Bioprocessing Separations Consortium Overview, Project Management, & Diversity, Equity, and Inclusion

BETO 2023 Project Peer Review

Performance-Advantaged Bioproducts and Bioprocessing Separations

April 6, 2023

This presentation does not contain any proprietary, confidential, or otherwise restricted information

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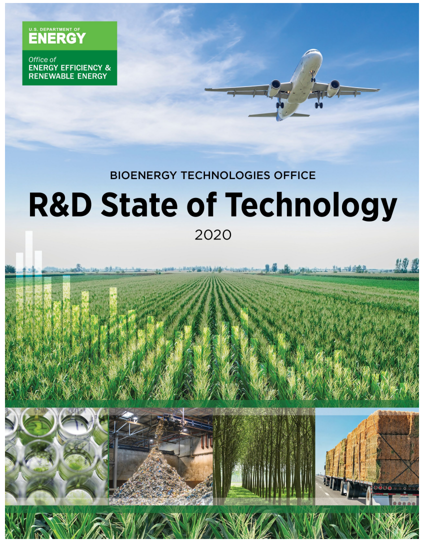
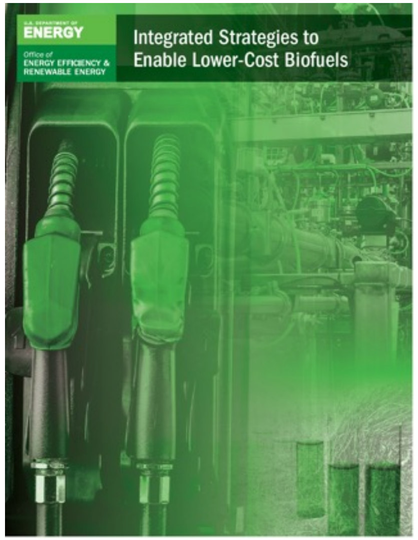
Bioprocessing Separations

- Chemical separations account for up to 15% of total energy consumption in the United States.[1]
- Separations account for up to 50-70% of processing costs for biofuels and bioproducts.[2,3]
- Efficient separation and purification steps are key integration challenges for all technology pathways.[4]
- There is a need to raise technical maturity of biobased processes, including separations. Improving separations will positively affect the entire bioeconomy.[5]
- Additional research is needed to bridge the gap between small-scale and large-scale technologies.[5]
- Synergy of separations with conversion processes has the potential to reduce costs while maintaining high recovery rates and yields.[6]

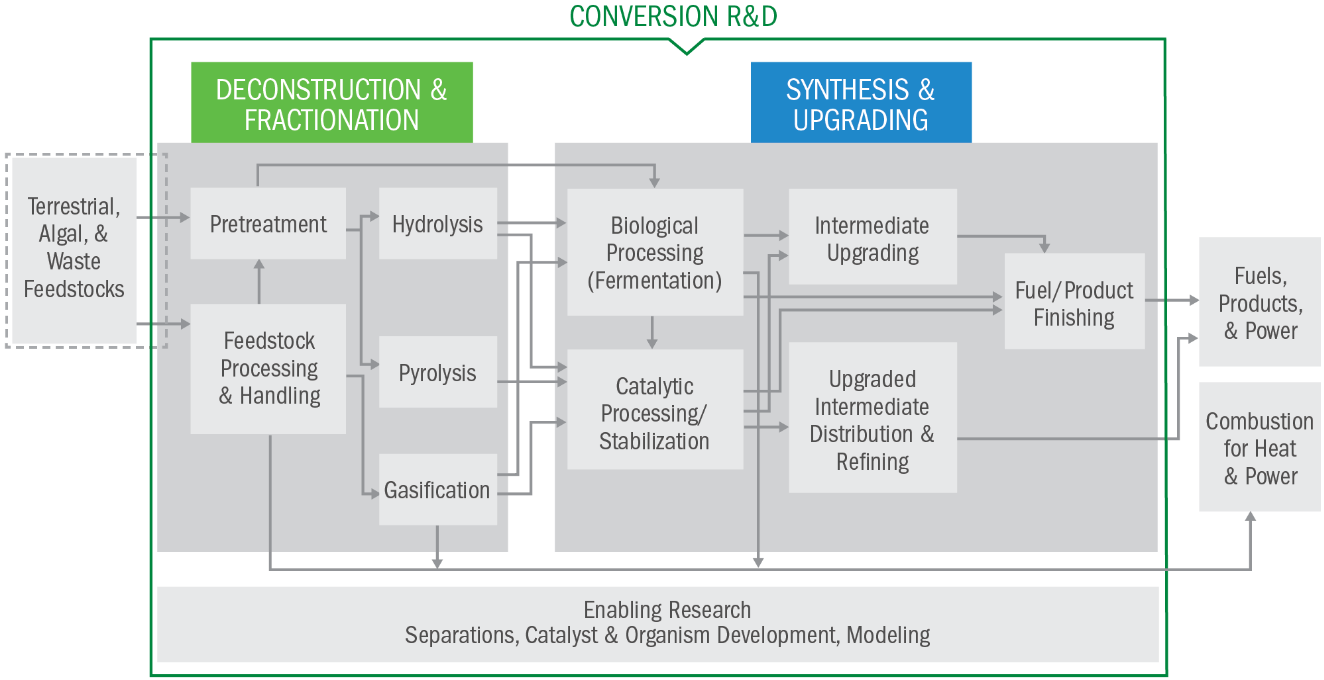




1. Sholl and Lively. "Seven chemical separations to change the world," *Nature*, **2016** 532: 425-437.
2. EERE. 2016. Strategic Plan for a Thriving and Sustainable Bioeconomy.
3. Biddy et al. "The Techno-Economic Basis for Coproduct Manufacturing To Enable Hydrocarbon Fuel Production from Lignocellulosic Biomass." *ACS Sustainable Chem. Eng.* **2016** 4: 3196-3211.
4. EERE. 2022. Bioenergy Technologies Office 2020 R&D State of Technology.
5. EERE. 2018. Moving Beyond Drop-In Replacements: Performance-Advantaged Biobased Chemicals
6. EERE. 2020. Integrated Strategies to Enable Lower-Cost Biofuels.

Bioprocessing Separations Consortium

- The Consortium formed in FY17 to address stakeholder feedback that separations challenges are an obstacle to cost-competitive production of biofuels and bioproducts.
- BETO analyses identified separation challenges that, if resolved, could reduce the minimum fuel selling price of biofuels by up to 50%-70%.
- All biomass conversion pathways require cost-effective, molecularly-efficient separations.



Project Overview

Bioprocessing Separations Consortium

Goals	Develop cost-effective, high-performing separations technologies through coordinated separations research that targets industry-relevant bioprocessing separations challenges. Address BETO decarbonization priorities (see slide 5) by identifying and overcoming separations challenges associated with the conversion of biomass into fuels and chemicals.
Current status	Separations are a key driver of bioprocessing costs. Case-by-case approaches in the absence of general design principles, data, and strategies lead to energy and water intensive separations processes that are expensive. Bio-based separation processes have not reached the technical maturity level for commercialization.
Importance	Separations often impede cost-effective and sustainable bioprocess development.
Risks	Technologies under development could be more costly than existing approaches. New materials may prove unstable or perform poorly in bioprocessing environments.

Approach – FY22 Organizational Chart

Bioenergy Technologies Office

Steering Committee

Advisory Board

Analysis

R&D-guiding TEA and LCA
(E. Tan and C. Freeman)

Update Assessment of BETO Separation Challenges/ Opportunities
(E.Tan and C. Freeman)

Computational Separations
(V. Glezakou)

BETO Collaborative Projects

Lignin-Rich Stream Fractionation and Purification
(E. Karp)

Redox-based Electrochemical Separations
(E. Barry)

2,3-Butanediol Separations
(A. Church)

New Capability Development

Counter Current Chromatography
(E. Karp)

Volatile Product Recovery
(P. Laible)

New Objectives and Emphasis Areas for FY23

- Consortium underwent Merit Review in 2022
- Continue solving separations challenges that can have broad and significant technical, cost, and life cycle impacts across biomanufacturing platforms
- Emphasis on developing and advancing energy and carbon-efficient separations to support **decarbonization of transportation and industry**
- Scale-up, deployment, and commercialization of **sustainable aviation fuels** along with **low-carbon bio-products**
- Emphasis on mature conversion processes that are otherwise limited by separations efficiency
- Increased **coordination and collaboration with other BETO projects and consortia** are encouraged
- Increased focus on **diversity, equity, and inclusion**

BETO strategic goals

- **Decarbonize the transportation sector** through research, development, and demonstration to produce cost-effective, **sustainable aviation fuel** and other strategic fuels.
- **Decarbonize the industrial sector** through research, development, and demonstration to produce cost-effective, **sustainable chemicals, materials, and processes** utilizing biomass and waste resources.

Approach

Consortium Approach to FY23 Portfolio Selection

Identify separation challenges	Assess consortium applicable capabilities	Concept development and submission	Concept review and down-selection	Advisory Board Feedback
Industry Advisory Board and listening days	Material design, development, and evaluation	Technology description	Review sub-committee with representation from each lab + BETO	Technical merit/feasibility
Conference special sessions	Process development	Comparison to technical baseline	Proposals rated as high, medium, and low for each category	Industrial relevance of the separations challenge addressed
Directed Funding Opportunity	TEA/LCA	Why separations development is required in industry and/or for other BETO projects		Potential to transition process or technology to industry
BETO design cases	Computation	Progress to-date /potential to advance separations		
BETO consortia		Path to reducing bioprocessing costs and GHG impacts		
SepCon challenge stream analysis				

Approach – FY23 Organizational Chart

Sponsor
Provides high-level decision, input, and resources.

- Bioenergy Technologies Office

Consortium leadership
Provides technical guidance, monitors progress and impact, coordinates external communications, and manages Consortium reporting and monthly calls.

- Principal Investigator
- Task 1
  Project Management and Integration

Laboratory leadership
Advises on Consortium direction and strategy and oversees project teams and activities at each laboratory.

- Laboratory Executive Board
- Task 1
  Project Management and Integration

- Advisory Board

Project guidance
Helps Consortium maintain an industry-relevant focus, reviews results, and provides feedback regarding prioritization of research projects.

Project teams
Provide subject matter expertise, leadership, and accountability for tasks.

- Task 2
  Core Experimental Projects
- Task 3
  Analysis and Computation
- Task 4
  Diversity, Equity, and Inclusion

Collaborations with BETO Consortia and Projects

Target compounds	Separation challenges	Modeling insight
Agile BioFoundry BOTTLE Co-Optima Lignin Utilization	ChemCatBio Lignin-First Biorefinery Development Strategies for Co-processing in Refineries	Consortium for Computational Physics and Chemistry Data, Modeling, and Analysis Program

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BIOPROCESSING SEPARATIONS CONSORTIUM

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Technology solutions through new separation materials, processes, and modeling-based insights

FY23 Research Project Portfolio

		Separation technology	Stream	Separation target(s)	BETO Collaborations	Industry collaborations
Experimental	**Adsorption Based ISPR for ABF Products**	Adsorption	Fermentation broth	Muconic, β-ketoadipatic, aconitic, 3-HP, and xylonic acids	Agile BioFoundry	Fermworx, Compound Solutions, SEMBA biosciences, and upfront chromatography
	Co-optimization of Scalable Membrane Separation Processes and Materials	Nanofiltration / reverse osmosis	HTL or pyrolysis oil, upgraded product streams	Hydrocarbons and oxygenates	Biochemical and thermochemical processing,	Evonik, Exxon Mobil, ADM, Sulzer, ICM, and Virent
	Continuous Counter Current Chromatography	Counter current chromatography	RCF oil	C_7-C_{16} SAF range molecules	Lignin Utilization, Lignin to SAF project, Lignin-First Biorefinery Development	Dynamic extractions, Ubiquity biosciences, Lignolix
	Diol Separations	Membrane-assisted liquid-liquid extraction, Microfiltration membrane, Wiped-film evaporation	Fermentation broth	2,3-butanediol, ethylene glycol	Biochem, ChemCatBio, BOTTLE	Pall Corporation, Catalyxx, Honeywell, Filtration Solutions, and Sironix, Google X
	Electrochemical Separation Technologies to Extract Intermediate Organic Compounds	Shock-wave electrodialysis, Capacitive deionization	Fermentation broth	C_5-C_8, e.g., muconic, β-ketoadipic, aconitic acid, itaconic acid, mevalonic acid	Agile BioFoundry	Visolis
	Enabling SAF Production by Adsorptive Denitrogenation (ADN)	Adsorption	HTL biocrude	Nitrogen-containing species	PNNL Hydrothermal PDUs, Strategies for Co-processing in Refineries	Kern Energy, Ecolube
	Volatile Product Recovery	Adsorption	Fermenter off-gas	Aromatics (3-methyl anisole), alcohols (isoprenol) and their esters, and terpenes (cineole, etc.)	Agile BioFoundry, Co-Optima, BOTTLE, SAF strains/processes	Praj Matrix, Amyris, LanzaTech, Rho Renewables, iMicrobes, Dow/DuPont, and the U.S. Navy
Analysis & Computation	**Computational Studies Supporting Experimental Designs**				Consortium for Computational Physics and Chemistry	
	Cross-cutting Analysis: Core TEA and LCA				Cross-cutting	

Challenges

- Bioprocessing separations challenges are diverse and wide-ranging
  - We have developed a method to screen and prioritize multiple possible projects
- Baselines are not always available; process models and accompanying economic and sustainability analyses must be developed
  - Analysis and experimental researchers work in conjunction to establish clear baselines, followed by detailed modeling and analysis
- Staying up-to-date with separations challenges
  - The Consortium's Advisory Board and participation in and leading of conference sessions keeps Consortium members up-to-date with separations challenges
  - Input from and interaction with other BETO-funded projects inform knowledge of challenges

Go/No-Go Decisions

- Is the problem relevant to industry and BETO stakeholders?
- Does the Consortium have the expertise and technique(s) to tackle the problem?
- Does internal analysis suggest the Consortium is making meaningful progress?
- Is the project making meaningful technical progress?
- Does the Consortium have sufficient funding to continue the project?

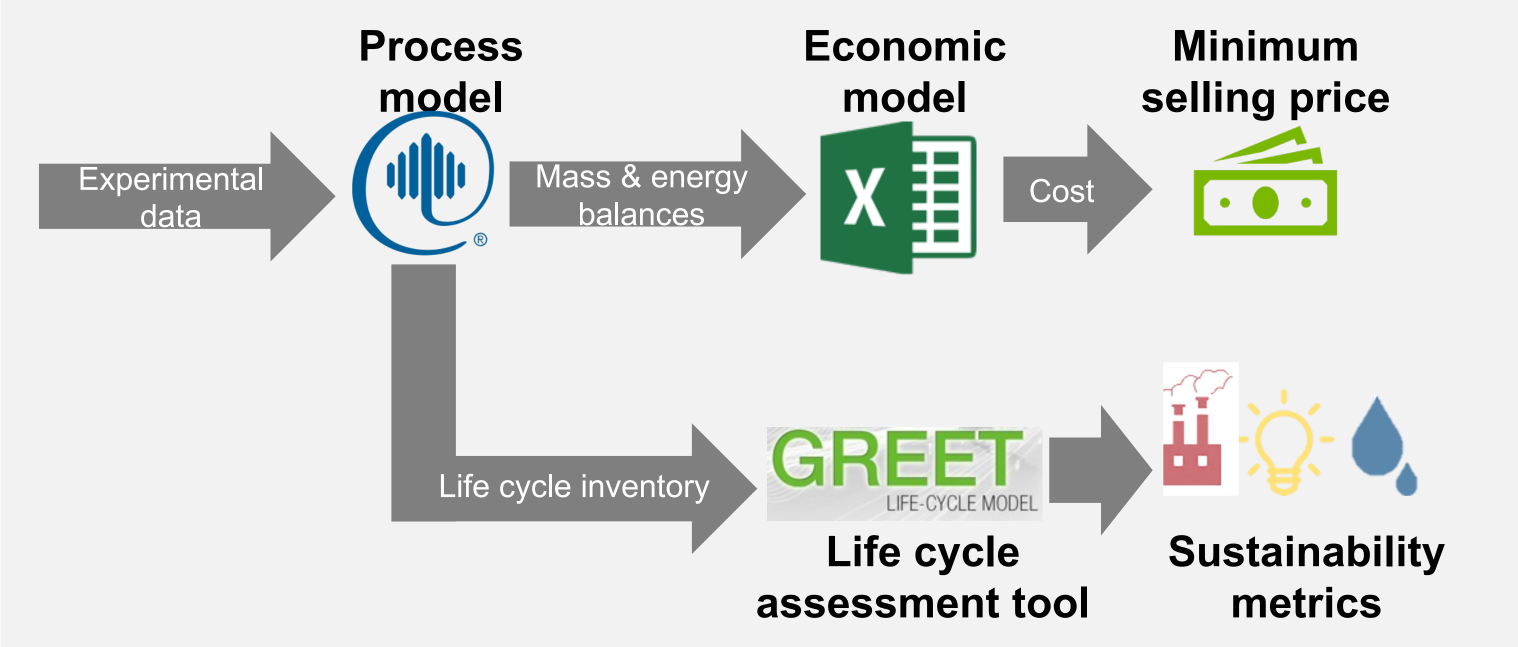
If YES, continue the project.

If NO, discontinue project.

Approach to Integrated Analysis

Assess technical, economic, & environmental feasibility of bioproduct/biofuel conversion processes

- Detailed process analysis with rigorous mass and energy balances
- Identify data needs and further R&D need to improve overall cost and efficiency
- Assess environmental impacts (greenhouse gas emissions, fossil fuel consumption, and water consumption)
- Approach is consistent with other BETO-sponsored analyses



Experimental and Analysis Task Plans

Guide consistent analysis across Consortium, transparent workflow for both experimental and analysis, and interdependencies

Key Elements

- Technology description
- Consortium R&D focus
- Biorefinery level block diagram
- Methodology/approach for techno-economic, life cycle, and computational analysis
- Existing process, economic, and life cycle assessment models
- Baseline comparison technology
- Unknowns and data gaps
- Task schedule

Important Points

- Plans are iterative and require ongoing communication among experimental and analysis teams
- Techno-economic analysis (TEA) and life cycle assessment (LCA) are independent of experimental work to provide objective evaluations

Approach

Risks and Mitigation

Risk	Mitigation
Tasks not coordinated, duplicative, or not leveraging advances elsewhere in Consortium	Task kickoff meetings, monthly Consortium calls, and regular task calls. Example: TEA/LCA team meets biweekly
Tasks not on progress towards milestones; task interdependencies cause unforeseen slow-downs	Task plans used to monitor interdependencies and progress towards milestones
Technical targets don't account for economic viability and/or sustainability	Cross-cutting analysis team engages with each R&D team to inform technical targets and economic viability and sustainability using consistent baselines and methods
Tasks use different input streams leading to results that can't be compared	Exemplary streams used across tasks
Team lacks access to project management documents	Box folder used for file sharing
Limited interactions with outside stakeholders constrains innovation	External facing website (bioesep.org), external events, and interaction with Advisory Board keep Consortium up-to-date on separations challenges

Approach

Advisory Board

Helps the Consortium maintain an industry-relevant focus and knowledge of recent technology advances and challenges

Roles and responsibilities

- Participate in twice-yearly in-person Consortium meetings
- Provide guidance on Consortium projects/technologies
- Share expertise on emerging technology or scientific advances
- Review progress in comparison with work plans
- Provide input on separation opportunities and prioritization of research projects

New additions in FY23

FY23 Approach to Diversity, Equity and Inclusion

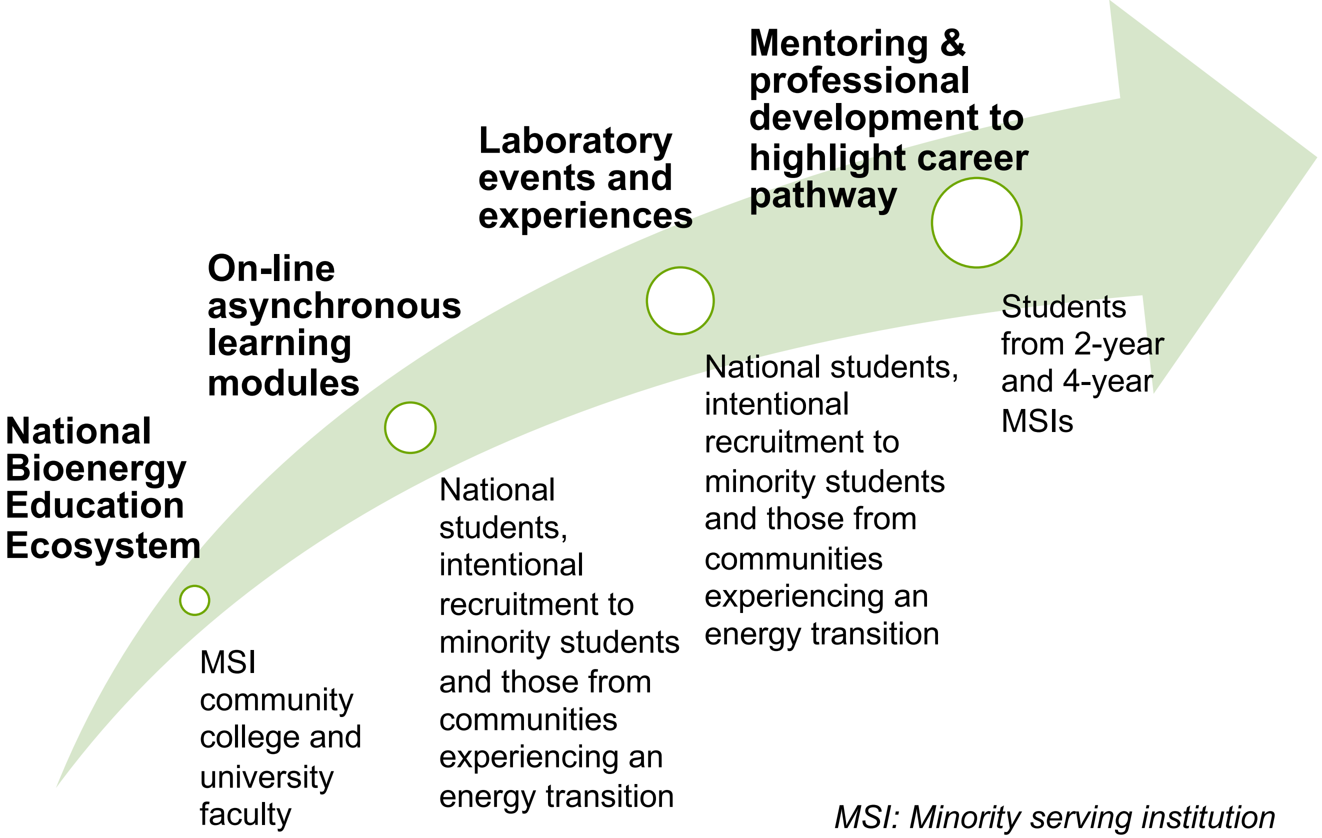
Investing in the Future Bioenergy Leaders

Challenge: lack of trained talent, limited student interest in bioenergy, and lack of curricular resources that showcase bioenergy careers as their barriers

Approach: Create a national program that brings together academia (including community colleges) and provides a curricular backbone, while highlighting DOE research and bioenergy careers

Goal: Increase the diversity and number of students seeking educational and career opportunities in bioenergy

Target Audience: Community college and undergraduate students from under-resourced communities and/or within traditionally underrepresented groups in STEM



MSI: Minority serving institution

Bioenergy Bridge to Career Program

Week-long virtual summer workshop (FY23)

- Highlight Consortium research
- Focus on the skills and professional development needed to pursue a career in bioenergy

Week-long in-person summer workshop (FY24 and FY25)

- Run simultaneously at 3 of the labs
- Focus on the skills and professional development needed to pursue a career in bioenergy

Hybrid mentorship program (FY24 and FY25)

- Focusing on networking, professional development, and bioenergy
- Tropics will include presentations by stakeholders from industry, the community, and Consortium researchers

Project Management and Integration

Objectives	Provide Consortium guidance by leading discussions on technical direction, external engagement Monitor progress and impact by evaluating milestone status and risks affecting projects in the portfolio Coordinate external communications, including interactions with Advisory Board and external website Manage Consortium business including reporting and monthly conference calls
Accomplishments	Coordinated interactions with Advisory Board Hosted listening day and symposium at ACS Green Chemistry & Engineering Conference Maintained website to communicate Consortium capabilities and progress Managed Consortium reporting and monthly communications Led development of project highlight documents (FY22 Q4 annual milestone)

FY22 Consortium Technical Accomplishments

Technology	BETO Collabor-ative Project	New Capability Development	Lignin fractionation	Process intensification	Dilute C recovery	Inhibitor removal	Materials	Processes	Analysis & computation	
			Challenges addressed				Capabilities applied			
Tangential flow filtration	■		■		■			■	■	
Rotating ceramic disk filtration	■		■		■			■	■	
Electrodeionization	■		■	■	■	■	■	■	■	
Capacitive deionization	■	■		■	■		■	■	■	
Polymeric membranes	■		■		■		■	■	■	■
Ceramic membranes	■		■		■		■	■	■	
Pervaporation	■			■	■		■	■	■	
Wiped film evaporation	■				■			■	■	
Reactive extraction	■			■	■	■		■	■	
Membrane solvent extraction	■			■	■	■	■	■	■	■
Counter current chromatography		■	■		■			■	■	
Adsorbents	■	■		■	■		■	■	■	■

FY22 Consortium Technical Accomplishments

Completed 3 annual milestones (slides 22 and 25) to conclude the Consortium's 2nd 3-year cycle

Lignin Fractionation and Purification

- Achieved up to 35-fold increase in concentration of low molecular weight products using membranes and resin wafer electrodeionization for <$1/kg dry lignin aromatics
- Worked with 3 industrial partners to conduct pilot-scale field demonstrations and advance separation material development

Redox-Based Electrochemical Separations

- Demonstrated electrically driven ion adsorption and recovery for butyrate
- Designed and developed a customized program for system control and data logging, enabling automated experimentation
- Identified electrode capacity and energy consumption as critical cost drivers

2,3-Butanediol Separations

- Revised technical approach to investigate 3 new non-thermal approaches to extract and recover BDO following 2021 Peer Review feedback and internal economic analysis
- Completed an initial technical and economic assessment to guide current work to a membrane liquid-liquid extraction approach
- Computationally screened extraction solvents

Note: Techno-economic analysis, life cycle assessment, and computational accomplishments are integrated within the tasks

FY22 Consortium Technical Accomplishments

Counter-Current Chromatography

- Demonstrated that individual lignin monomers can be separated using counter-current chromatography
- Showed 10x energy reduction relative to baseline and provided >25 g monomers to Lignin Utilization project

Volatile Products Recovery

- Demonstrated reuse over >100 cycles and that inexpensive cartridge designs transfer well across sites and scales in pilot studies
- Predicted economic and environmental savings exceeding 20% when compared to baseline technology across all scenarios modeled to date
- Uncovered differences in adsorption free energies of bioproducts and water for various functional groups on the xerogel surfaces

Update Assessment of BETO Separation Challenges/Opportunities

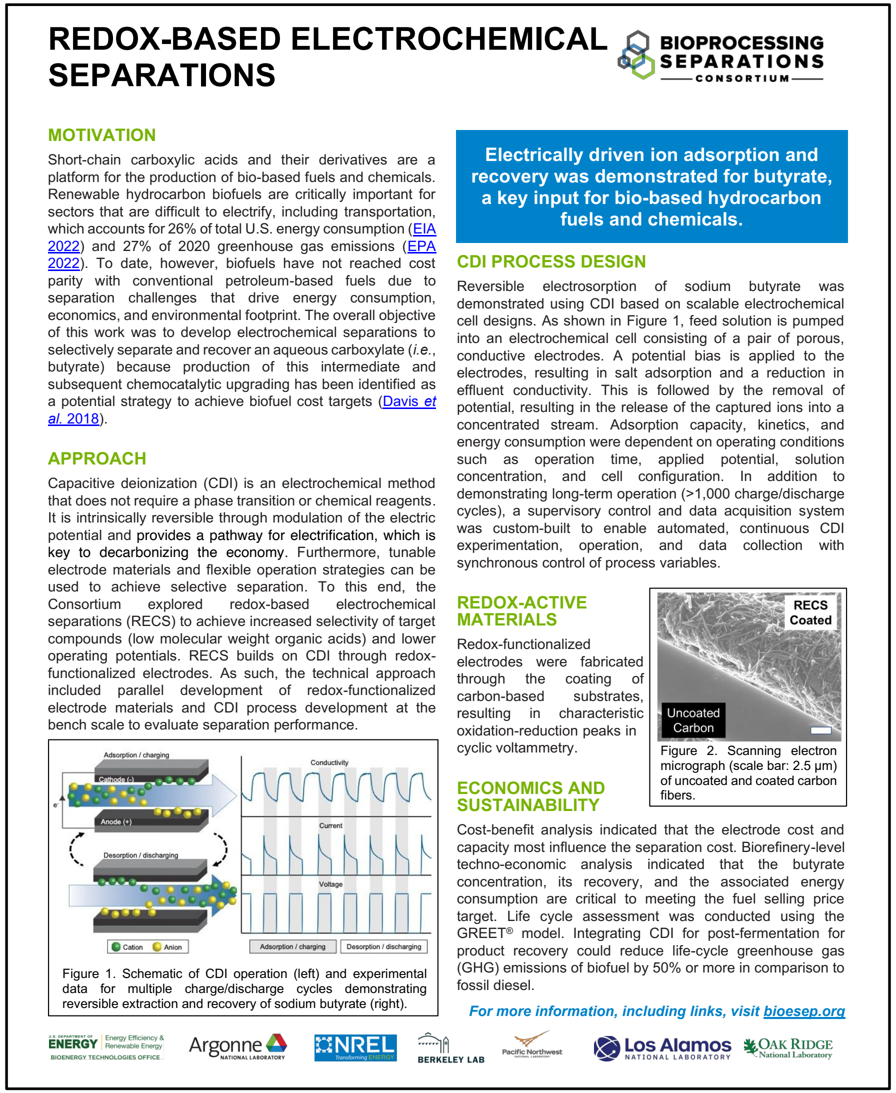
- Developed an assessment methodology to better understand the potential impacts of addressing a given separation challenge and applied the methodology to 10 different separation challenge streams from 2 different biomass conversion platforms
- Identified 3 challenge streams with maximum possible cost savings predictions >20% and associated reductions in process energy carbon intensity ranging from 0 to 54%

Note: Techno-economic analysis, life cycle assessment, and computational accomplishments are integrated within the tasks

Impact

Disseminating Results (2021–present)

- Developed 1-page highlights for each FY20-22 project including TEA, LCA, and computational work (**FY22 Q4 annual milestone** – see additional slides)
- Website content, including capabilities and publications, updated regularly
- Consortium work showcased during ACS Green Chemistry & Engineering Conference and Advanced Bioeconomy Leadership Conference (ABLC) 2022



REDOX-BASED ELECTROCHEMICAL SEPARATIONS

BIOPROCESSING SEPARATIONS CONSORTIUM

MOTIVATION

Short-chain carboxylic acids and their derivatives are a platform for the production of bio-based fuels and chemicals. Renewable hydrocarbon biofuels are critically important for sectors that are difficult to electrify, including transportation, which accounts for 26% of total U.S. energy consumption (EIA 2022) and 27% of 2020 greenhouse gas emissions (EPA 2022). To date, however, biofuels have not reached cost parity with conventional petroleum-based fuels due to separation challenges that drive energy consumption, economics, and environmental footprint. The overall objective of this work was to develop electrochemical separations to selectively separate and recover an aqueous carboxylate (*i.e.*, butyrate) because production of this intermediate and subsequent chemocatalytic upgrading has been identified as a potential strategy to achieve biofuel cost targets (Davis *et al.* 2018).

APPROACH

Capacitive deionization (CDI) is an electrochemical method that does not require a phase transition or chemical reagents. It is intrinsically reversible through modulation of the electric potential and provides a pathway for electrification, which is key to decarbonizing the economy. Furthermore, tunable electrode materials and flexible operation strategies can be used to achieve selective separation. To this end, the Consortium explored redox-based electrochemical separations (RECS) to achieve increased selectivity of target compounds (low molecular weight organic acids) and lower operating potentials. RECS builds on CDI through redox-functionalized electrodes. As such, the technical approach included parallel development of redox-functionalized electrode materials and CDI process development at the bench scale to evaluate separation performance.

Figure 1. Schematic of CDI operation (left) and experimental data for multiple charge/discharge cycles demonstrating reversible extraction and recovery of sodium butyrate (right).

Electrically driven ion adsorption and recovery was demonstrated for butyrate, a key input for bio-based hydrocarbon fuels and chemicals.

CDI PROCESS DESIGN

Reversible electrosorption of sodium butyrate was demonstrated using CDI based on scalable electrochemical cell designs. As shown in Figure 1, feed solution is pumped into an electrochemical cell consisting of a pair of porous, conductive electrodes. A potential bias is applied to the electrodes, resulting in salt adsorption and a reduction in effluent conductivity. This is followed by the removal of potential, resulting in the release of the captured ions into a concentrated stream. Adsorption capacity, kinetics, and energy consumption were dependent on operating conditions such as operation time, applied potential, solution concentration, and cell configuration. In addition to demonstrating long-term operation (>1,000 charge/discharge cycles), a supervisory control and data acquisition system was custom-built to enable automated, continuous CDI experimentation, operation, and data collection with synchronous control of process variables.

REDOX-ACTIVE MATERIALS

Redox-functionalized electrodes were fabricated through the coating of carbon-based substrates, resulting in characteristic oxidation-reduction peaks in cyclic voltammetry.

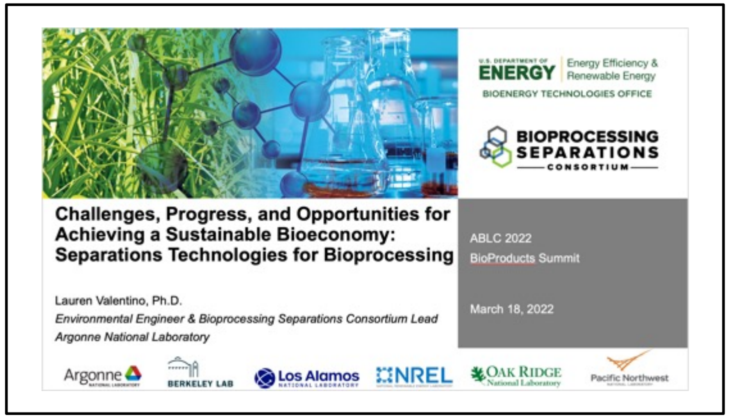
Figure 2. Scanning electron micrograph (scale bar: 2.5 μm) of uncoated and coated carbon fibers.

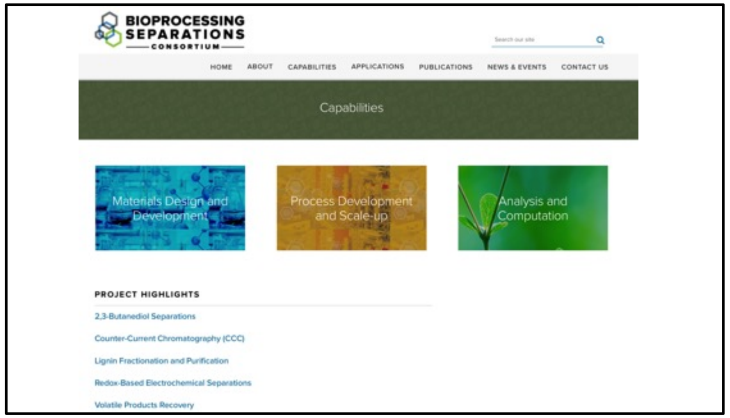
ECONOMICS AND SUSTAINABILITY

Cost-benefit analysis indicated that the electrode cost and capacity most influence the separation cost. Biorefinery-level techno-economic analysis indicated that the butyrate concentration, its recovery, and the associated energy consumption are critical to meeting the fuel selling price target. Life cycle assessment was conducted using the GREET® model. Integrating CDI for post-fermentation for product recovery could reduce life-cycle greenhouse gas (GHG) emissions of biofuel by 50% or more in comparison to fossil diesel.

For more information, including links, visit bioesep.org

U.S. DEPARTMENT OF ENERGY Energy Efficiency & Renewable Energy BIOENERGY TECHNOLOGIES OFFICE · Argonne NATIONAL LABORATORY · NREL · BERKELEY LAB · Pacific Northwest · Los Alamos NATIONAL LABORATORY · OAK RIDGE National Laboratory





Impact

Publications and IP (2021–present)

- 17 journal publications + 6 patents, patent applications, or invention disclosures filed
- Separation technologies: adsorption, extraction-distillation, crystallization, membrane, capacitive deionization, electrodeionization, ultrasonic, and chromatography
- Separation applications: Product separation and recovery, new material development, process intensification
- Cross-cutting TEA, LCA, and computational modeling
- Additional journal articles and patents in progress

Green Chemistry — PAPER

Energy and techno-economic analysis of bio-based carboxylic acid recovery by adsorption†

Patrick O. Saboe, Lorenz P. Manker,‡ Hanna R. Monroe, William E. Michener, Stefan Haugen, Eric C. D. Tan, Ryan L. Prestangen, Gregg T. Beckham and Eric M. Karp*

Experiment + TEA

ACS Sustainable Chemistry & Engineering — Research Article

Computational and Experimental Study for the Denitrification of Biomass-Derived Hydrothermal Liquefaction Oil

Pradeep Kumar Gurunathan, Difan Zhang, Vassiliki-Alexandra Glezakou,* Roger Rousseau, Huamin Wang, Aimee Lu Church, William Beatrez, Michael Z. Hu, and Suh-Jane Lee

Experiment + computation, cross-institutional

frontiers | Frontiers in Sustainability — Original Research, Published 23 November 2022, DOI 10.3389/frsus.2022.1056580

Methodology for assessing the maximum potential impact of separations opportunities in industrial processes

Jian Liu[1]*, Jacob W. Dempsey[2], Shuyun Li[1], Yuan Jiang[1], Lesley J. Snowden-Swan[1], William L. Kubic[3], Eric C. D. Tan[2]* and Charles J. Freeman[1]*

TEA, cross-institutional

ACS Sustainable Chemistry & Engineering — Research Article

Integrated Ion-Exchange Membrane Resin Wafer Assemblies for Aromatic Organic Acid Separations Using Electrodeionization

Matthew L. Jordan, Grzegorz Kokoszka, Hishara Keshani Gallage Dona, Dodangodage Ishara Senadheera, Revati Kumar,* Yupo J. Lin,* and Christopher G. Arges*

I&EC research — Article

Fractionation of Lignin Streams Using Tangential Flow Filtration

Jipeng Yan, Eric C. D. Tan, Rui Katahira, Todd R. Pray, and Ning Sun*

Separation and Purification Technology 285 (2022) 120330 — ELSEVIER

The cell utilized partitioning model as a predictive tool for optimizing counter-current chromatography processes

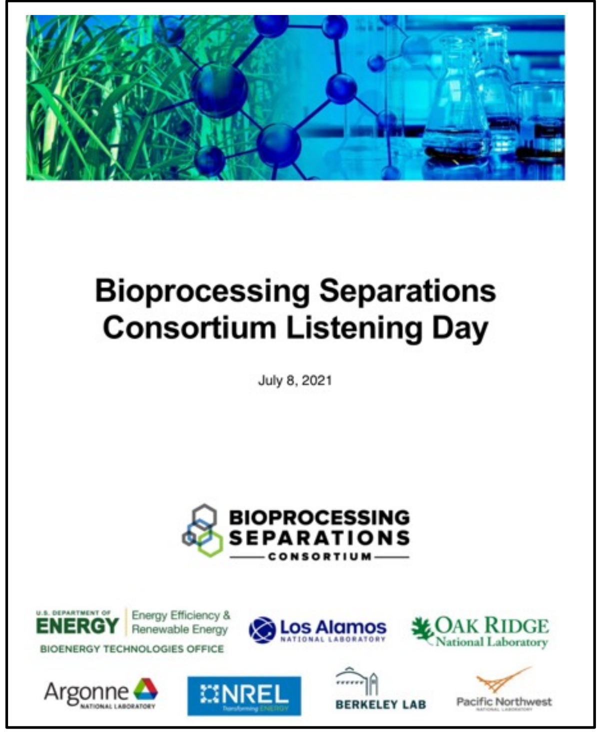
Hoon Choi, Nathan E. Soland, Marisa R. Moss, Jian Liu, Ryan R. Prestangen, Rui Katahira, Suh-Jane Lee, Michael R. Thorson, Charles J. Freeman, Eric M. Karp

Experiment + modeling, cross-institutional

Impact

Outreach (2021–present)

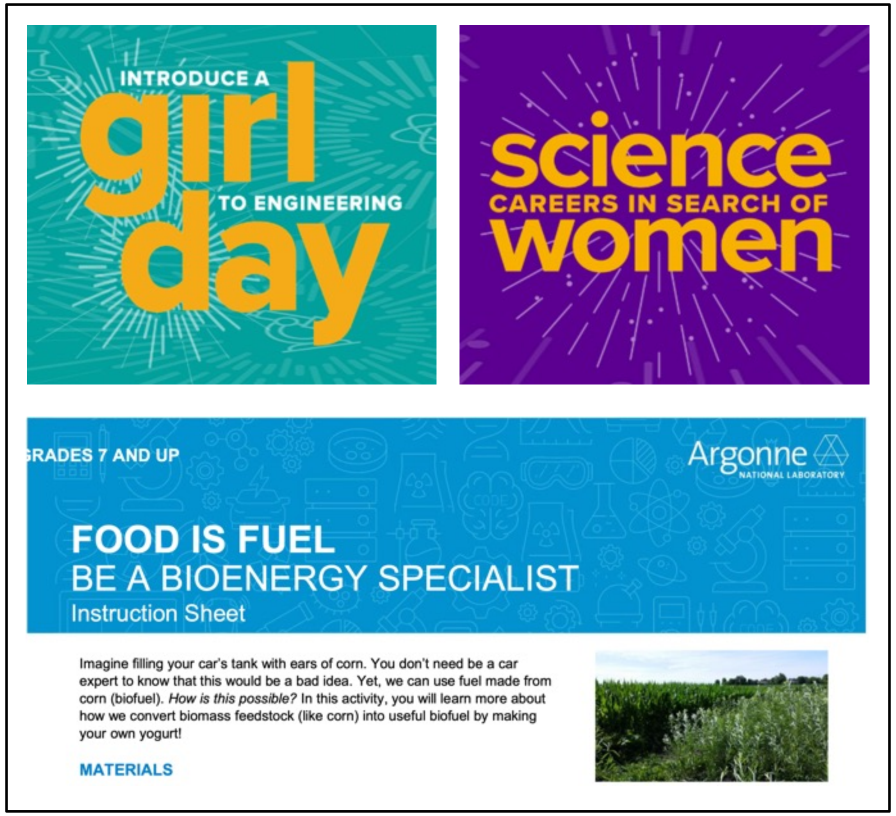
- 25th Annual Green Chemistry & Engineering Conference
  - Organized technical session (Bioprocessing Separations: Advancing a Research Agenda) + panel discussions
  - 90 participants in technical sessions and 50+ participants in panel discussions
  - Participants from industry, academia, and government from 13 countries
- Separations Consortium Listening Day
  - 110+ attendees from industry, academia, National Laboratories, and other government agencies
  - 11 technical talks that framed breakout discussions to identify technical opportunities, explore knowledge gaps, and prioritize separations challenges for bioprocessing separations
- Resultant stakeholder input was used to inform the Consortium's multi-year R&D plan



Report available for download at bioesep.org

Impact

FY22 DEI Activities



Research team

- Diverse team including scientists and engineers from STEM underrepresented groups

Outreach activities

- 15 total – meeting criteria for **FY22 Q4 annual milestone**
- Introduce a Girl to Engineering Day and Science Careers in Search of Women
- Lab tours for Argonne in Chicago and at ABPDU
- Educational Programs and Outreach newsletter with at home separations activity for students in grade 7+
- Lectures/seminars at Northwestern University, UC Berkeley, Columbia Basin College, Washington State University, and Chiawana High School
- Bioenergy STEM chats with 5th grade students

Student mentoring

- 8 undergraduate student internships – meeting criteria for **FY22 Q4 annual milestone**

Summary

	Project Management and Integration	Diversity, Equity, and Inclusion
Approach	Select Consortium projects based on understanding of BETO and industry needs Use economic and sustainability analysis as guiding tools in project evaluation and selection Interact with internal and external stakeholders to achieve impactful project portfolio and disseminate results	Include individuals from underrepresented groups Leverage ongoing diversity programs and initiatives at each National Lab to provide student internships Participate in evidence-based diversity-focused education and outreach programs
Progress	Met biannually with Advisory Board, organized ACS Green Chemistry & Engineering session, and hosted listening day Released project highlights for FY20-22 Coordinated intra-Consortium planning, communications, and reporting	Participated in 15 outreach events and hosted 8 undergraduate students in FY22 Initiated Bioenergy Bridge to Career Program with virtual workshop planned for summer 2023
Impact	Provide leadership to Consortium and support task leads in their accomplishments of objectives Disseminate results through publications, presentations, and outreach to other stakeholders New materials and processes to lower bioprocessing costs and life cycle impacts	Introduce students to key topics, knowledge, and skills related to the field of bioenergy Provide undergraduate students opportunities to participate in professional development Broaden participation of underrepresented minorities in STEM within the bioenergy community

Consortium Quad Chart

Timeline

- Project start date: October 2022
- Project end date: September 2025

	FY22 Costed	FY23-25 Total Award
DOE Funding	$3,625K ANL: $1,040K LANL: $212K LBNL: $390K NREL: $998K ORNL: $370K PNNL: $615K	$15,000K ANL: $5,196K LANL: $406.8K LBNL: $1,576.8K NREL: $3,598.8K ORNL: $1,936.8K PNNL: $2,284.8K
Project Cost Share *	N/A	N/A

TRL at Project Start: Varies by task
TRL at Project End: Varies by task

Project Goal

Develop separation technologies that enable end-to-end bio-processes for at least 5 products or fuels that decrease bioprocessing costs while meeting >70% reduction in GHG emissions compared to a petroleum baseline

End of Project Milestone

Technical goals will be covered in each task presentation.

The overall end of project milestone is to demonstrate the Consortium's value to BETO and the biofuel and bioproduct communities through documentation of technical advances, influence on process economics, and potential industrial applications of Consortium technologies.

Project Partners

- ANL
- LANL
- LBNL
- NREL
- ORNL
- PNNL

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U.S. DEPARTMENT OF ENERGY | Energy Efficiency & Renewable Energy
BIOENERGY TECHNOLOGIES OFFICE

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- Anthony Sorbera

BERKELEY LAB

- Ning Sun
- Eric Sundstrom
- Colette Flood
- Julio Jaramillo
- Laleh Cote

Los Alamos NATIONAL LABORATORY

- Taraka Dale
- Bill Kubic
- Nicole Holzclaw-Stone

Argonne NATIONAL LABORATORY

- Jennifer Dunn
- Phil Laible
- Yupo Lin
- Thathiana Benavides
- Meltem Urgun-Demirtas
- Yuepeng Zhang
- Bethany Frank

NREL NATIONAL RENEWABLE ENERGY LABORATORY

- Gregg Beckham
- Eric Karp (former)
- Eric Tan
- Jacob Dempsey
- Hakan Olcay
- Jahi Simbai

OAK RIDGE National Laboratory

- Ramesh Bhave
- Syed Islam
- Vanda Glezakou
- Andy Sutton
- Josh Thompson
- Aimee Church (former)
- Ja'Wanda Grant

Pacific Northwest NATIONAL LABORATORY

- Mike Thorson
- Jian Liu
- Difan Zhang
- Miki Santosa
- Margarita Magana
- Evangelina Shreeve
- Ann Wright-Mockler

Thank you!

Additional Slides

Responses to Previous Reviewers' Comments

A better survey / outreach to the private sector (and academia) among those providing technical services in separations might encourage a broader set of resources. May want to add other equipment manufacturers to the Industrial Advisory Group to enable faster adoption in application areas even beyond the biorefinery goals of the consortium.

In June/July 2021, the Consortium organized a special session at the ACS Green Chemistry and Engineering Conference and hosted a virtual listening day workshop to engage experts from industry, research institutions, academia, nonprofit organizations, national laboratories, and other government agencies.

We agree that it is essential to have Board members who can provide input on a number of topics including equipment and processes that may be relevant to addressing challenges in bioprocessing separations. While most experts on our Board represent companies that produce biofuels/bioproducts and advise us on the main technical challenges they face, we do have equipment and materials developers on the Board. We are also in the process of recruiting and refreshing our Advisory Board to align with the Consortium's FY23 portfolio.

The website (bioesep.org) seems lean to capture a viewer/visitor. Are there restrictions in posting more content, e.g., links to critical references (publications, reviews, patents and talks)?

We have updated our website to expand the resources to which it serves as a gateway. This includes the Consortium's capabilities, publications, and news, which are updated regularly.

FY22 Consortium Technology Highlights

Technology			Challenges addressed				Capabilities applied			
	BETO Collabor-ative Project	New Capability Development	Lignin fractionation	Process intensification	Dilute C recovery	Inhibitor removal	Materials	Processes	Analysis & computation	
Tangential flow filtration										
Rotating ceramic disk filtration										
Electrodeionization										
Capacitive deionization										
Polymeric membranes										
Ceramic membranes										
Pervaporation										
Wiped film evaporation										
Reactive extraction										
Membrane solvent extraction										
Counter current chromatography										
Adsorbents										

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Demonstrated re-use over >100 cycles; inexpensive cartridge designs transfer well across sites and scales in pilot studies

FY22 Project Highlight

LIGNIN FRACTIONATION AND PURIFICATION

BIOPROCESSING SEPARATIONS CONSORTIUM

OPPORTUNITIES AND CHALLENGES

Lignin is an abundant renewable resource that is a promising substrate for upgrading to biofuels and biochemicals. Depolymerized lignin is a chemically complex stream that consists of aromatic monomers and high-molecular-weight (HMW) oligomers. Regardless of the upstream deconstruction process, separation of monomers and oligomers is a major challenge and opportunity for improved biorefining, given the importance of lignin valorization in lignocellulosic biorefining. Work in the Separations Consortium used an exemplary lignin-rich stream, namely alkaline pretreated liquor (APL), with (1) ceramic membranes to separate the low-molecular-weight (LMW) from the HMW fraction, (2) resin wafer electrodeionization (RW-EDI) to isolate LMW organic acids from APL, and (3) cascading tangential flow filtration (TFF) with ceramic membranes.

MEMBRANE SEPARATIONS

We demonstrated a membrane process to separate APL into HMW and LMW fractions by applying microfiltration (MF) and nanofiltration (NF), or separately, TFF. We characterized the permeance and selectivity of ceramic membranes. The chemical composition, molecular weight distribution, and carbon content of the APL fractions were evaluated to show the impact of filtration. The developed process exhibits up to 98.5% rejection of HMW (MW>1,000 Daltons) species and generates a permeate stream with >80% recovery of LMW lignin-related compounds, including aromatic species such as

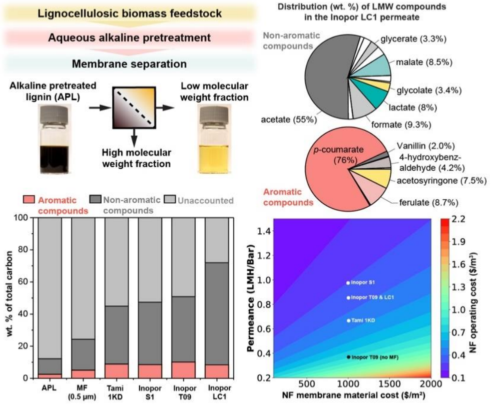

Figure 1. Comparison of separation performance of several membrane options and the resulting LMW distribution in the permeate.

Lignin monomer-oligomer separations targeted >90% selectivity. Membranes rejected up to 98.5% of HMW lignin. Up to a 35-fold increase in concentration of LMW products achieved with membranes and RW-EDI.	Y. Lin *et al.* (2023), *ACS Sus. Chem. & Eng.* P.O. Saboe *et al.* (2022), *Green Chem.* J. Yan *et al.* (2022), *Ind. Eng. Chem. Res.*

p-coumarate and ferulate, resulting in a 6-fold enrichment in LMW organic compounds relative to the crude APL (Figure 1). A report of this work was published in *Green Chemistry.*

TECHNO-ECONOMIC ANALYSIS AND MODELING OF MEMBRANE PROCESSES

A continuous process model was developed to estimate stream flowrates, compositions, and LMW compound recoveries for a two-stage filtration process. Briefly, we used the irreversible thermodynamic model adapted to a multicomponent NF process. The model was developed in Python, and the code is available on GitHub. We used this process model to conduct a TEA to elucidate cost and performance drivers for APL fractionation (Figure 1). Similar TEA was conducted for the cascading TFF study as well.

MEMBRANE-WAFER-ASSEMBLY ELECTRODEIONIZATION (MWA-EDI)

To overcome the high transfer resistance of commercial ion-conductive membranes for aromatic acids, *i.e.*, *p*-coumarate and ferulate, a new MWA material was manufactured. We successfully developed the MWA-EDI device and demonstrated an enhancement of aromatic acids capture ratio along with significant reduction of energy consumption and processing cost, as shown in Figure 2.

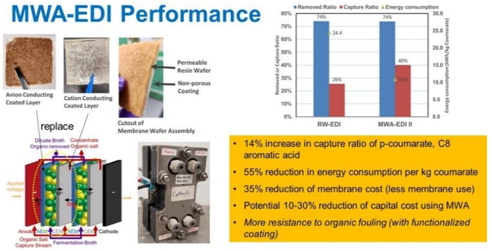

Figure 2. Comparison of separation performance by RW-EDI and MWA-EDI technologies.

For more information, including links, visit bioesep.org

FY22 Project Highlight

REDOX-BASED ELECTROCHEMICAL SEPARATIONS

BIOPROCESSING SEPARATIONS CONSORTIUM

MOTIVATION

Short-chain carboxylic acids and their derivatives are a platform for the production of bio-based fuels and chemicals. Renewable hydrocarbon biofuels are critically important for sectors that are difficult to electrify, including transportation, which accounts for 26% of total U.S. energy consumption (EIA 2022) and 27% of 2020 greenhouse gas emissions (EPA 2022). To date, however, biofuels have not reached cost parity with conventional petroleum-based fuels due to separation challenges that drive energy consumption, economics, and environmental footprint. The overall objective of this work was to develop electrochemical separations to selectively separate and recover an aqueous carboxylate (*i.e.*, butyrate) because production of this intermediate and subsequent chemocatalytic upgrading has been identified as a potential strategy to achieve biofuel cost targets (Davis *et al.* 2018).

APPROACH

Capacitive deionization (CDI) is an electrochemical method that does not require a phase transition or chemical reagents. It is intrinsically reversible through modulation of the electric potential and provides a pathway for electrification, which is key to decarbonizing the economy. Furthermore, tunable electrode materials and flexible operation strategies can be used to achieve selective separation. To this end, the Consortium explored redox-based electrochemical separations (RECS) to achieve increased selectivity of target compounds (low molecular weight organic acids) and lower operating potentials. RECS builds on CDI through redox-functionalized electrodes. As such, the technical approach included parallel development of redox-functionalized electrode materials and CDI process development at the bench scale to evaluate separation performance.

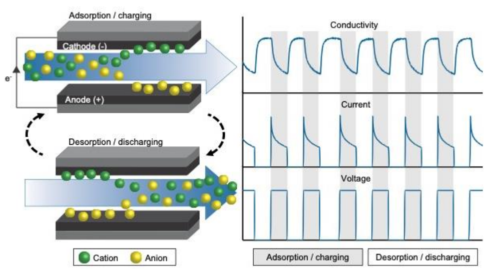

Figure 1. Schematic of CDI operation (left) and experimental data for multiple charge/discharge cycles demonstrating reversible extraction and recovery of sodium butyrate (right).

Electrically driven ion adsorption and recovery was demonstrated for butyrate, a key input for bio-based hydrocarbon fuels and chemicals.

CDI PROCESS DESIGN

Reversible electrosorption of sodium butyrate was demonstrated using CDI based on scalable electrochemical cell designs. As shown in Figure 1, feed solution is pumped into an electrochemical cell consisting of a pair of porous, conductive electrodes. A potential bias is applied to the electrodes, resulting in salt adsorption and a reduction in effluent conductivity. This is followed by the removal of potential, resulting in the release of the captured ions into a concentrated stream. Adsorption capacity, kinetics, and energy consumption were dependent on operating conditions such as operation time, applied potential, solution concentration, and cell configuration. In addition to demonstrating long-term operation (>1,000 charge/discharge cycles), a supervisory control and data acquisition system was custom-built to enable automated, continuous CDI experimentation, operation, and data collection with synchronous control of process variables.

REDOX-ACTIVE MATERIALS

Redox-functionalized electrodes were fabricated through the coating of carbon-based substrates, resulting in characteristic oxidation-reduction peaks in cyclic voltammetry.

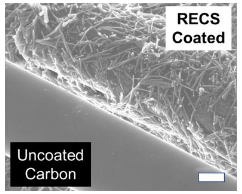

Figure 2. Scanning electron micrograph (scale bar: 2.5 μm) of uncoated and coated carbon fibers.

ECONOMICS AND SUSTAINABILITY

Cost-benefit analysis indicated that the electrode cost and capacity most influence the separation cost. Biorefinery-level techno-economic analysis indicated that the butyrate concentration, its recovery, and the associated energy consumption are critical to meeting the fuel selling price target. Life cycle assessment was conducted using the GREET® model. Integrating CDI for post-fermentation for product recovery could reduce life-cycle greenhouse gas (GHG) emissions of biofuel by 50% or more in comparison to fossil diesel.

For more information, including links, visit bioesep.org

FY22 Project Highlight

2,3-BUTANEDIOL SEPARATIONS

BACKGROUND AND GOALS

2,3-butanediol (BDO) is gaining attention in the global market as an intermediate product for several applications, such as liquid fuel or as a fuel additive. It can be produced through fermentation in low concentrations (<10 wt%). Therefore, BDO separation/concentration is required for subsequent catalytic upgrading. However, separation of 2,3-BDO poses many challenges due to its inherent characteristics, such as its high affinity with water and high boiling point. A common commercial separation/concentration technology for BDO involves evaporation and multi-stage distillation, both of which are energy intensive, and the high distillation temperatures (130-165 °C) can produce oligomers that require subsequent hydrogenation. Further, some components in the fermentation broth can inhibit or poison BDO upgrading catalysts and require removal upstream. The goal of this work was to develop low-cost and energy-efficient separation technologies, supported by techno-economic analysis (TEA), life cycle assessment (LCA), and computational modeling, to recover BDO from fermentation broth.

TECHNICAL APPROACH

The Consortium has investigated multiple approaches to recover 2,3-BDO from fermentation broth because current industrial methods, involving both distillation and hydrogenation, are energy-intensive. TEA and LCA were integrated to evaluate the tradeoffs related to separation cost, energy intensity, and sustainability of the potential approaches. Computational simulations were also integrated to provide guidance on improving the materials and processes. Initial efforts focused on a hybrid approach including pervaporation; however, TEA indicated that pervaporation could not achieve energy/cost savings in comparison to the state of technology. This motivated investigation of 3 alternative, non-thermal approaches to extract and recovery 2,3-BDO: 1) solvent extraction followed by membrane separation or evaporation and BDO upgrading to fuel, 2) reactive extraction to produce dioxolane, which can be subsequently upgraded to fuel products, and 3) an extension of (2) to include the reaction of the dioxolane back into BDO and distillation of BDO from the ketone/aldehyde. Analysis of these approaches at the both the separation process and biorefinery levels guided work to the current approach, which proposes membrane assisted liquid-liquid extraction (MLLE) to effectively and economically separate, concentrate, and purify BDO.

BDO SEPARATION AND PURIFICATION

Novel 2,3-BDO recovery approaches were investigated using conventional separatory funnels as well as a continuous liquid-liquid extraction (LLE) system with multistage, membrane-based extraction. A range of solvents (1-Butanol, oleyl alcohol, 2-Ethyl-1-hexanol, 1-Hexanol, and ethyl acetate) were used to extract BDO from an aqueous stream. A porous hydrophobic membrane was selected based on pore size, wettability, and chemical compatibility. Preliminary data showed that the membrane contactor can separate BDO from aqueous solution using hexanol in a continuous mode of operation. MLLE is an energy-efficient scalable approach that provides high surface area for contact between the organic and aqueous phase and prevents emulsion formation.

ECONOMIC AND COMPUTATIONAL MODELING

An initial technical and economic assessment for removing charged species, inorganic salts, and organic acids from BDO fermentation broth was completed. A sub-model for determining the thermodynamics and basic processing performance for the preconcentration of 2,3-BDO using multistage membrane pervaporation was also developed. This model helped guide research and development toward the membrane-assisted LLE approach. In the case of LLE, TEA results also indicated that solvent drying to remove residual water in the extraction solvent is important to realize energy savings. A computational screening of LLE solvents was performed using ab initio calculations, which were complemented using a random forest machine learning algorithm. This screening revealed that 1-Butanol and 1-Hexanol are promising candidates for solvent extraction of 2,3-BDO from water. Additionally, atomistic modeling using ab initio calculations and classical molecular dynamics suggest that sulfonation of polybenzimidazole (PBI) membranes effectively modifies the interaction between PBI polymer chains, 2,3-BDO, and water. The covalently bound sulfonated PBI (sPBI) exhibits an improved water/2,3-BDO separation selectivity, which can be experimentally synthesized by acid treatment followed by heat treatment.

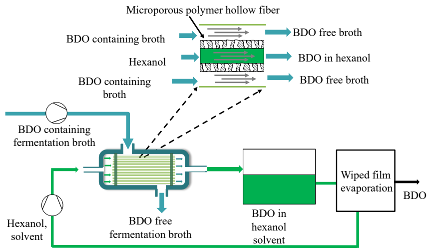

Figure 1. Schematic of BDO separation using membrane contactor system

For more information, visit bioesep.org

FY22 Project Highlight

COUNTER-CURRENT CHROMATOGRAPHY (CCC)

BIOPROCESSING SEPARATIONS CONSORTIUM

SEPARATION CHALLENGES FOR LIGNIN VALORIZATION

In biochemical conversion processes, the valorization of lignin, a plant biopolymer, has been shown to be critical for both economics and biorefinery sustainability. However, separations processes to this end have received little attention to date. Accordingly, there is an urgent need to develop low-energy separation processes to separate lignin-derived chemicals to produce target co-products and to reduce overall biorefinery costs. Lignin streams are often a complex mixture of oligomers, dimers, and monomers, requiring multiple filtration and purification processes. A novel approach for this application is counter-current chromatography (CCC), which can separate lignin monomers from lignin oils. CCC is a promising process to recover bioproducts from these complex streams because it employs two liquid phases for both the mobile and stationary phases and, therefore, does not require a solid-liquid separation. Thus, the goals of this project were to develop an energy-efficient separation process with CCC to recover lignin-derived chemicals, to demonstrate the process at scale, and to evaluate the economic feasibility of the downstream process.

CCC IS CHROMATOGRAPHY THAT USES A LIQUID STATIONARY PHASE

CCC is a dynamic liquid-liquid chromatography process where the upper or lower phases of a biphasic solvent system can be used as the stationary phase of CCC. With the unique planetary motion of CCC, hundreds to thousands of mixing and settling events can occur in a small column. As a result, lignin-derived compounds can be separated based on their different polarities and solubilities in biphasic solvent systems. CCC can handle high feed loadings and solids in the feed, enables full product recovery with various elution modes, uses inexpensive solvents for the stationary phase, and avoids resin fouling issues.

Individual lignin monomers can be separated using CCC.

The CCC process is more energy-efficient and economical than Simulated Moving Bed (SMB).

LIGNIN-DERIVED MONOMERS FROM THE LIGNIN OIL USING CCC

With CCC, lignin-derived compounds were separated from lignin oil produced from reductive catalytic fractionation (RCF). Phenolic monomers were separated from oligomers using biphasic solvent systems in CCC, with yields and purities of individual aromatic monomers, both up to 99%. Tuning the polarity of the solvent can also separate dimers from higher molecular weight compounds.

MATHEMATICAL MODEL FOR CCC OPTIMIZATION

The Cell Utilized Partition (CUP) model was developed by NREL and PNNL from basic separation (LLE) measurements. The CUP model can predict the chromatograms for various elution modes of CCC or for a general liquid-liquid chromatography process. Accordingly, the model can be used to narrow down the most viable feedstocks to target with the technology and to optimize the process for high purity, yield, and productivity. The details of the model can be found in Choi *et al.* (2022). The CUP model source codes are publicly available on GitHub.

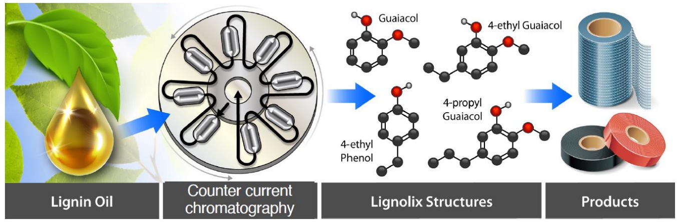

Figure 1. Overview of the downstream process using CCC. Lignin-derived chemicals are separated from lignin oil using CCC. The purified phenolic monomers can be used for high-value products.

For more information, including links, visit bioesep.org

FY22 Project Highlight

VOLATILE PRODUCTS RECOVERY

BIOPROCESSING SEPARATIONS CONSORTIUM

CONTEXT AND GOALS

A variety of high potential bioproducts are volatilized to bioreactor off-gases during aerobic fermentation. Efficient recovery of these molecules requires development of innovative, low-cost, and efficient volatile product capture strategies. Baseline approaches rely on *in situ* organic overlays or on low temperature condensation of fermenter off-gas, but these methods are costly, energy intensive, and necessitate additional dewatering or solvent recovery steps. Retention of hydrophobic products in the fermentor also increases product toxicity, reducing production rates and achievable titers. ANL researchers have developed technology to passively capture volatile products with minimal water co-capture using advanced adsorbent materials. These materials were adapted based on successful application for liquid-liquid capture within active fermentation processes. After 3 years, results from ≥3 industrial-derived SAF bioprocesses (with a 300-L scale demonstration) will highlight cost advantages of the alternative adsorptive approach and demonstrate near-quantitative bioproduct capture. This approach results in purities allowing for immediate use (≥ 95% pure SAFs or precursors), continuing broad industrial interest.

TECHNICAL APPROACH

Current recovery methods for volatile product capture utilize condensation and distillation to separate organic products from condensed vapors (Figure 1), requiring multiple heating and cooling steps. The adsorbent-based advanced materials approach works at near-ambient temperatures with tunable surfaces specifically targeting bioproducts, minimizing energy use. This approach allows the adsorbent to be placed in-line during the fermentation process and subsequently removed after collecting the product in high purity. The adsorbent can then be replaced in-line, or a second parallel system can be utilized. This allows for maximum product adsorption as the fermentation and production process can continue endlessly.

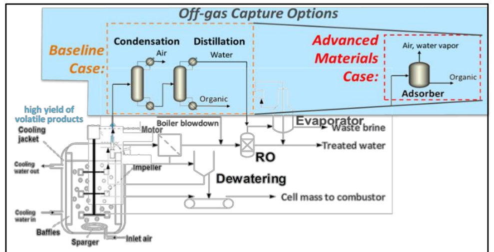

Figure 1. Volatile bioproducts from bioreactors with off-gas capture operations (blue) using the baseline technology (orange) or more efficient adsorbent-based recovery (red).

Advanced adsorbent materials enable specific recovery of volatile bioproducts from fermentor off-gas at ambient temperature with minimal water contamination.

Efficient mechanical desorption and high material longevity result in predicted cost savings of >20% when compared to conventional condensation and distillation.

ACCOMPLISHMENTS AND RESULTS

To date, advanced xerogel materials developed for liquid-liquid product capture were successfully transitioned towards capture of volatile products. They accommodate high gas flows and high moisture content, characteristic of fermentation off-gasses, with minimal pressure drop. Via scalable cartridge designs, recoveries of >83% have been achieved, even for low boiling bioproducts unrecoverable via condensation alone. These high yield recoveries are coupled with minimal water co-adsorption (typical non-specific condensation << 5%), well below the initial goal of 50% product purities. These successes were realized by increasing product-adsorbent resonance times within the advanced materials to allow for surface interactions and capture with high gas flow rates. Additionally, desorptive recovery of product from the material via low energy compression was optimized, demonstrating unwavering materials performance for over 50 adsorptive and desorptive cycles. High durability further reduces the cost of this approach, with predicted economic and environmental savings exceeding 20% when compared to baseline technology across all scenarios modeled to date. Multiple product classes are currently under investigation for coupled fermentation and off-gas capture, including collaborations with Praj Matrix and the US Navy to scale production of volatile sustainable aviation fuel precursors.

COMPUTATIONAL-GUIDED ADVANCEMENTS

Atomistic simulations aided with xerogel surface modifications for improved product capture and selectivity for volatile bioproduct recoveries from humid gas streams. Density-functional theory (DFT) uncovered differences in adsorption free energies of bioproducts and water as the functional groups on the xerogel surface are altered, *e.g.*, alkyl (C3-C8), vinyl, and phenyl groups. Classical molecular dynamics predicted the transport properties of bioproducts in xerogel models and suggest that alkyl chains >C3 (propyl to octyl) enable higher selectivity for many target compounds when compared to shorter alkyl, vinyl, and phenyl groups.

For more information, visit bioesep.org

Publication, Patents, Presentations, Awards, and Commercialization

Publications

- 1-page project highlights for FY20-22 work (https://bioesep.org/capabilities/)
  - Counter-Curent Chromatography
  - Lignin Fractionation and Purification
  - Redox-Based Electrochemical
  - Volatile Products Recovery
  - 2,3-Butanediol Separations
- Listening day report (https://bioesep.org/publications/)

Conferences

- Chaired the "Bioprocessing Separations: Advancing a Research Agenda" session at the American Chemical Society (ACS) Green Chemistry and Engineering Conference, held virtually June 18
- Valentino L. “Bioprocessing Separations Research at the National Laboratories” *ACS Green Chemistry and Engineering Conference. 14-18* June 2021. virtual
- Valentino L. “Challenges, Progress, and Opportunities for Achieving a Sustainable Bioeconomy: Separations Technologies for Bioprocessing”. *ABLC 2022*. 16-18 March 2022. Washington D.C.