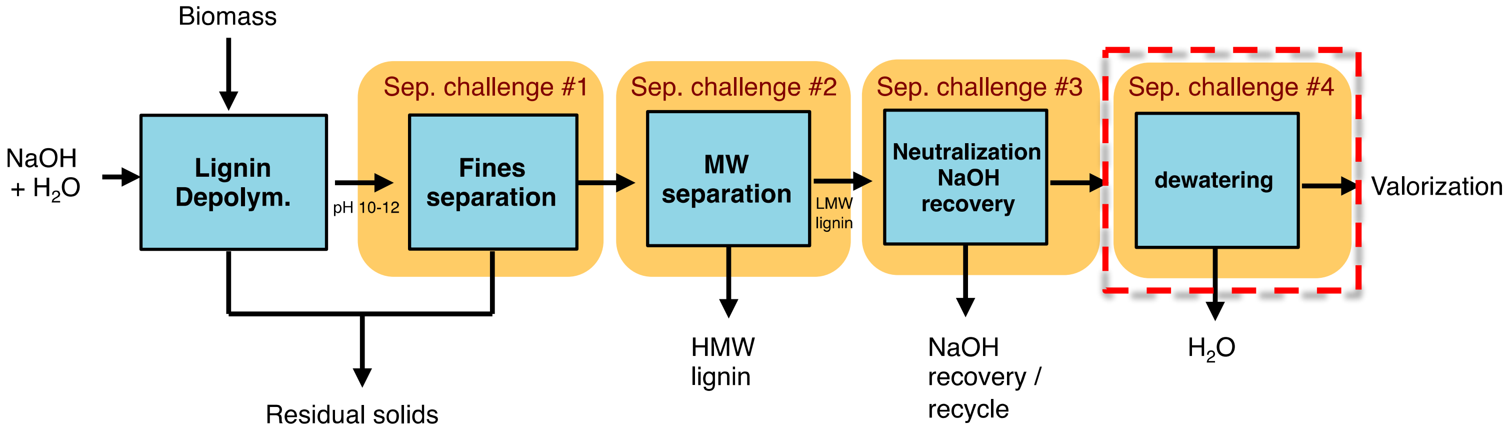
Homogeneous catalyst recovery

- Electrochemical membrane technology for continuous separation
- Ion exchange resin assisted capture
- Selective separation process to recycle homogeneous catalyst and capture aromatic acids in a single device
- Can greatly reduce the salt and wastewater burden on the biorefinery



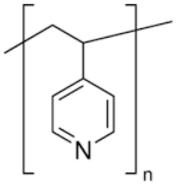
Approach: Homogeneous depolymerization

HOMOGENEOUS LIGNIN DEPOLYMERIZATION

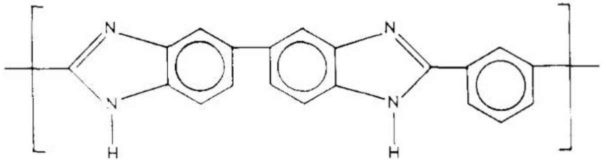


Dewatering

- Resin testing and design
- Electrochemical membrane separation platform technology
- Target 10 -100x lignin-derived aromatics concentration increase
- Generalizable well beyond APL

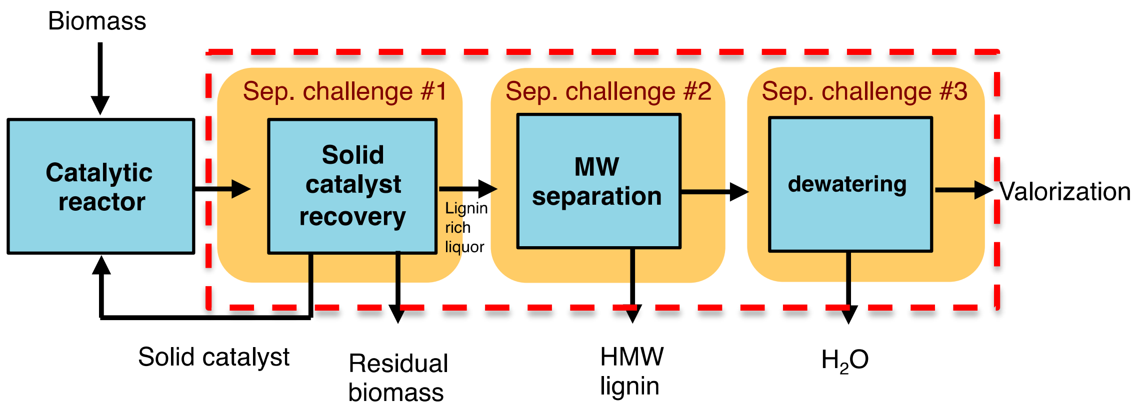


Polyvinylpyridine (PVP)
Polybenzimidazole (PBI)



Approach: Heterogeneous depolymerization

HETEROGENEOUS LIGNIN DEPOLYMERIZATION



Solid catalyst recovery

- Filtration
- Batch reactions with metal and metal oxides as **example solid catalysts**

MW separations

- Membrane filtration
- Chromatographic approaches (simulated moving bed)
- Membranes with targeted MW sizes
- Common problem in lignin processing

Dewatering

- Resin testing and design
- Electrochemical membrane separation platform technology
- Require high concentrations of aromatic compounds for bioreactor feed or direct use in valorization stream
- Generalizable for most lignin streams

Technical Approach (and Future Work) – Fermentation Sep.

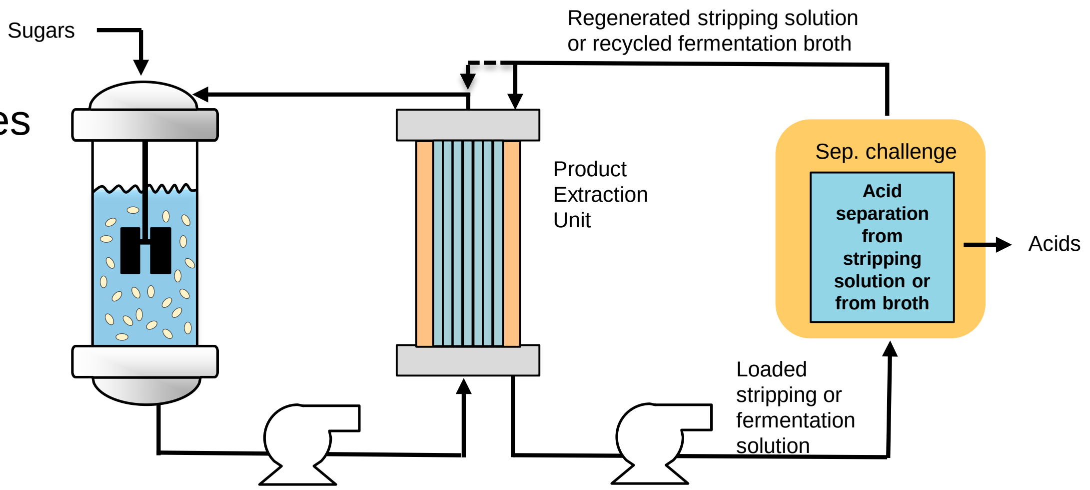
Approach: examine recovery of challenging fermentation intermediates

- Carboxylic acids as intermediates (often most expensive and challenging products to remove)
- Common problem in biorefining given many value-added chemicals from biomass are acids
- Determine if extraction is a scalable separations solution

Challenges and Critical Success Factors

- Developing scalable, cost-effective extraction processes – two processes
  - Solvent recycle and recovery or
  - Resin wafer separation for direct acid capture from ferment. broth
- Membrane fouling
- Minimization of salt generation
- Produce **acids,** not carboxylates

BIOLOGICAL PRODUCTION OF ACIDS

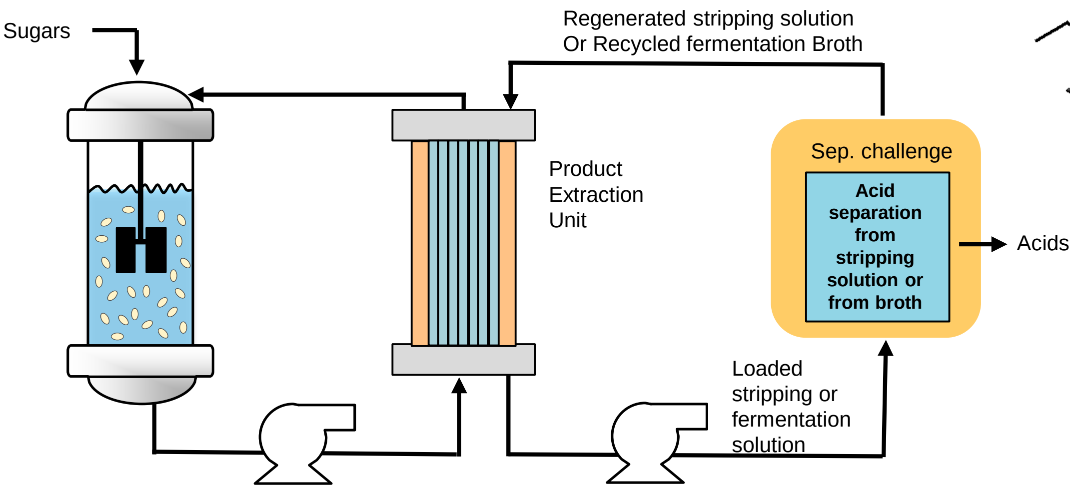


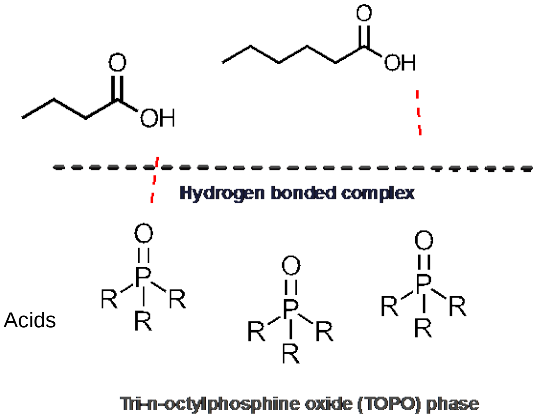
Working towards **quantitative separations targets** based on TEA

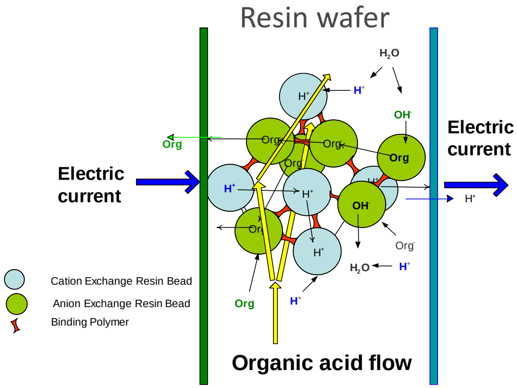
- Applying technical approaches to exemplary lignin streams mainly from NREL pilot plant
- Common problems for lignin valorization: results will be applicable to many lignin streams

Approach: Fermentation separations

BIOLOGICAL PRODUCTION OF ACIDS







Developing more efficient methods for extractive fermentation

- Using **TOPO** as extractant for online extraction
- Enables recovery of carboxylates in protonated form
- Using ion-exchange **resin-wafer electrodeionization** to selective separate organic acid
- Enable direct capture and concentration of carboxylates in the acid form
- Simplifies downstream processing

Biochem- Relevance, Current Progress, and Impact

Relevance: Develop new separation strategies that will improve the efficiency and costs of lignin valorization and ease recovery of fermentation products and intermediates.

Progress to Date:

Lignin Separations

- Developed baseline protocol to produce lignin streams for distribution to partners
- Screened ion-exchange resin wafer compositions for ability to capture the targeted aromatic acids vanillic, perulic and coumaric acids
- Began measurements of particle size distribution in alkaline pretreated liquor

Fermentation Separations

- Identify optimal resin wafer compositions to effectively ionize carboxylic acids and succinic acids in the aqueous phase. These compositions will be used in an electrochemical membrane separation system to extract organic acids from fermentation broth.
- Began working with TOPO and other novel extraction agents to understand carboxylate removal from fermentation.

Impact: Biochemical conversion routes will become more cost competitive because a low molecular weight aromatic acid stream – a valuable co-product - will be cost effectively produced from biomass and costs to recover intermediates and final products from fermentation broth will decline.

Goal Statement – Thermochemical (TC) Separations

Increase catalyst lifetime & renewable carbon efficiency to fuels and reduce need for external non-renewable hydrogen through the use of enhanced separations

Relevance to Conversion Platform, BETO, and industry:

- High C and H atom efficiencies are essential to achieve BETO cost target of a $3/gge MFSP and >50% GHG reduction
- Frequent catalyst replacement and maintenance currently represent a major cost in thermochemical conversion processes

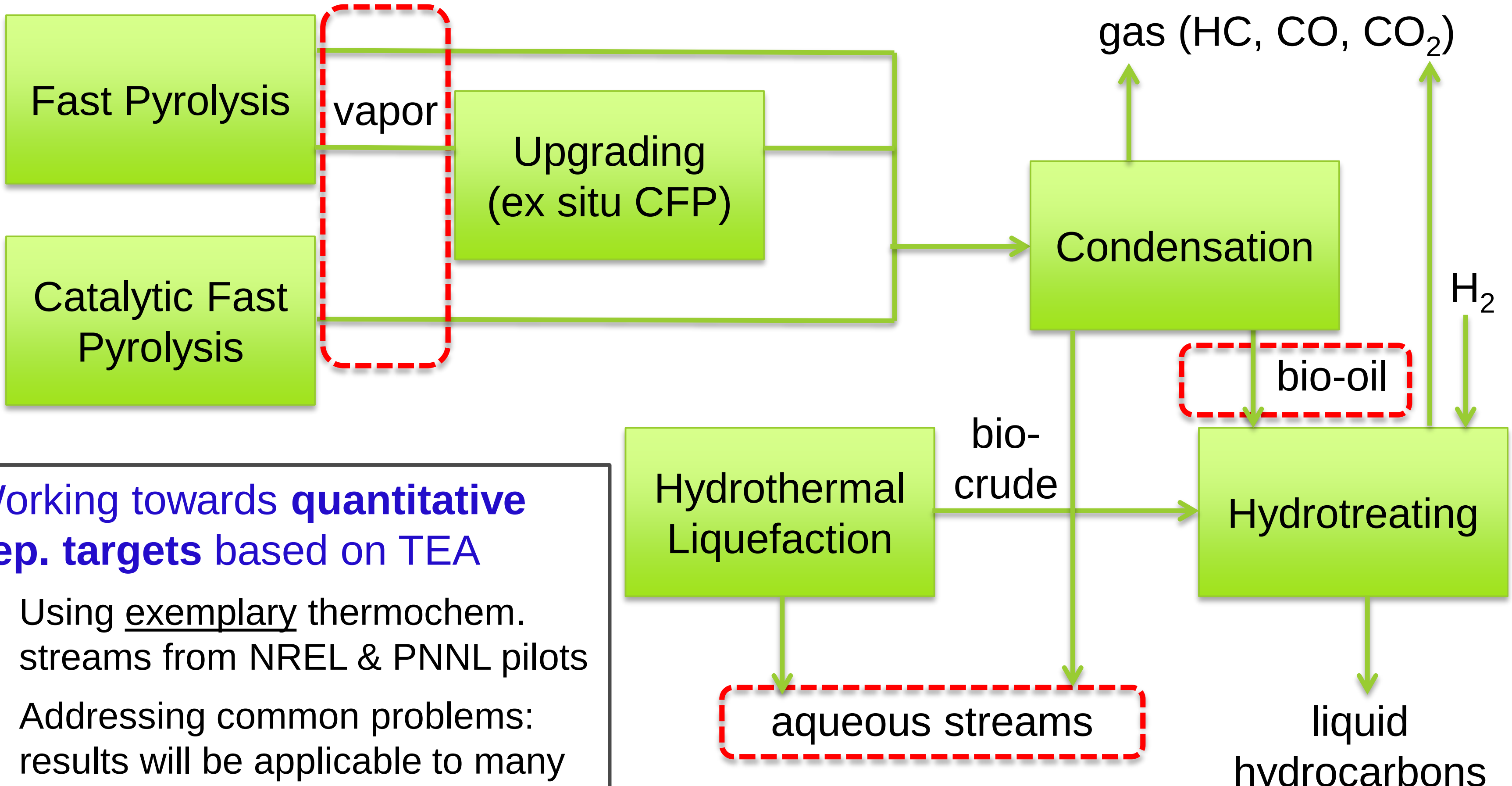
Team from 4 national labs:

Argonne NATIONAL LABORATORY — NREL NATIONAL RENEWABLE ENERGY LABORATORY — OAK RIDGE National Laboratory — Pacific Northwest NATIONAL LABORATORY

Overall Technical Approach – Thermochemical Separations

Approach: examine separation methods & their integration for enhanced C & H atom efficiency & hydrotreating catalyst durability

[red dashed box]: separation challenges
- Pyrolysis vapor clean up
- Liquid bio-oil clean up
- Aqueous stream HC recovery

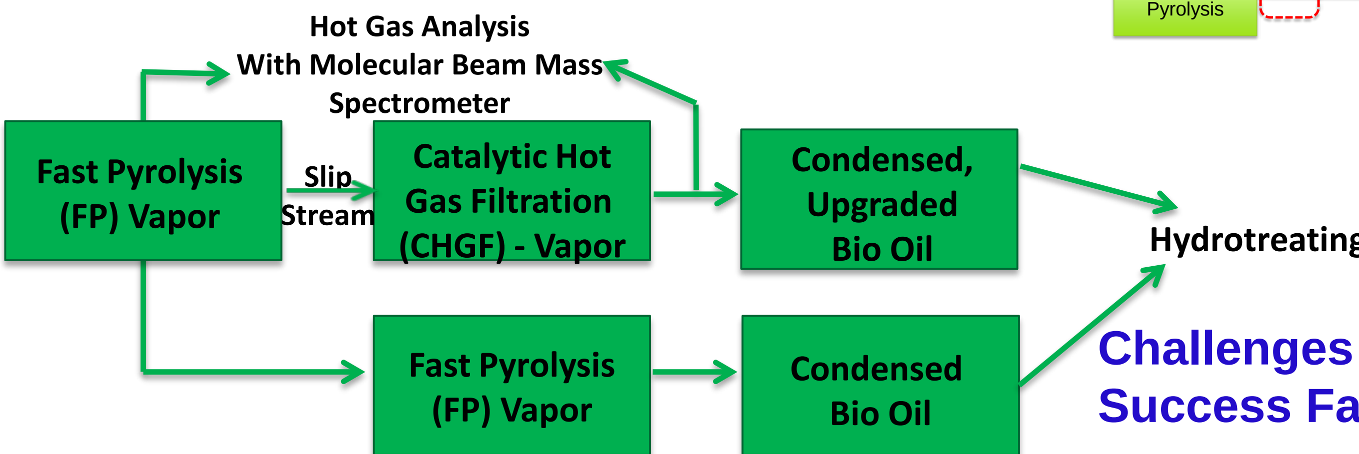


Working towards **quantitative sep. targets** based on TEA

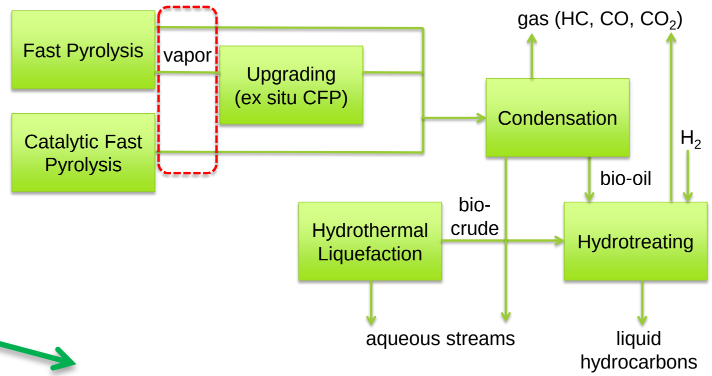
- Using <u>exemplary</u> thermochem. streams from NREL & PNNL pilots
- Addressing common problems: results will be applicable to many other bio-oil and aqueous streams

Approach – Catalytic Hot Gas Filtration

Approach: filter-based upgrading of hot pyrolysis vapors to remove char and alkali metals



- Optimize HGF filtration performance
- ***Add catalysts*** to HGF to **pre-deoxygenate** vapors for downstream upgrading
- Condensed oils will be hydrotreated at PNNL
- Conduct TEA for cost impact assessment

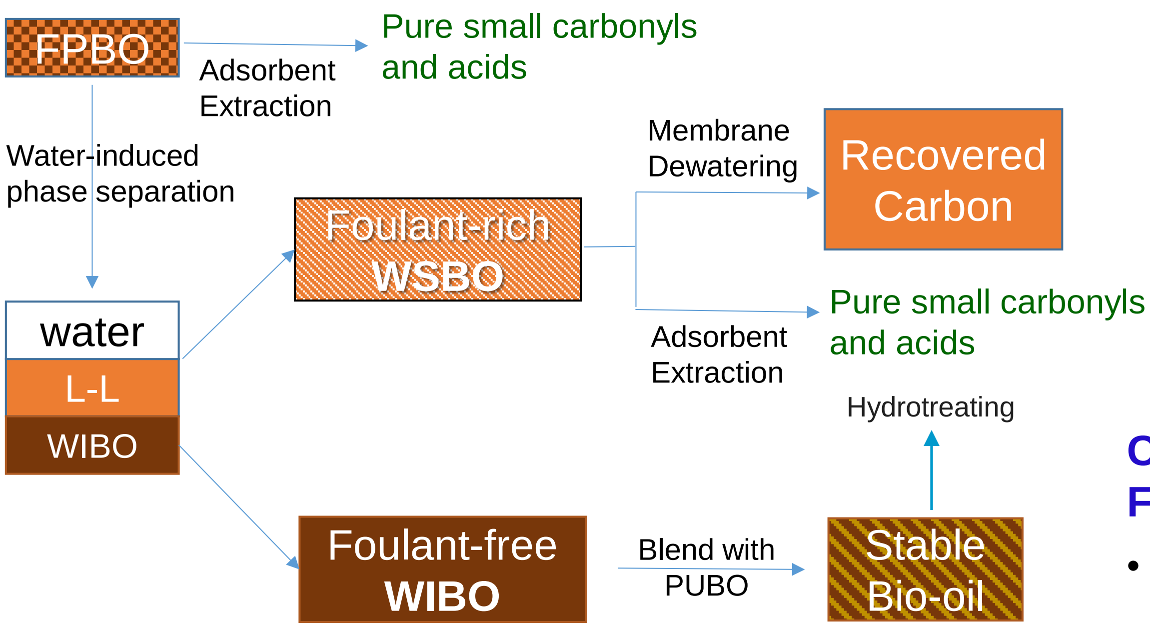


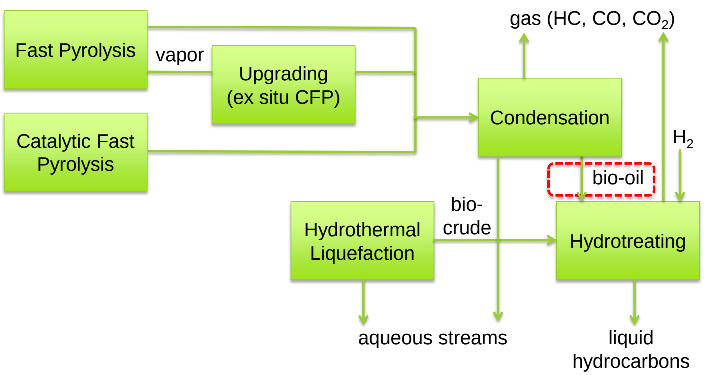
Challenges and Critical Success Factors

- Challenge: filter plugging and deactivation => consider dual beds swing operation, as pressure drops to maintain performance
- Targets for upgraded bio oil:
  - Reduce metals and char to < 0.01%
  - De-oxygenate vapors to <30% oxygen content (water-free basis)

Approach – Molecular Removal Pre-Processing of Liquid Bio-oils

Approach: Integration of solvent extraction, adsorbents & membranes for selective separation of catalyst foulants (carbonyls, carboxylic acids)





FPBO: Fast Pyrolysis Bio-Oil
WSBO: Water Soluble Bio-Oil
WIBO: Water Insoluble Bio-Oil
PUBO: Partially Upgraded Bio-Oil

- Develop efficient solvent extraction methods to concentrate foulant molecules
- Selective adsorptive removal of reactive carbonyl and carboxylic acid molecules
- Pre-processed oils will be hydrotreated at PNNL
- Conduct TEA for cost impact assessment

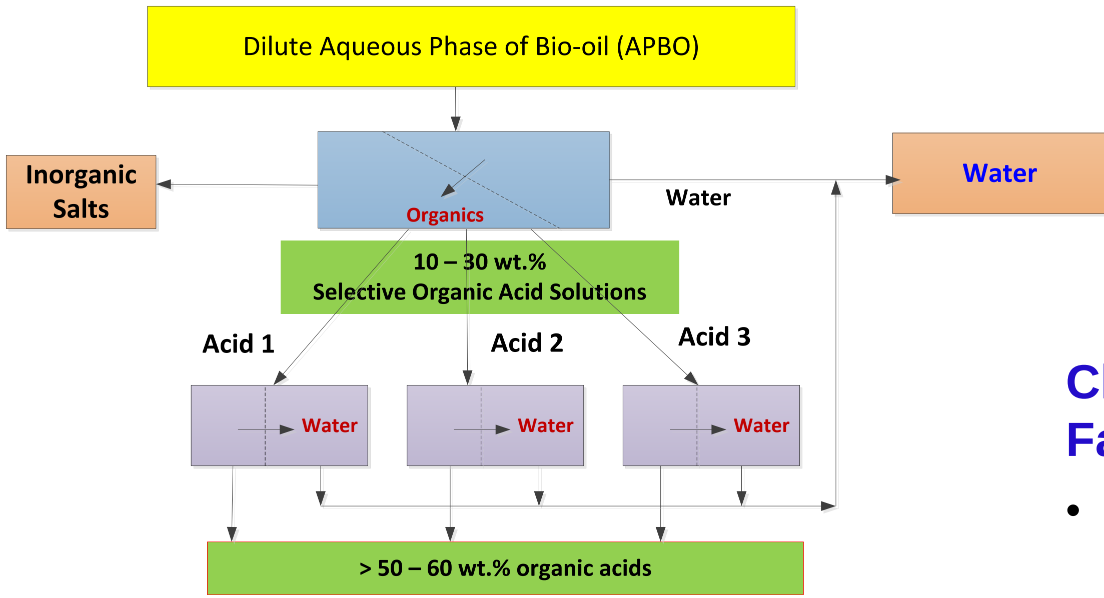
Challenges and Critical Success Factors

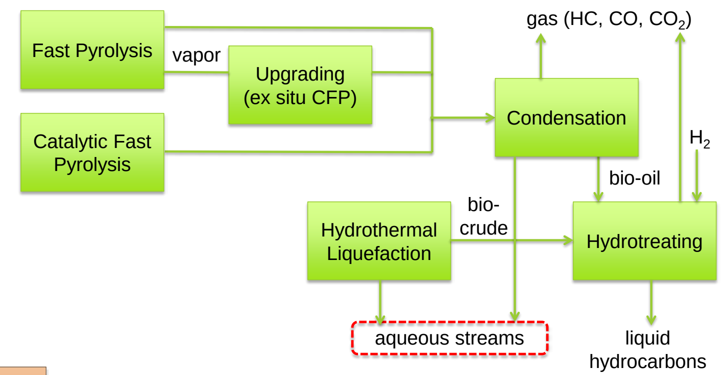
- Developing scalable & regenerable adsorption processes
- Targets
  - Foulant concentration in stabilized bio-oil >90% less than in raw bio-oil
  - >200% catalyst lifetime enhancement & >25% conversion cost reduction

Approach – Carbon Recovery from Aqueous Streams

Approach: Integration of ANL polymer wafers (extraction) with ORNL advanced membranes (dewatering) to obtain concentrated acids

Selective Separations-Dewatering





- Wafer-Membrane selective separations of mixed organic acids in APBO into pure organic acid streams
- Effective water removal membrane separation to concentrate organic acids
- Conduct TEA for cost impact assessment

Challenges and Critical Success Factors

- Challenge: selectivity and material stability to handle concentrated organic acids
- Targets:
  - Organic acid concentration >60 wt%
  - 90% acid capture and <$0.25/lb acid

Thermochem-Relevance, Current Progress, and Impact

Relevance: Use advanced separations technology to improve carbon efficiency and operability of downstream upgrading unit operations by reducing catalyst poisons, removing acids, and recovering lost carbon from aqueous streams

Progress to Date:

Catalytic Hot Gas Filtration

- Fabricated and tested filter holder assembly and pyrolyzer slipstream reactor
- Characterized Pall ceramic filters for surface area, porosity, fluid uptake (wettability), metal content, pressure drop
- Conducted slipstream testing of uncoated and 1% NiO coated ceramic filters
- Completed preliminary TEA

Molecular Removal Pre-Processing of Liquid Bio-oils

- Began screening of adsorbents (eg. molecular sieves and resins) for extraction of carbonyl and acid components from bio-oil with and without water induced phase separation
- Analysis of dozen extraction samples completed (eg. TAN, CAN, carbonyl content)

Carbon Recovery from Aqueous Streams

- Conducted screening of resin wafer composition for acid extraction
- Resin wafer membrane selective separation increased concentration of 1 wt% acetic acid solution in water to ~15-20wt.% (separation factor = 20)

Impact: If successful, this project will lead to dramatically enhanced catalyst lifetime and C/H atom efficiency of TC intermediates upgrading and monetization of aqueous stream molecules.

Goal Statement – Ionic Liquid (IL) Separations

Development of ionic liquid recycling technologies to enable cost-effective biomass processing

Relevance to Conversion Platform, BETO, and industry:

- Enable innovative biomass deconstruction approaches to improve yields and lower the cost of fermentable sugars
- IL recycling is essential to enable cost-effective IL deconstruction technologies (IL process TEAs assume >95% IL recycling)

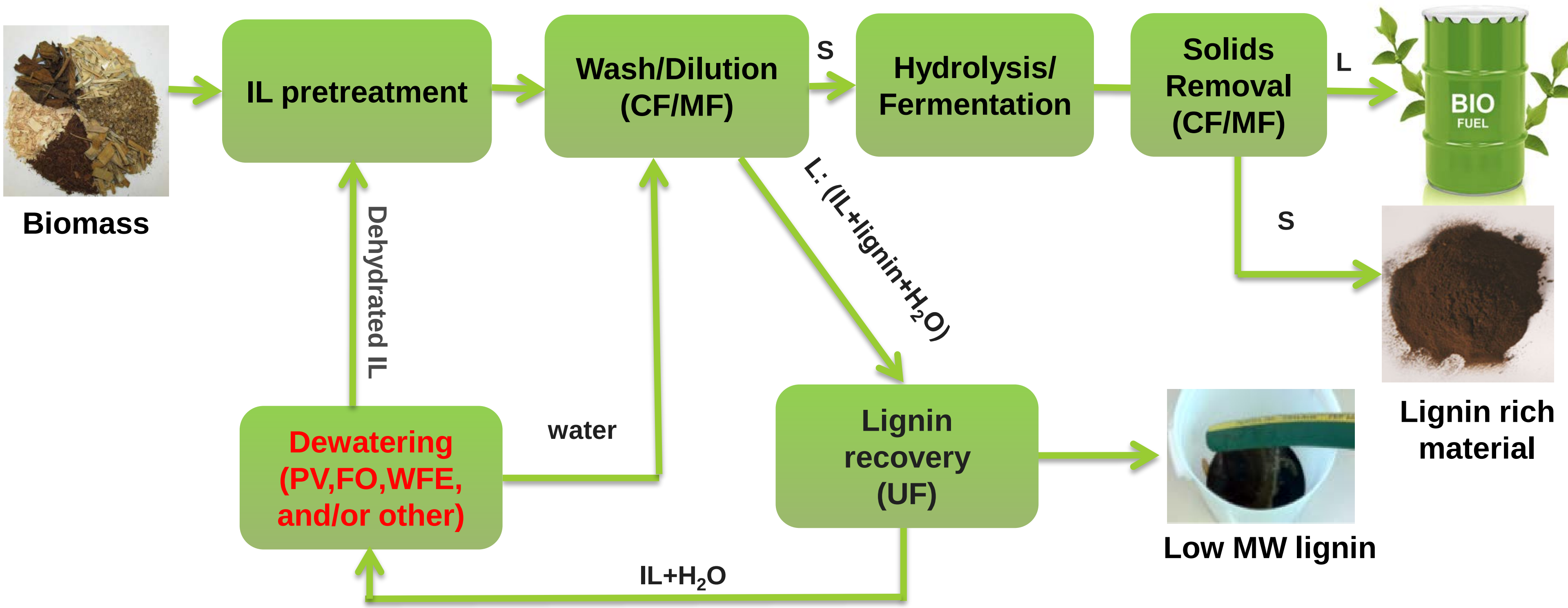
BERKELEY LAB
Lawrence Berkeley National Laboratory

Sandia National Laboratories

INL
Idaho National Laboratory

Technical Approach-Overall process

Team will evaluate different ILs' recyclability and identify potential links with other SepCon teams (e.g. biochemical team-lignin recovery, biocatalyst preservation, etc.)



PV: Pervaporation, FO: Forward Osmosis, WFE: wiped film evaporator, CF: Centrifugation, MF: Micro-filtration, UF: Ultra-filtration, S: solid stream, L: liquid stream

Pic from http://www.yelloblu.com/blog/biofuels-part-three-biomass-future-fuels

Technical Approach-PV, FO, WFE

- **Challenges of IL/H_2O separation using current methods**
  - **Conventional Distillation: High energy cost, low selectivity**
  - **Extraction: Low recovery rate, multi-step, waste stream**
  - **Electrodialysis: High IL loss, limited concentration ranges**
- **Approach I: pervaporation (PV)**
  - **Low operation temperature**
  - **Excellent resistance of IL permeation (IL recovery >99.9%)**
  - **High separation selectivity for azeotrope**
- **Approach II: forward osmosis (FO)**
  - **Addresses high concentration solutions**
  - **Low operation temperature (70-80 ºC)**
  - **High energy efficiency**
  - **Better fouling resistance**
- **Approach III: wiped film evaporation (WFE)**
  - **High efficiency to remove water to ppm level**
  - **Industrial relevant process used for IL production**
  - **Easily scalable**

Ionic Liquid Separations - Relevance, Current Progress, and Impact

Relevance: Development of ionic liquid (IL) recycling technologies is necessary to enable IL-based biomass deconstruction approaches to improve yields and lower the cost of fermentable sugars

Progress to Date:

- Selected ILs and IL concentration ranges to be evaluated with proposed dewatering technologies
- Measured the water concentration-dependent osmotic pressure of one selected IL
- Started to establish high-performance liquid chromatography and conductivity methods to assess IL concentration, tritration-based methods to quantify trace water

Impact: If this team is successful, the underlying assumption of 95-99% IL recycle in the techno-economic analysis of conversion processes with IL-based deconstruction will be validated, improving techno-economic evaluation of these processes as an element of BETO's research and development portfolio.

BERKELEY LAB Lawrence Berkeley National Laboratory

Sandia National Laboratories

INL Idaho National Laboratory

Goal Statement – Algae Separations Seed Project

Two aims and associated outcomes:

- Enable cost and energy effective algae dewatering
- Enable cost effective lipid recovery from disrupted algae with little or no toxic solvents

Relevance to Conversion Platform, BETO, and industry:

- TEA results indicate reducing cost of algae dewatering is essential to reducing cost of algae platforms.
- Lipid separation accounts for 12% of biofuel cost ($0.523/gge)
- Solution must be scalable and commercially viable

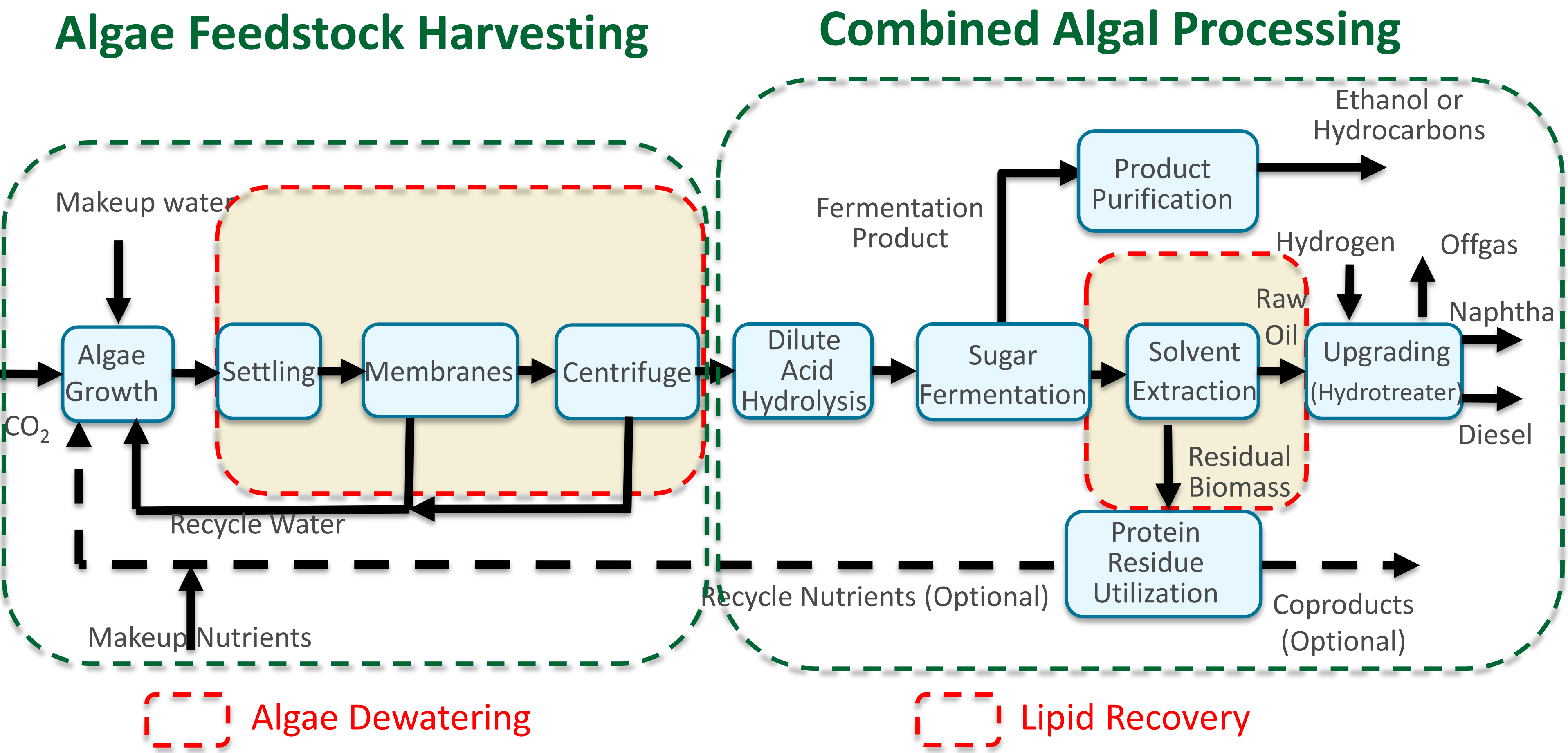
Team from 3 national labs:

Oak Ridge National Laboratory

Los Alamos National Laboratory EST. 1943

NREL National Renewable Energy Laboratory

Algae Seed Project – Separation challenges targeted



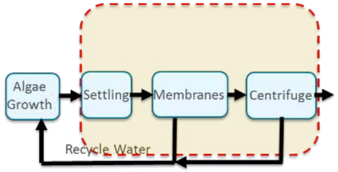
- Challenge: At a minimum, dewatering accounts for 10% of feedstock costs for "well-behaving" types of algae
- **Dewatering solution is applicable for a broad range of algae species and platforms**

Combined Algal Processing (CAP)

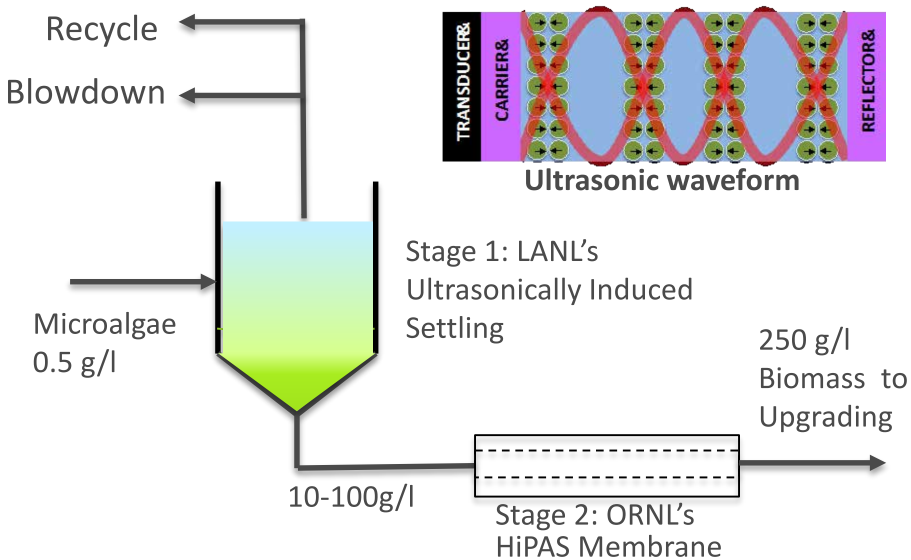
- SOT lipid separation accounts for 12% of biofuel cost ($0.523/gge)
- **Reduce costs by using less or eliminating solvent**

Algae Seed Project – Technical Approach

Algae Dewatering

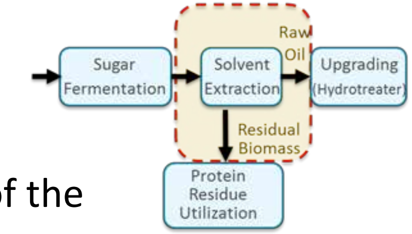


- **Ultrasonics** have the potential to reduce 1st stage harvesting energy requirements and costs by more than a factor of 10
- Ultrasonics moves particles to nearby nodes to make big particles easier to separate
- **Removes 99% of the water before filtration**
- Employs **robust fouling-resistant membranes**
- Use **recyclable flocculants** to reduce flocculant costs by 85% and recover more biomass.



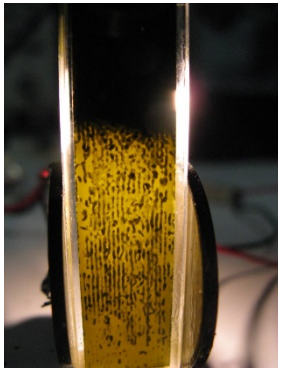
Ultrasonic waveform

Lipid Recovery

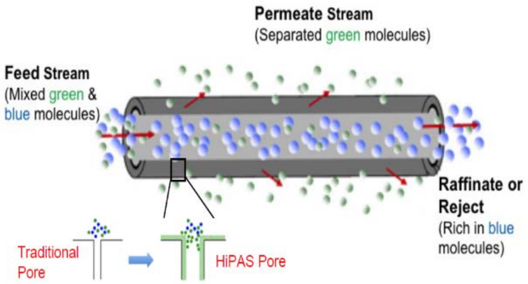


- Post sugar fermentation of the microalgae
- Eliminate toxic solvents by using ultrasound to agglomerate lipids and advanced membranes to remove the water.

(Step 1) Ultrasonic Separation



(Step 2) Crossflow Membrane

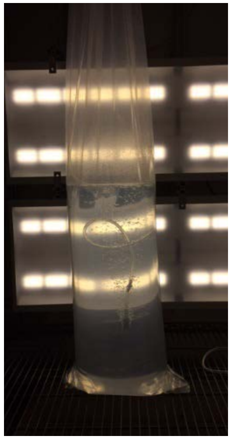


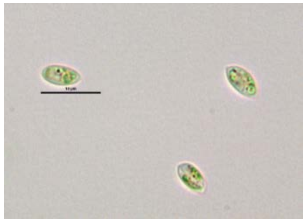
Applicable to *any* microalgae feedstock pathway!

Algae Seed Project – Role of each lab

NREL

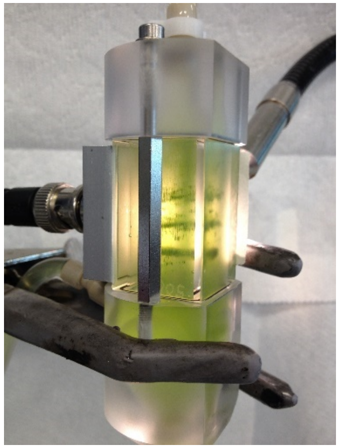
- Stream steward
- Whole microalgae
- Acid-disrupted algae





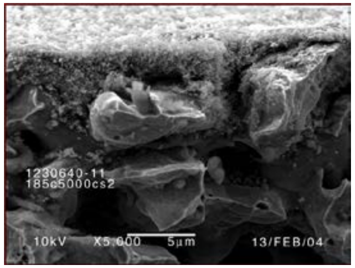
Los Alamos National Laboratory

- Separations-relevant properties
- Ultrasonic separations



Oak Ridge National Laboratory

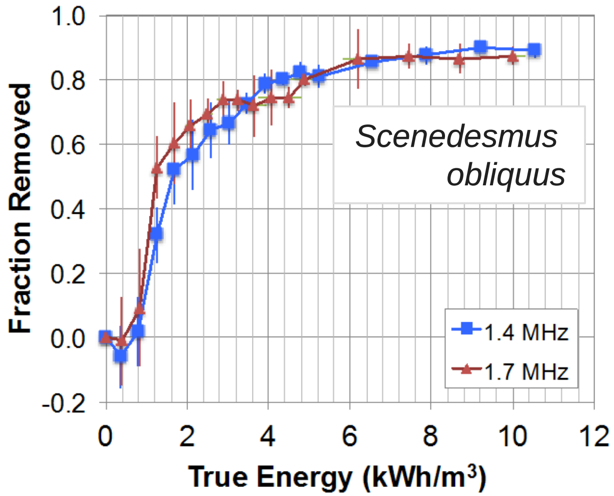
- Membrane separations
- Use surface enhanced robust inorganic membranes to minimize fouling
- Test cleanability of membranes



Algae (Seed) -Relevance, Current Progress, and Impact

Our technology moves tiny particles (algae, lipids, etc) a short distance for aggregation and dewatering, generating less volume of material to filter, and requires >10 times less energy than conventional separation technologies.

Progress to date (LANL):



Ultrasonic removal tests in combination with passivity measurements (not shown) will determine dewatering costs using ultrasound. Passivity is a property proportional to the energy required for removal by ultrasound.

Progress to date (ORNL):

Superhydrophilic membranes and a crossflow membrane test system have been fabricated for algae tests.

Impact:

If this project is successful, energy consumption and algae dewatering costs will be up to 90% less for all algae strains and toxic solvent use in lipid recovery will be reduced.

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

Goal Statement – Biocatalysts Preservation Seed Project

Two aims and associated outcomes:

- Enable cost-effective **synthesis of advanced materials for separations**
- Improve bioconversion efficiency by extending lifetimes of enzymes and fermenting microorganisms by **removing toxins and inhibitors from bioreactor feed and fermentation broth**

Relevance to Conversion Platform, BETO, and industry:

- Distinct molecular contaminants from bioreactor feed and metabolic intermediates in conversion deter the cost-effective production of biofuels/products by biocatalytic routes
- Targeted removal of toxins and inhibitors will increase production rates, extend biochemical catalyst lifetimes, enable reactor-integrated separations and process intensification.

Technology development:

Argonne NATIONAL LABORATORY

Feedstock/bio-processing streams — NREL NATIONAL RENEWABLE ENERGY LABORATORY

Synergistic materials approaches — OAK RIDGE National Laboratory

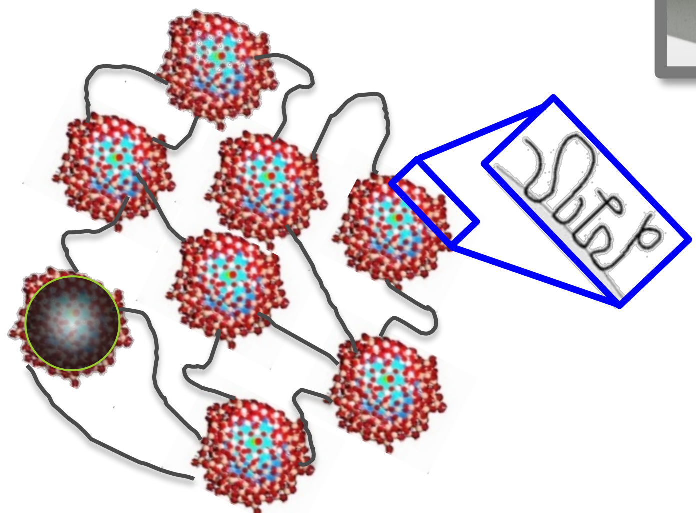
Technical Approach (and Future Work) – Materials Synthesis

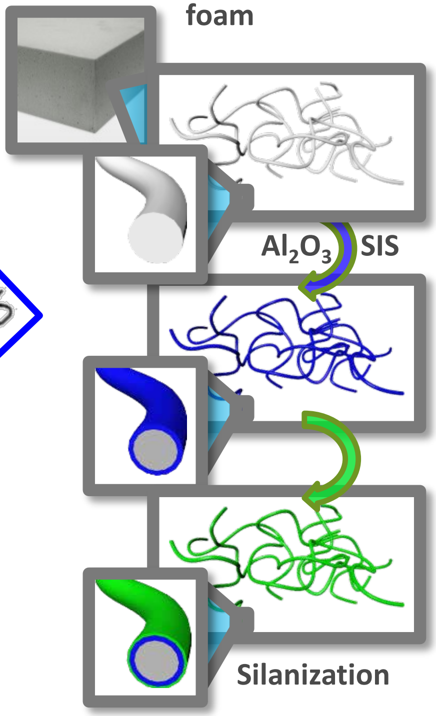
Approach: examine the utility of two varieties of solid, high-surface-area adsorbents

- Surface-treated, nanostructured polymeric networks
- Reusable, environmentally benign adsorbent foams

Challenges and Critical Success Factors

- Developing scalable, cost-effective syntheses
- Designing surface treatments with high capacity and selectivity
- Understanding factors which allow for materials reuse
- Minimizing materials and biological interferences


heterogeneous vapor-phase polymerization



Working towards **quantitative separations targets** based on TEA

- Realistic projections for materials production costs at scale will be needed
- Demonstrated reuse (100X) within a bioreactor will be key for low-cost deployment

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

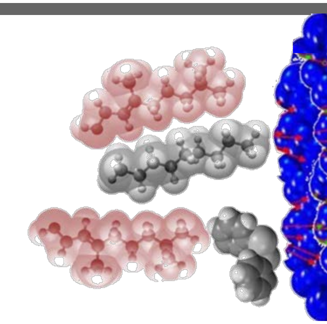
Technical Approach (and Future Work) – Targets and Costs

Approach:

- Target toxins from bioreactor feed and inhibitors from fermentation
- Develop economically-favorable, bioreactor-integrated processes

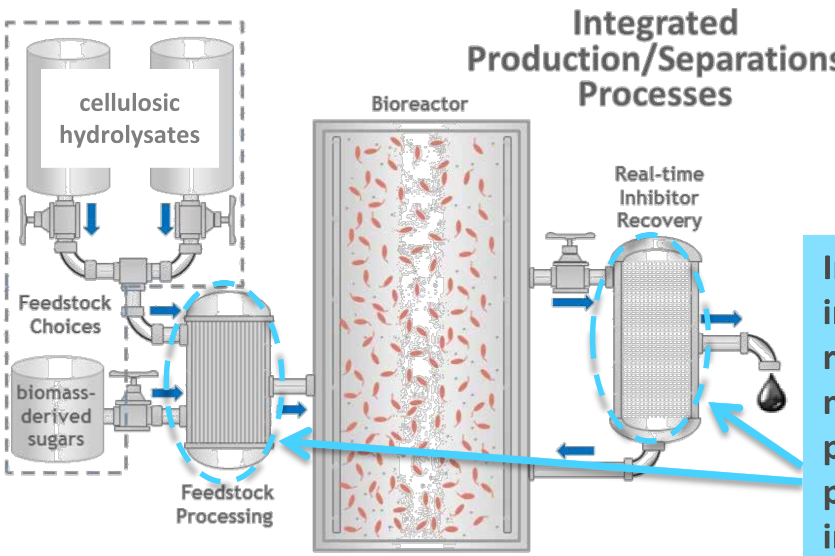
.....from sugar and lignin streams
Salts, proteins, inhibitory oligomers, lignin-derived aromatics, aliphatic acids, or sugar-dehydration products

.....*in situ* intermediate removal from sugar or lignin upgrading
Short-chain alcohols, short-chain acids, diacids, or fatty alcohols from background of carbohydrates and aromatics



Challenges and Critical Success Factors

- Selecting targets of greatest impact that are amenable to materials approach
- Developing bioreactor-compatible separations processes
- Increase titer, rate, and yield for bioreactor integration



Impurities and inhibitors can be removed in multiple stages of process to raise product titers and increase production rates

Working towards **quantitative separations targets** based on TEA

- Binding affinities/specificities may prove more important than materials reuse
- Indirect impacts: favorable TEA depends upon appreciating influences on other processes

Biocat. Pres. Relevance, Current Progress, and Impact

Relevance: Develop novel materials for separations to specifically remove toxins and inhibitors from bioreactor inputs and fermentation processes

Progress to Date:

- Identified sets of target compounds
  - approachable with materials platform
  - many known to deter efficiency of bioconversion strategies using cellulosics
- Preparing process energy and material use data for cross-cutting analysis team
- Designing surface treatments for selective adsorption

	Toxin or inhibitory species	
Target Number	Bioreactor feed	Fermentation Broth
1	Furfural / methyl furfural	Butyraldehyde
2	Ferulic acid	Malondialdehyde
3	Benzoic acid	Succindialdehyde
4	Coumaric acid	Tolualdehyde
5	Phenyl acetate	Isoprenoid species

Targets to be used to test the molecular recognition approaches used within the Biocatalyst Preservation Seed Project

Impact: Substantially improve titers, rates and yields and approach continuous fermentation to reduce costs of bioconversion of cellulosics to fuels and products

Summary

Overview

Move cost-effective, high-performing separations technology to market faster, targeting challenges relevant to BETO's priority pathways and industry.

Approach

Harness expertise across eight national labs to undertake research projects informed by their influence on process economics. Concentrate efforts in biochemical, thermochemical, algae, ionic liquid separations.

Progress

Technical work commenced. Industrial advisory board engaged and listening day scheduled. Cross-cutting analysis team engaging with teams to advise regarding technology influence on process economics.

Relevance

The consortium addresses separations challenges identified in the MYPP, strategic plan and by industry that are a significant contributor to biofuel and bioproduct production costs.

Future Work

Informed through techno-economic and life cycle analyses & industry engagement, the consortium will carry out a portfolio of research covering biochemical, thermochemical, algal, and ionic-liquid based processes.

QUESTIONS?

ADDITIONAL SLIDES

Industrial Advisory Board Charter

The Bioprocessing Separations (BioESep) Consortium Industry Advisory Board (IAB) will represent experts from industry and other entities in bioenergy, biomanufacturing, industrial biotechnology, and related fields. The IAB will **help the consortium maintain an industry-relevant focus and knowledge of recent technology advances and challenges.** BioESep anticipates that the IAB will be comprised of 7-9 members on a 3-year rotation, with the ability to continue beyond should the interaction be mutually valuable.

The board will meet with the steering committee and team leads quarterly, with two meetings in person per year. The board will **provide advice** to the steering committee and team leads, **review results and progress** in comparison with work plans, **provide feedback regarding prioritization of research projects** (experimental and analytical), and **inform development of the consortium's strategy** for out years. Furthermore, the IAB will help in planning an industry workshop within the first year of BioESep insofar as providing feedback regarding the agenda and identifying relevant invitees.

Relevance to MYPP goals and barriers

MYPP Goals and Barriers	Consortium Approach to Addressing	MYPP citation
Separations and cleanup are overarching issues in conversion	The consortium was created to address this key challenge.	2-67
Cost-effective routes to eliminating impurities in intermediates that inhibit chemical and biological catalysts	The Biochemical Separations and Biochemical Catalyst Preservation Teams both address this barrier.	2-68, 2-73
BETO is targeting improved separations processes to increase yields and purity including solid-gas (e.g., hot gas filtration), solid-liquid, gas-liquid, and liquid-liquid separtaions.	The Thermochemical Separations team's catalytic hot gas filtration project addresses this barrier whereas the overall consortium addresses many of these areas of interest.	2-76