Separations Consortium

2,3-Butandiol (BDO) Separations

March 11, 2021

Technology Area Session: Performance-Advantaged Bioproducts, Bioprocessing Separations, and Plastics

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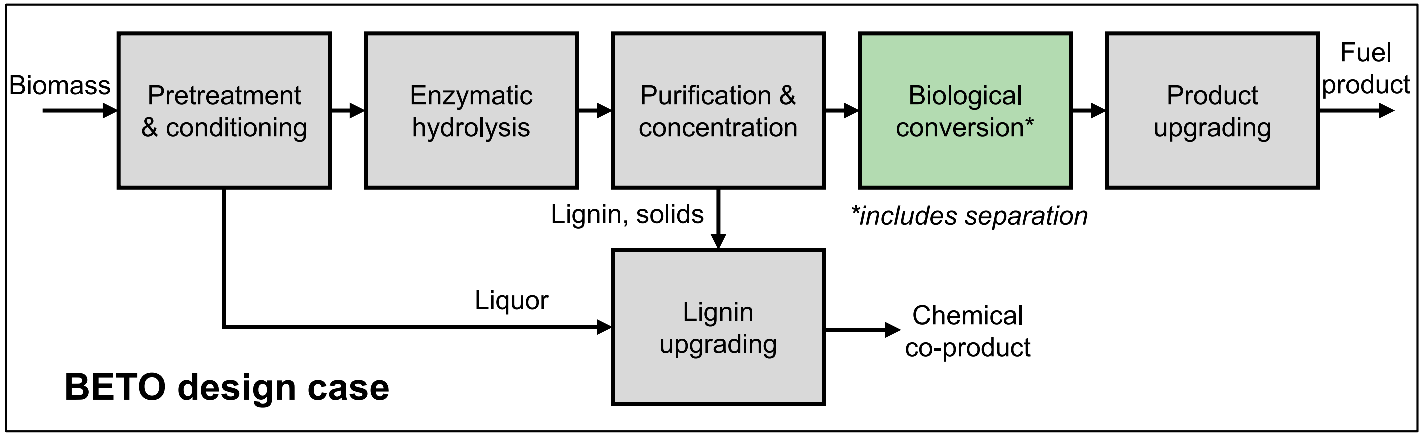
ORNL, LBNL, NREL, PNNL, ANL

Project Overview

Context

- 2,3-Butanediol (BDO, $90 million market size by 2026) in fermentation broth is diluted (8 wt%) with impurities which hinders upgrading to fuel/chemicals
- BETO design case: Anaerobic production of 2, 3 – BDO is upgraded to fuel product
- A low-cost and efficient separation technology is the key bridge to enable bio-BDO pathway



Goal: Develop less energy-intensive integrated membrane-based separations for processing dilute fermentation BDO broth into suitable feed (50 wt% BDO in water) for downstream upgrading.

State of technology: Energy intensive distillation provides 99.8 wt%1,4- butanediol (boiling point 230 °C) from fermentation as the final product, attributes 70% cost of the whole process.

Importance: Low-cost integrated membrane-based separations enable BDO enrichment from the upstream fermentation process, impurities removal, and improved techno-economics separations for scale-up of biofuels and biochemicals. This project plays a key role in achieving a $2.47/GGE by 2030 (TEA projection for the design case).

Risks: Membrane separations has not been demonstrated for diol separation in a large scale. TEA/LCA will guide materials development.

Management

BDO fermentation (NREL) → BIOPROCESSING SEPARATIONS CONSORTIUM → BDO upgrading (NREL, ORNL, PNNL, LANL) ChemCatBio Chemical Catalysis for Bioenergy

Leveraging BETO Portfolio

- Biochem - fermentation
- ChemCatBio - catalysis
- CCPC(Consortium for Computational Physics and Chemistry): material design

Experimental work integration with computation and analysis

- Experimental data (ORNL/LBNL/NREL)
- Integrated Model/Biorefinery TEA (NREL/PNNL/ORNL)
- Computation (PNNL) — CCPC Consortium for Computational Physics and Chemistry, U.S. DEPARTMENT OF ENERGY BIOENERGY TECHNOLOGIES OFFICE
- LCA (ANL)

Bioenergy Technologies Office

Steering Committee | Advisory Board

Analysis	BETO Collaborative Projects	New Capability Development
R&D-guiding TEA and LCA (E. Tan and C. Freeman)	Lignin-Rich Stream Fractionation and Purification (E. Karp)	Counter Current Chromatography (E. Karp)
Update Assessment of BETO Separation Challenges/ Opportunities (E.Tan and C. Freeman)	Redox-based Electrochemical Separations (E. Barry)	Volatile Product Recovery (P. Laible)
Computational Separations (V. Glezakou)	2,3-Butanediol Separations (A. Church)	

Project Management

- Consortium monthly webinars
- Biannual face-to-face meetings for progress review with Industrial Advisory Board (IAB)
- Biweekly meeting with computational team
- Ad hoc meetings with biochem teams and upgrading teams
- IAB input (Genomatica, Compact Membrane Systems)

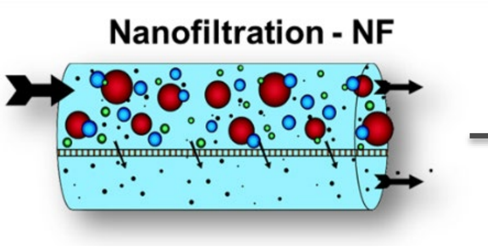
Abbreviations

- **BETO** – Bioenergy Technologies Office
- **BDO** – 2,3-butanediol
- **CCPC** – Consortium for Computational Physics and Chemistry
- **ChemCatBio** – Chemical Catalysis for Bioenergy
- **GGE** – Gasoline Gallon Equivalent
- **GO** – Graphene Oxide
- **GO-S-PCM** – Graphene Oxide Embedded Sulfonated Polymer Coated Porous Ceramic Membranes
- **IAB** – Industrial Advisory Board
- **LCA** – Life Cycle Assessment
- **MFSP** – Minimum Fuel Selling Price
- **MPV** – Membrane Pervaporation
- **NF** – Nanofiltration
- **PBI** – Polybenzimidazole
- **PCM** – Polymer Coated Porous Ceramic Membranes
- **QCM** – Quartz Crystal Microbalance
- **S-PBI** – Sulfonated Polybenzimidazole
- **S-PCM** – Sulfonated Polymer Coated porous ceramic Membranes
- **TEA** – Techno-Economic Analysis
- **WFE** –Wiped Film Evaporation

Approach

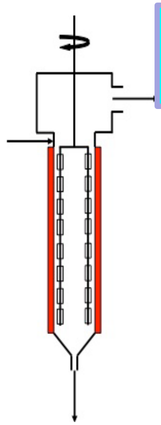
Apply novel membrane technology to separate BDO and water at lower temperatures, e.g., 80 °C

Pretreated BDO broth (NREL)



Remove bacteria, proteins, and heavy sugars

WFE= Wiped Film Evaporation (LBNL)

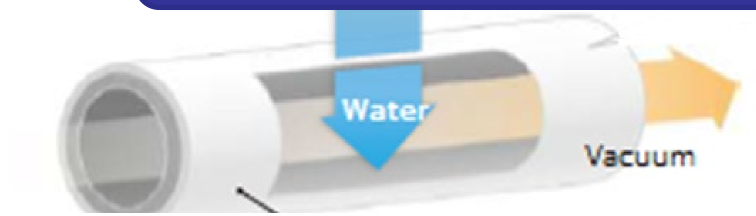


Sugars, e.g., xylitol

~20 wt% BDO in water

PCM Pervaporation (ORNL)

50 wt% - 80 wt% BDO in water



PCM=Polymer coated porous ceramic membranes (PCM)

Water is separated into the tube by passing through the polymer membrane

Final BDO concentration defined with:

Upgrading kinetics, yield, and catalyst durability

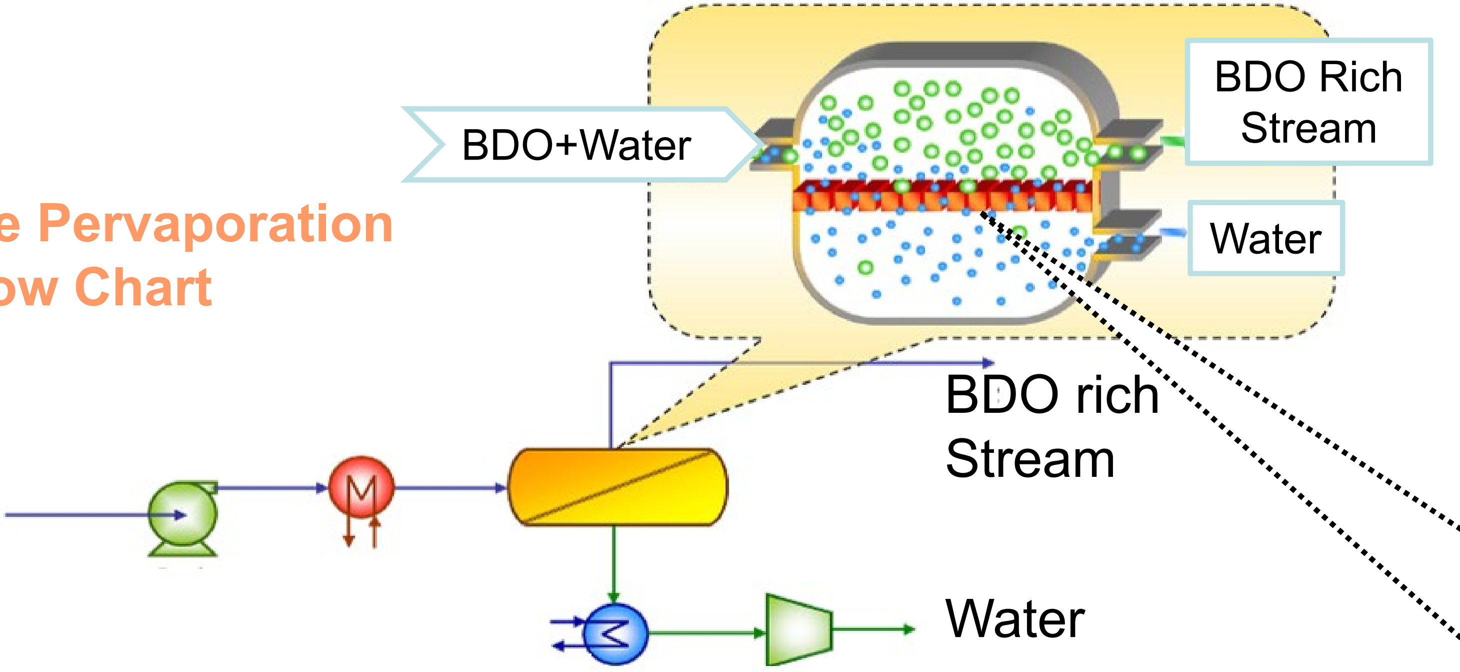
ChemCatBio
Chemical Catalysis for Bioenergy

TEA-based BDO cost target

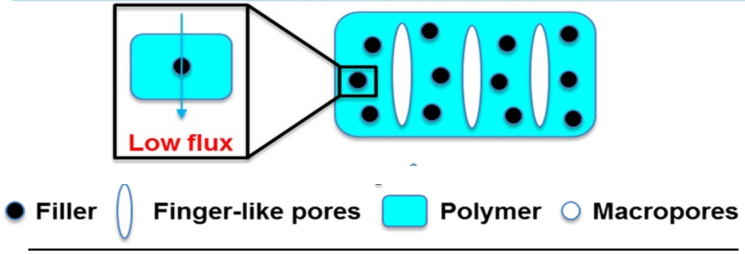
Hybrid separations targets low temperature selective removal:
Nanofiltration (NF) removes bacteria and heavy sugars;
Wiped film evaporation (WFE) can remove glycerol/salts;
And membrane pervaporation can effectively remove water and further concentrate BDO to 50 wt% - 80 wt%, depending on upgrading need and TEA cost targets .

Approach

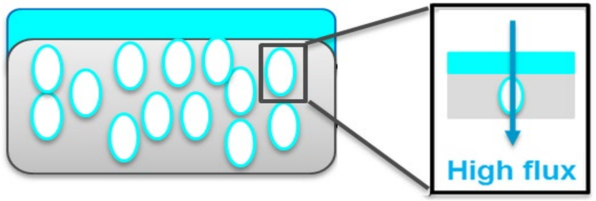
Membrane Pervaporation Flow Chart



Traditional mixed matrix membrane



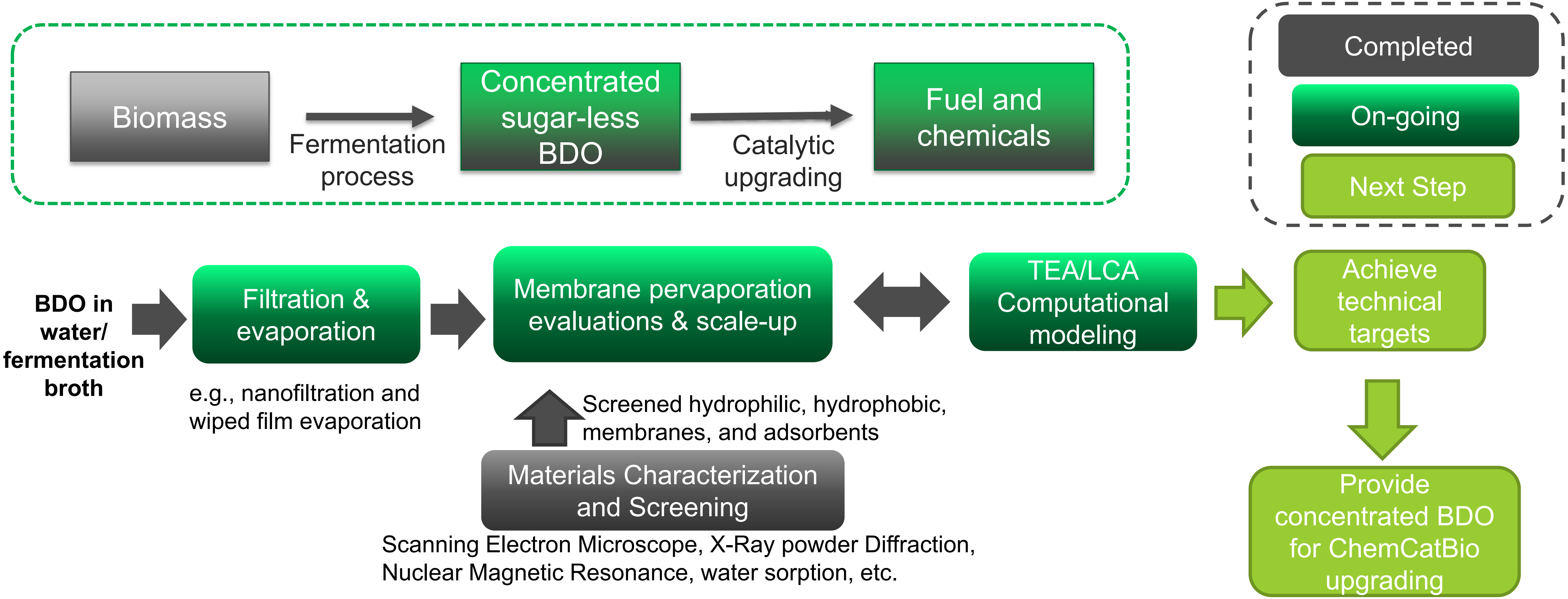
Our PCM=Polymer coated porous ceramic membranes (PCM)



- PCM pervaporation (Temp <80℃) could provide energy savings up to 50%[1], and reduce the BDO loss from 10% (evaporation) down to 3%. [2]
- The thin layer polymer coating on porous ceramic structure composes the novel scalable PCM, which provides higher flux and higher selectivity than traditional mixed matrix membrane.

1. *Van Hoof, Veerle, et al. Separation and Purification Technology 37.1 (2004): 33-49*
2. *Preliminary data, see extra slides.*

Approach



Milestone: [ORNL, LBL, NREL]: Deliver 1 L processed broth, which contains BDO > 50 wt% and sugar < 3 wt% with a durable membrane that can be run continuously for > 20 hours. Provide operating conditions, membrane material, and membrane performance data to the analysis team.	**6/30/2021**	**Quarterly Progress Measure (Regular)**

Impact

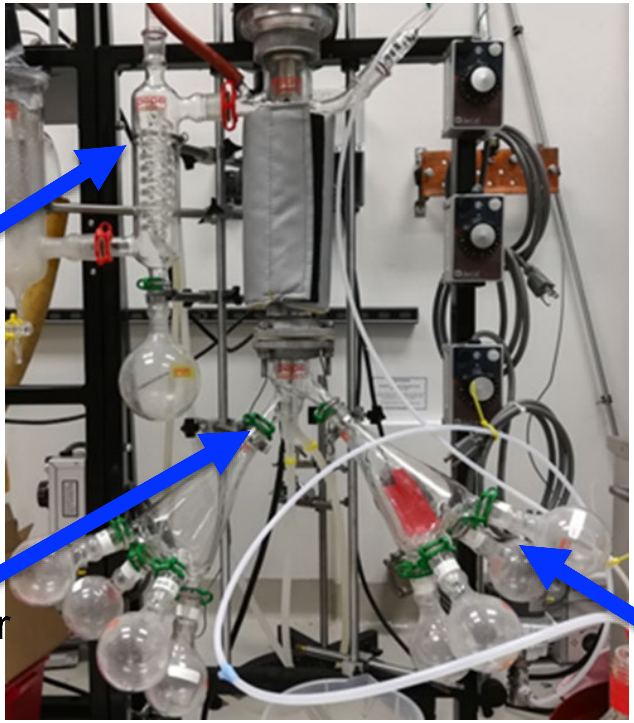
- Project directly supports BETO's mission: develop and transform biomass resources into commercially viable, refinery integrable biofuels.
- Separation project leverages other BETO BDO-centric projects, provides opportunity for:
  - Lower cost renewable fuel (TEA projects $2.47/GGE by 2030 projection for the design case)
  - Well suited for higher C (than ethanol) fuels and variety of co-products
- Integrated membrane process reduces overall separation cost, and excellent performance attributes (observed to-date) by high separation factor/flux at low temperatures, high thermal/chemical stability, and easy regeneration (water rinse).
- Polymer membrane developed readily applied to scalable modular, compact tubular systems, enables low-cost and less energy intensive separation and potentially replaces energy-intensive distillation
- Broader membrane applications
  - Diol separations in plastic deconstruction and upcycling
  - Wastewater treatment
  - Membrane reactor, e.g., Esterification, Membrane bioreactor
  - Gas separation (CO_2, N_2/O_2)
- Presentation at scientific conferences and publication in peer-reviewed journals will attract broader audiences. Ongoing discussions with industrial partners and invention disclosures will enable technology transfer.

Picture from Nanjing Tangent Fluid Technology Co., Ltd (example commercial system that BDO separation membrane could be applied in)

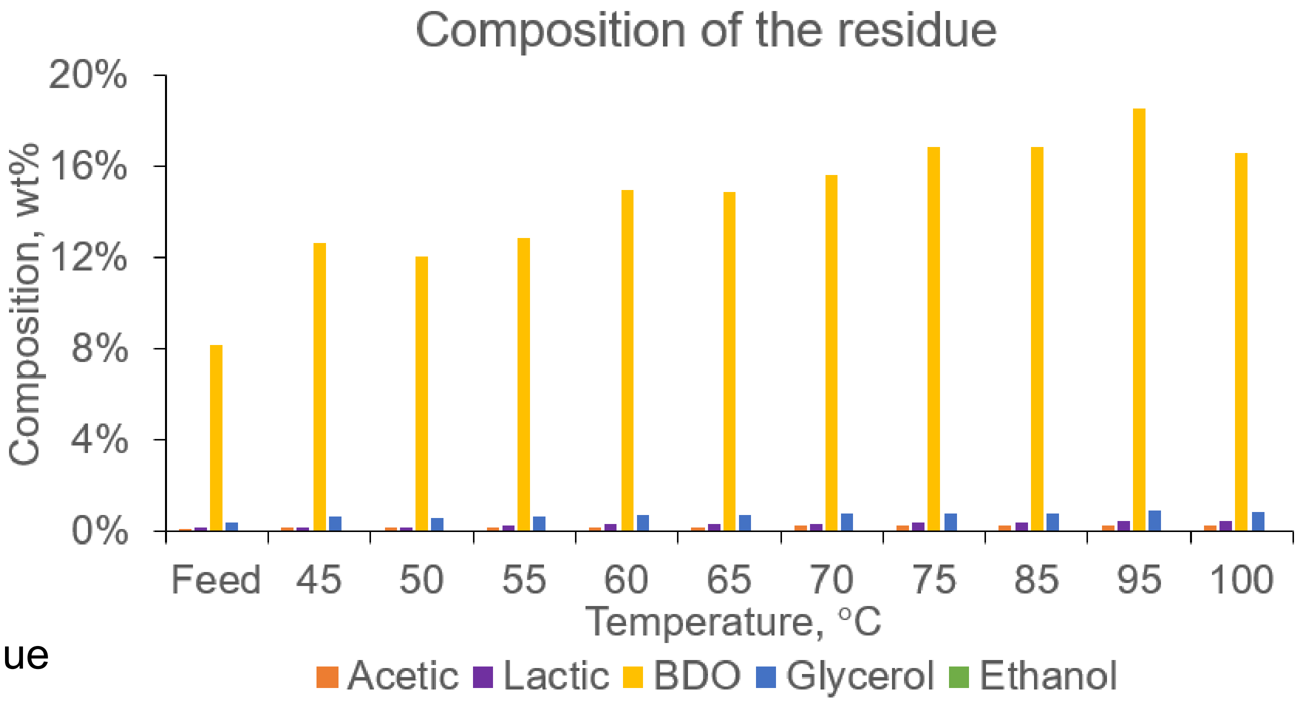
Progress update – vacuum evaporation is for the first stage concentration

Secondary condenser

First condenser



Residue



- BDO concentration reached the highest at 95 °C in the residue, 20 wt% BDO in water, for the next stage membrane separation.
- BDO concentration increased with temperatures in the condensate, reached the highest at 100 °C, 4.5 wt% BDO in water, which can be recycled back to fermentation process.

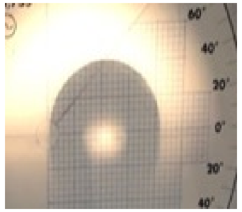
Progress update – Hydrophilic PCM improves performance

Defect-free crosslinked uniform membrane

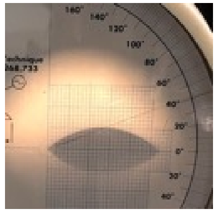
ORNL tubular polybenzimidazole coated porous ceramic membranes (PCM)

Contact Angle
Surface Wetting Studies

PCM

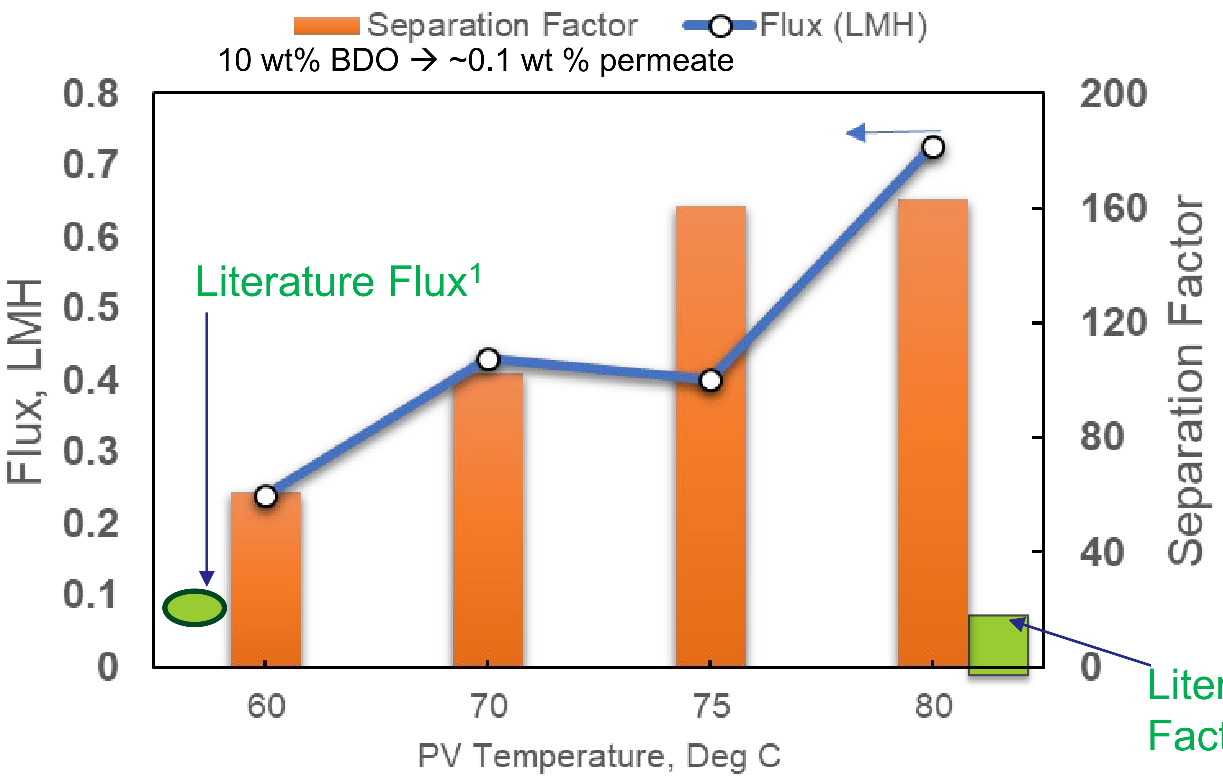


S-PCM



Hydrophilic Coating improved high flux (Sulfonated PCM)

10 wt% BDO Separation at Different Temperatures



Literature Separation Factor [1]

Flux $J = \frac{v_p}{A \cdot t}$

LMH = L/m^2/h

Separation Factor

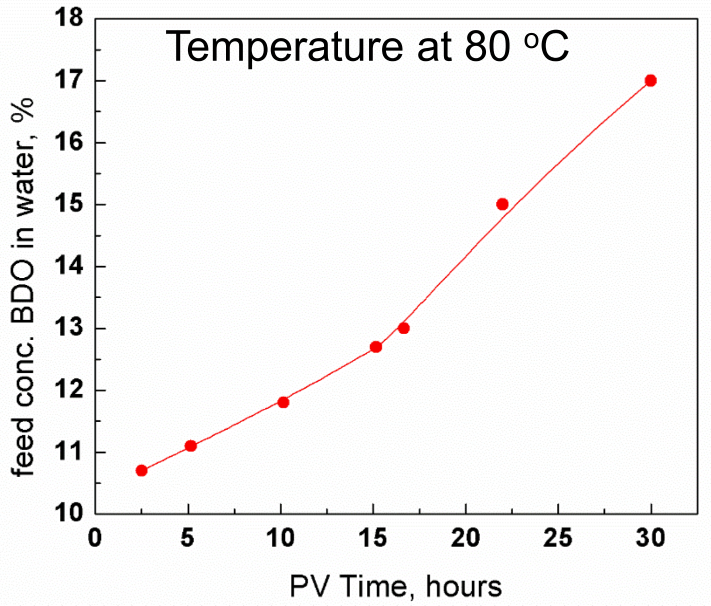
$$\alpha = \frac{y_B / y_W}{x_B / x_W}$$

Weight fractions of BDO/water in the feed and permeate

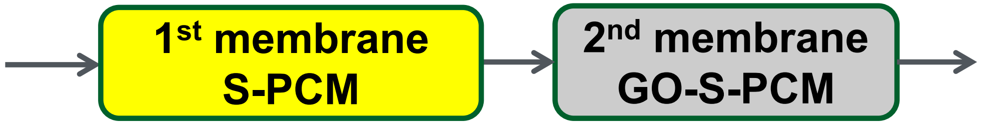
- We addressed defect issues in membrane synthesis with polymer crosslinking techniques
- We used sulfonation of the PCM (S-PCM) to improve water permselectivity and demonstrated it with contact angle surface wetting tests[2]
- We observed separation factors and fluxes with notable improvements beyond literature reports

1. Shao, Pinghai, et al Journal of Membrane Science 329.1-2 (2009): 160-168.
2. Wang, Yan, et al Journal of Membrane Science 415 (2012): 486-495.

Progress update – BDO enrichment demonstrated with S-PCM

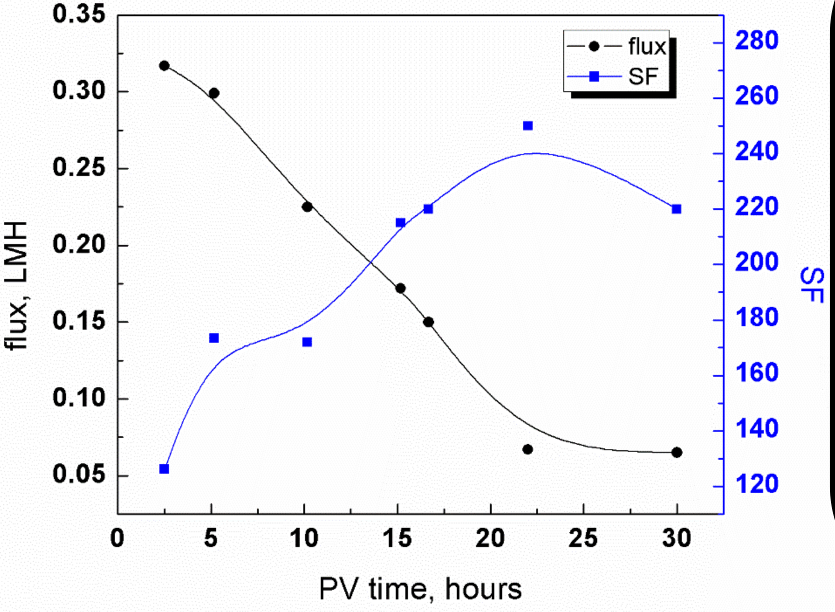


BDO concentration: 10 wt% to 17 wt%   17 wt% to 30 wt%

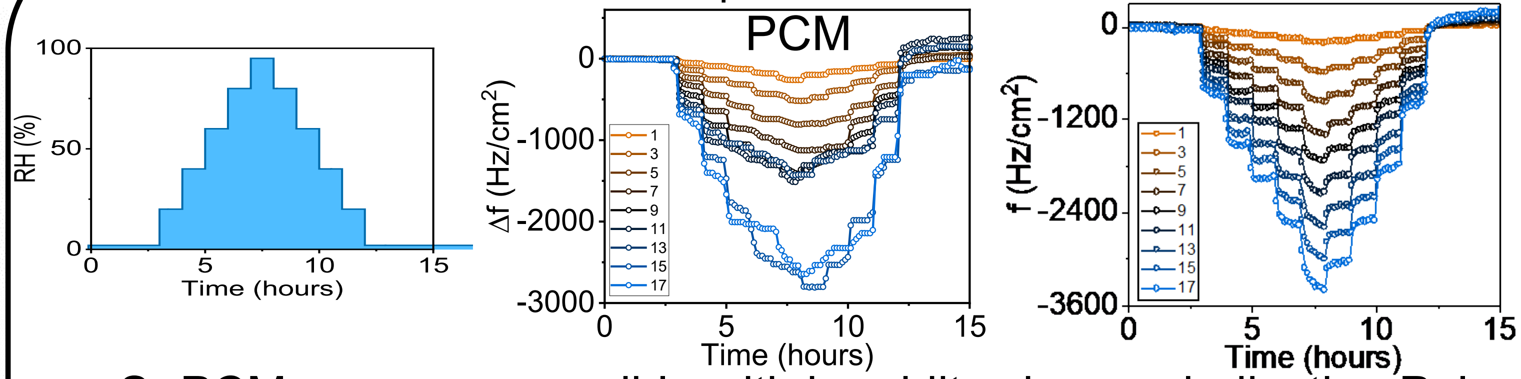


In 30-hour study:

- Observed BDO enrichment from 10.7% to 17.0%
- Observed decrease in flux of water vapor thru membrane (indicating fouling)
- We are working with CCPC collaborators to understand the membrane fouling mechanism via computational modeling

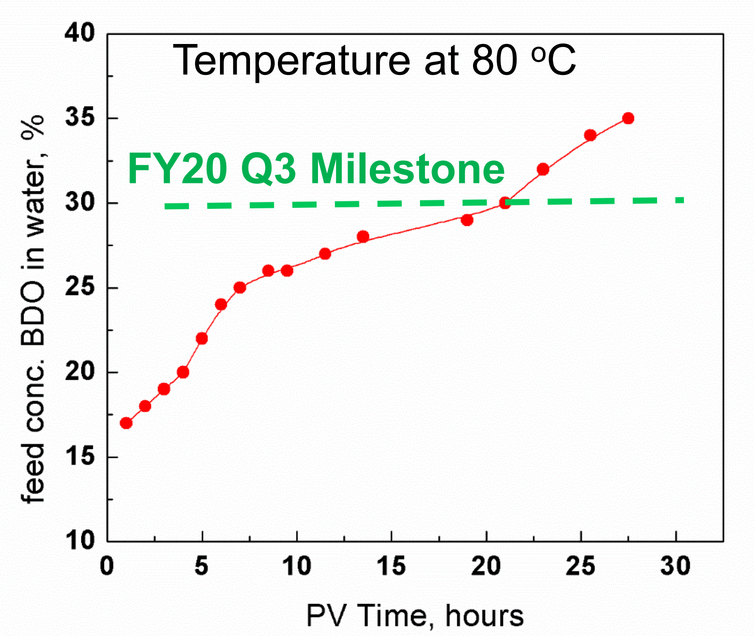


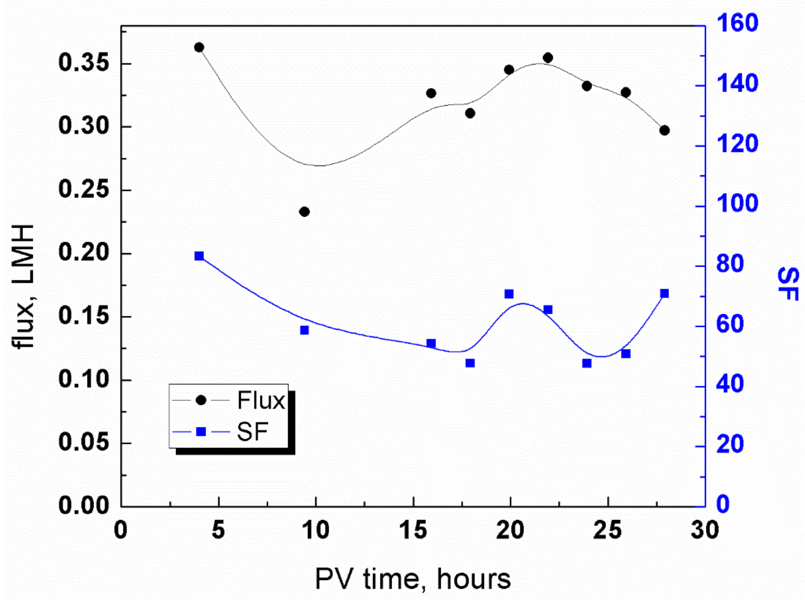
In-Situ QCM Water-Uptake Studies

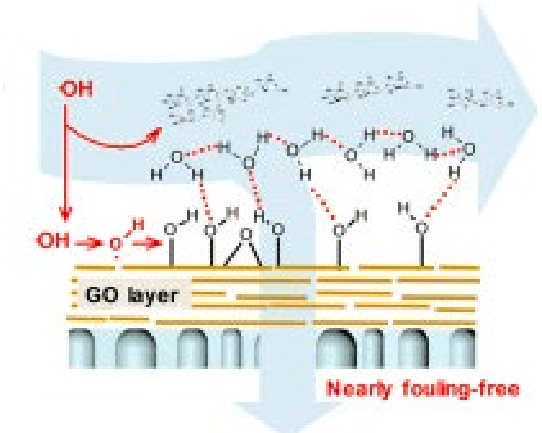


S- PCM appears reversible with humidity change, indicating Polymer swelling (common mechanism) does not appear to be cause for s-PCM in quartz crystal microbalance (QCM) study, so the main reason of reduced flux is fouling.

Progress update – Addition of Graphene Oxide (GO) to S-PCM improves stability of flux

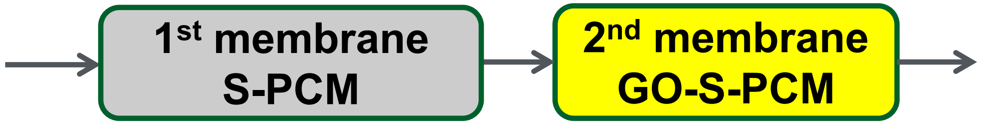


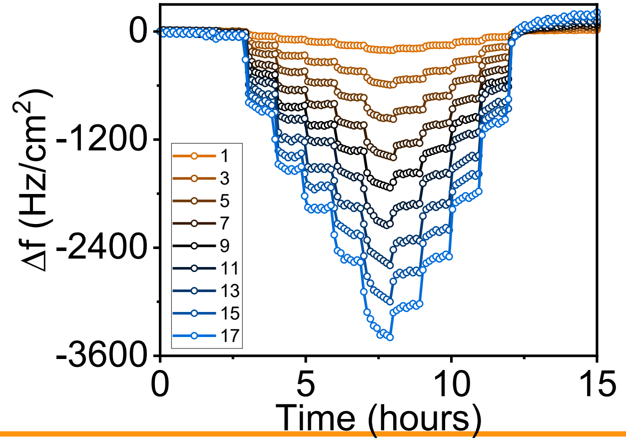




Graphene Oxide – Anti fouling*

BDO concentration: 10 wt% to 17 wt% 17 wt% to 30 wt%



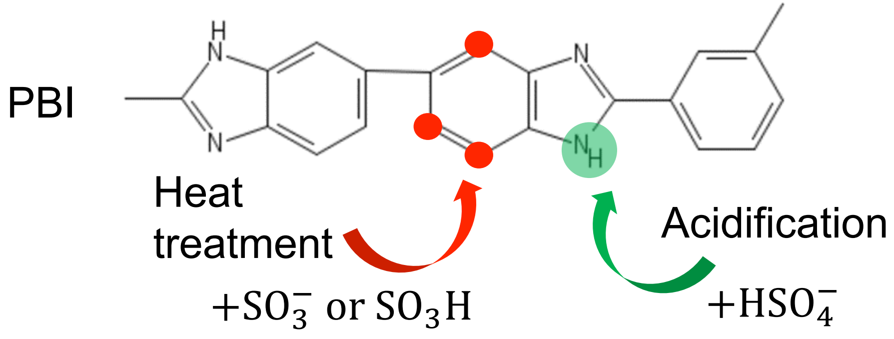


- Graphene Oxide addition improved stability of S-PCM performance
- **Achieved FY 20 Q3 milestone – reached BDO enrichment to 30 wt%** by cascading these two membranes!
- Next step – continue investigating:
  - Graphene Oxide and functional groups.
  - Alternative porous supports for lower materials cost.
  - Process scale-up

**Han, Jing-Long, et al. Journal of Membrane Science 576 (2019): 190-197.*

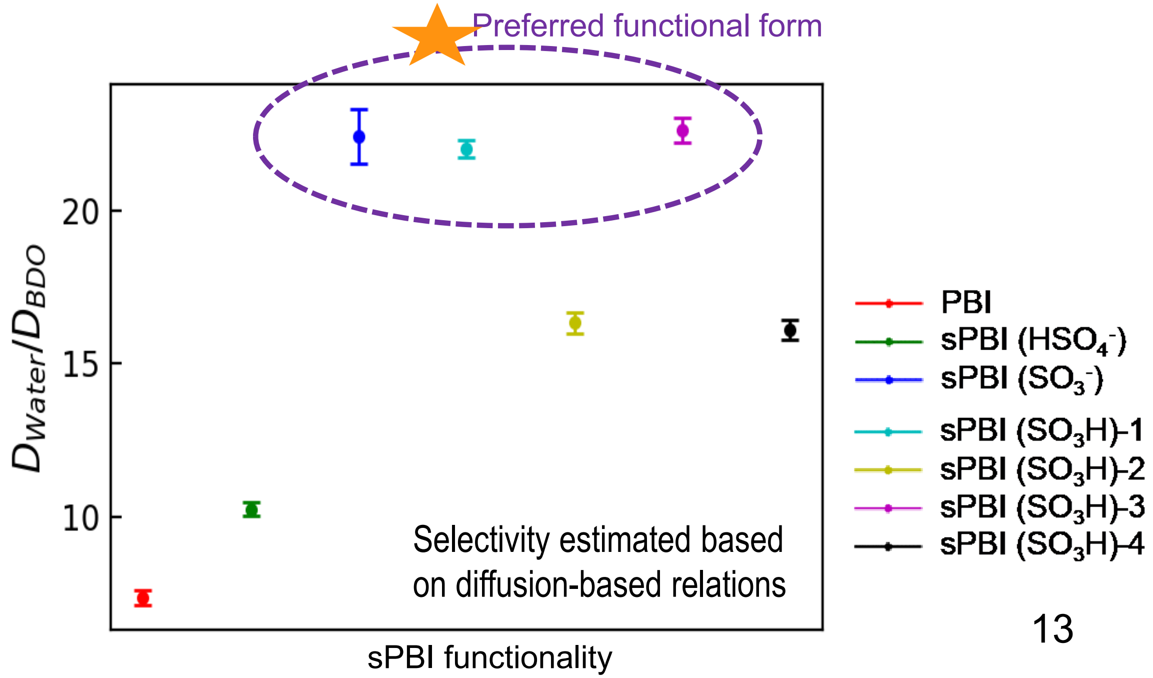
Computation reveals how various forms of sulfonated PBI affect the BDO/water separation

The thin layer polymer coating on porous ceramic structure : Sulfonated Polybenzimidazole (sPBI)



Binding energies (BE) (kJ/mol)

	BE(Water)	BE(BDO)	BE(Water)/BE(BDO
PBI	-37.6	-58.4	0.64
sPBI (HSO_4^-)	-50.0	-97.9	0.51
sPBI (SO_3^-)	-61.5	-131.7	0.46
sPBI (SO_3H)-1	**-59.4**	**-83.7**	**0.70**
sPBI (SO_3H)-2	-62.3	-92.8	0.67
sPBI (SO_3H)-3	-60.4	-89.2	0.67
sPBI (SO_3H)-4	-42.4	-61.7	0.68



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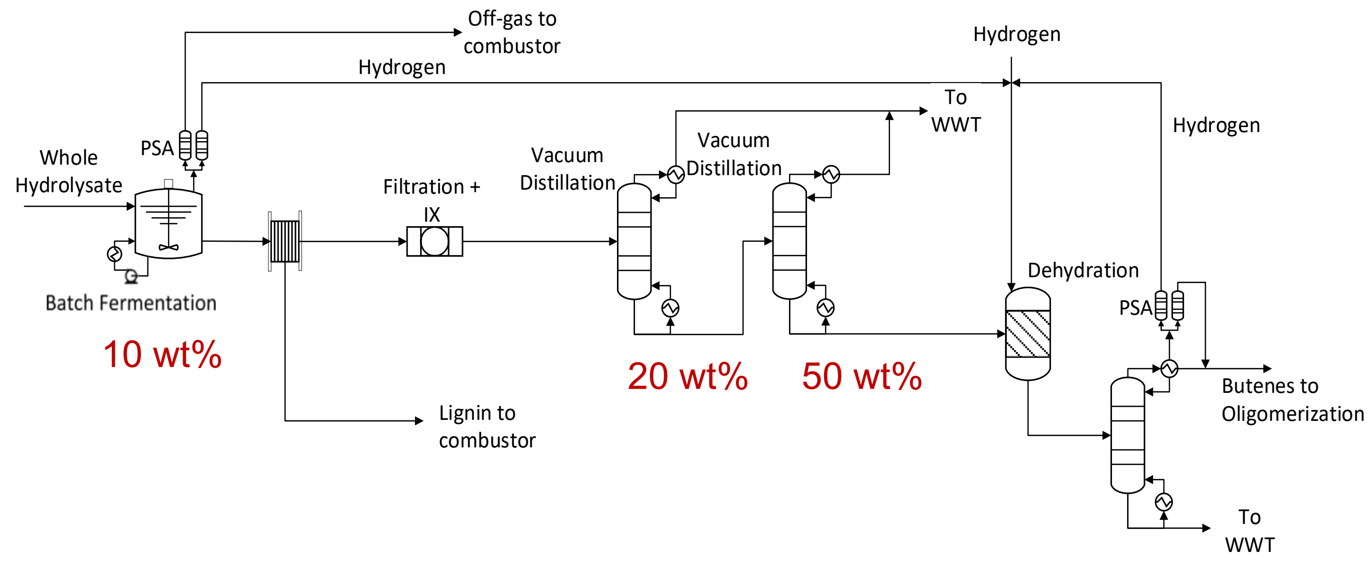
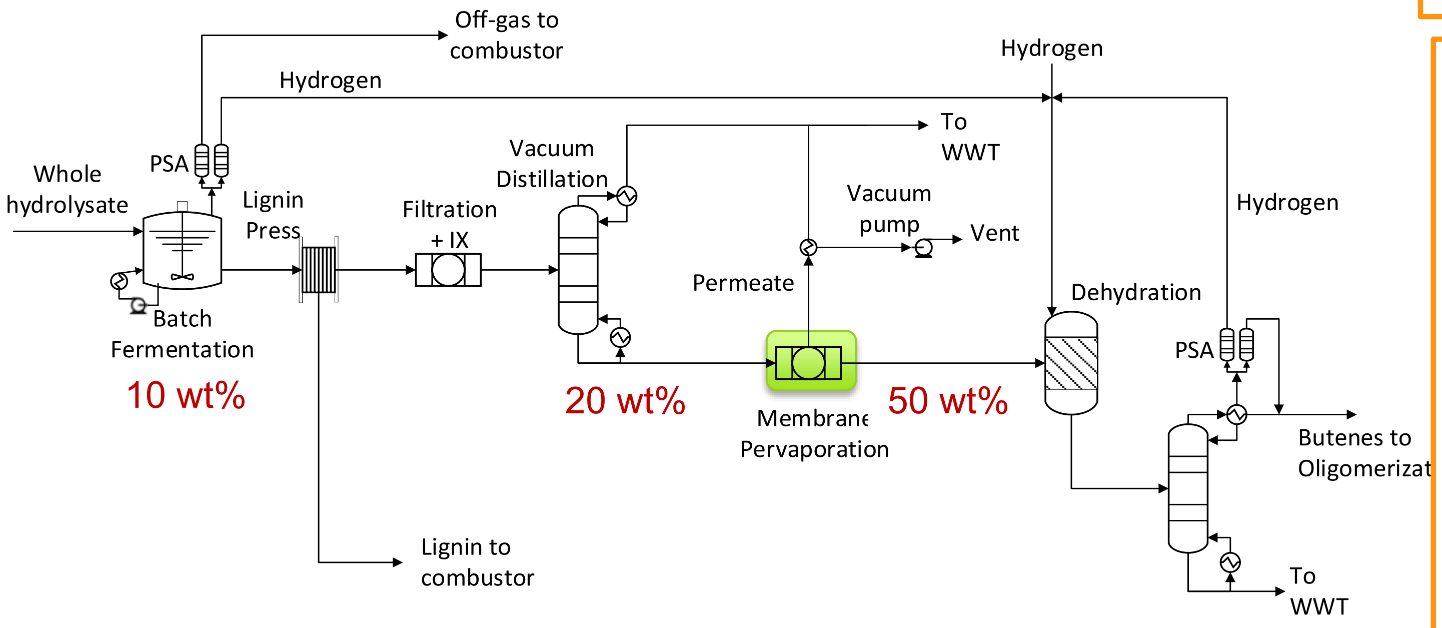
- Site functionalization impacts binding and diffusion.
- Atomistic simulations used to quantify the separation selectivity of each form of functionalization via molecular simulations.
- We are working on Graphene Oxide membrane modeling

Progress update - TEA Scenarios





Baseline Case

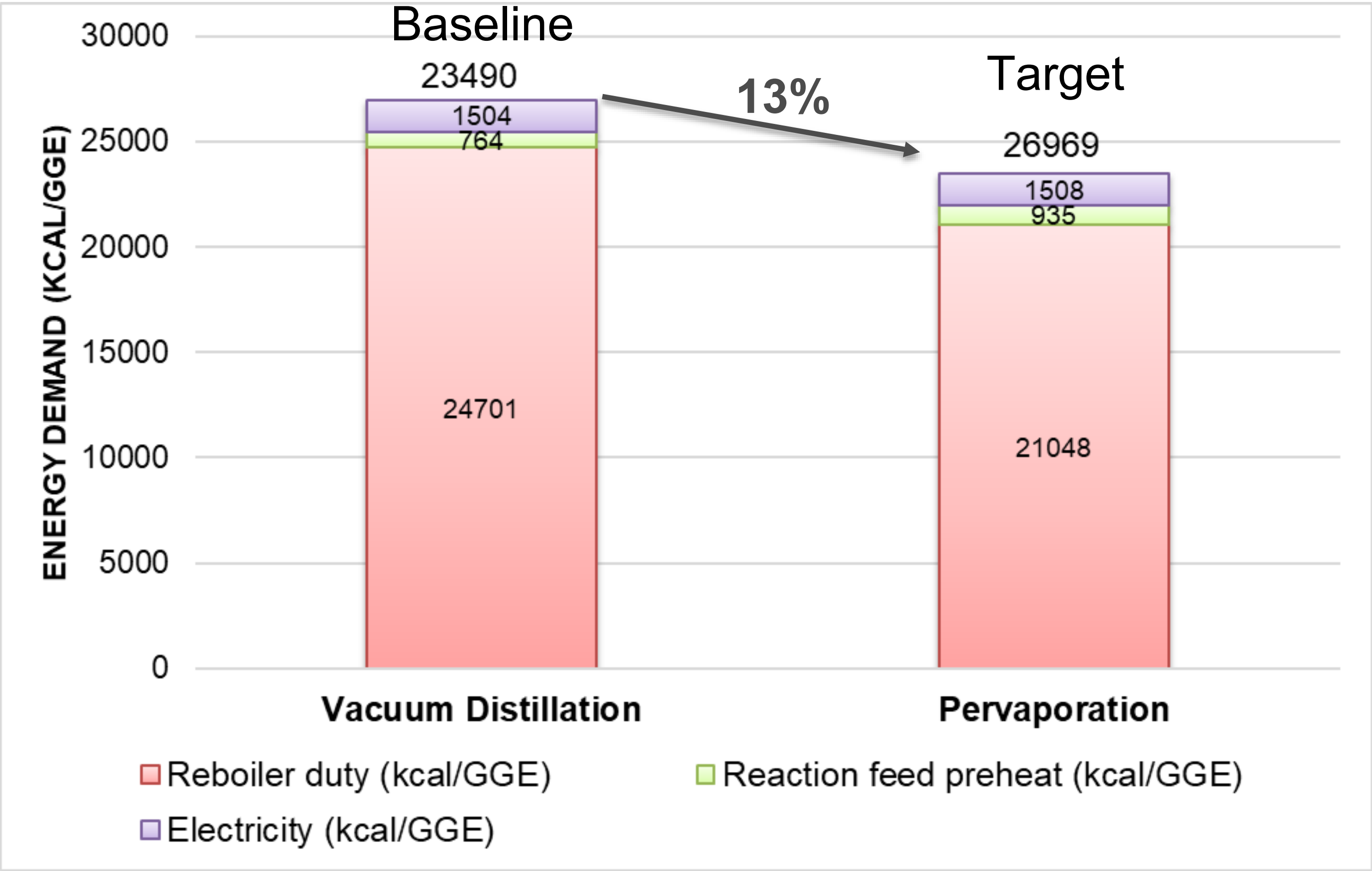
- With BDO pre-concentration
- Using two-stage vacuum evaporation to increase the BDO concentration to 50 wt%: 10 wt% → 20 wt% → 50 wt%
- Using vacuum distillation at a reduced pressure will decrease the boiling point (avoiding oligomerization).

Target Case

- With BDO pre-concentration
- Combination of the vacuum evaporation step and membrane pervaporation step.
- Vacuum evaporation increases the BDO concentration to 20 wt%, followed by the membrane pervaporation step to achieve 50 wt%.

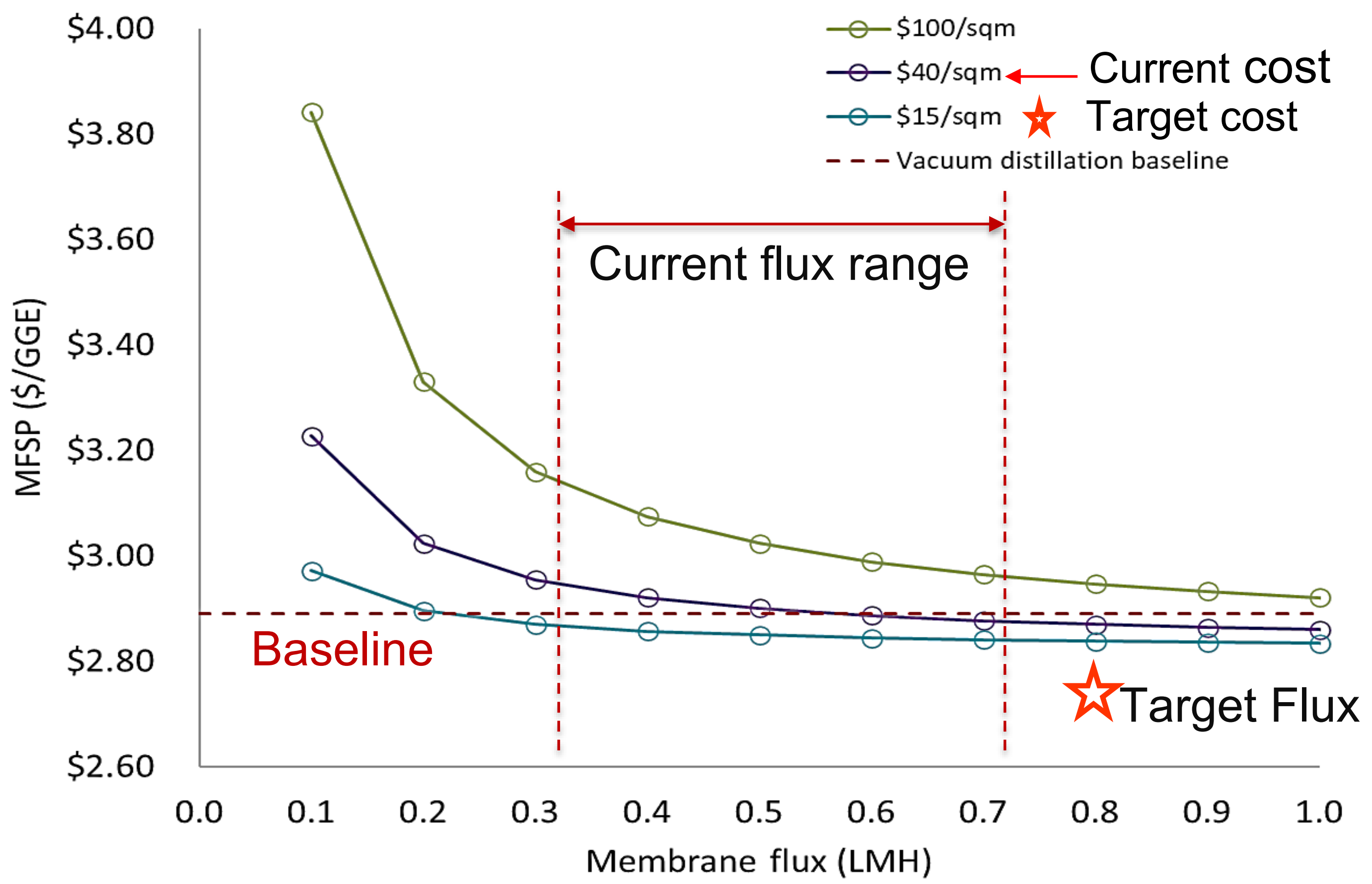
Progress update – BDO Pre-Concentration Energy Consumption

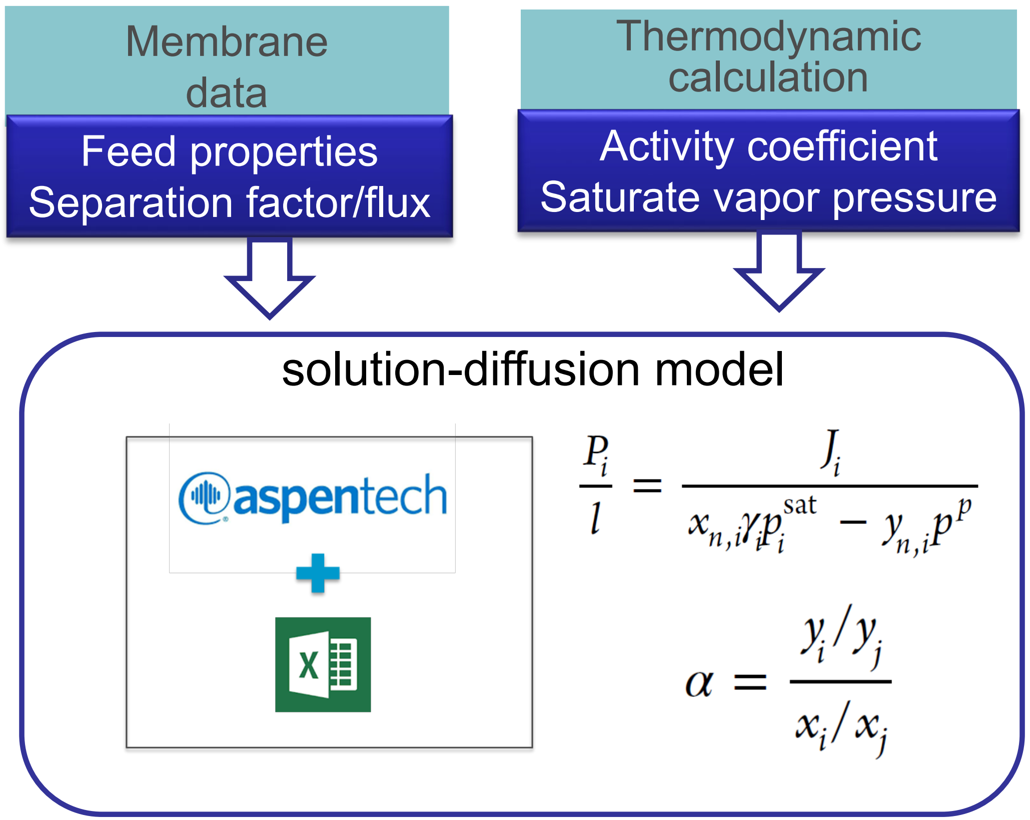
BDO 10% -- > 50%   BDO 10% -- > 50%



- System boundary: from BDO fermentation to upgrading reactor feed.
- The target case exhibits 13% lower energy consumption when compared to the baseline case.

Progress update – Biorefinery Level: Preliminary TEA Sensitivity Study





$$\frac{P_i}{l} = \frac{J_i}{x_{n,i}\gamma_i p_i^{sat} - y_{n,i} p^p}$$

$$\alpha = \frac{y_i / y_j}{x_i / x_j}$$

- Increasing the membrane flux will lower the MFSP.
- With the combination of low enough membrane cost and or high enough membrane flux, the target case can achieve lower MFSP than the baseline case.
- Solution-diffusion model can be used to estimate required membrane area and integrated into a full model.

Summary

Management	Frequent interaction and coordination among researchers at ORNL, LBNL, NREL, PNNL, LANL to meet project goals regarding experimental, computational, TEA/LCA and upstream/downstream work Regular updates and feedback from Industrial Advisory Board and steering committee
Technical approach	Development of low-cost, less-energy intensive separation technologies for processing dilute fermentation BDO broth into suitable feed for BDO upgrading pathways Integrated TEA and LCA identify research and development priorities and guide experimental work
Impact	Knowledge and tools to enable large scale membrane separation for bioprocessing Ongoing collaboration with industry and dissemination of results via conference presentations and peer-reviewed journal articles enable technology transfer
Progress	• We have achieved FY 20 Q3 milestone – reached 30 wt% BDO enrichment by membrane dewatering technology. And demonstrated evaporation and membrane separation for recovering BDO at low temperatures (80 ^{o}C) • We have worked closely with NREL biochem, ChemCatBio-upgrading team, TEA/LCA and computation teams in developing the separation technologies and BDO pathway. • TEA showed that the target case exhibits lower overall energy consumption (13% lower), when compared to the baseline case, and guide the materials selection. • We will continue improving the separation performance via integrated separations for removal of target molecular/ionic species from fermentation broths. We will provide the processed broth to upgrading team to evaluate impacts on the down-stream BDO upgrading catalyst and reactions.

Quad Chart Overview – Experimental work

Timeline
- Project start date: October 2019
- Project end date: September 2022

	FY20-22	Active Project
DOE Funding	(10/01/2019 – 9/30/2022)	*$1,020,000* *ORNL: $810,000* *LBNL: $120,000* *NREL: $90,000*

Project Partners
- ANL
- LANL
- NREL
- PNNL

Barriers addressed
Ot-B: Separation materials development

Ct-O: Selective separations of organic species

Ct-D: Advanced bioprocess development

Project Goal
The goal of this task is to develop low-cost, less-energy intensive separation technologies for processing dilute fermentation BDO broth into suitable feed for BDO upgrading pathways in aligning with BETO priority.

End of Project Milestone
Deliver 1 L processed broth, which contains BDO > 50 wt% and sugar < 3 wt% with the durable membrane that can be run continuously > 20 hours. Provide operating conditions, membrane material, and membrane performance data to the analysis team. [ORNL, LBNL, NREL – Q3 QPM]

Outcomes: Improved low-cost, less-energy intensive separation technologies for processing dilute fermentation BDO broth into suitable feed for BETO-priority CUBI-upgrading pathways.

Funding Mechanism
Merit-reviewed AOP-based Consortium

Quad Chart Overview - Analysis

Timeline

- Project start date: October 2019
- Project end date: September 2022

	FY20-22	Active Project
DOE Funding	(10/01/2019 – 9/30/2022)	*$3,125,000* *ANL: $565,000* *LANL: $300,000* *NREL: $890,000* *PNNL: $1,370,000*

Project Partners

- ANL
- LANL
- NREL
- PNNL

Barriers addressed

Ot-B: Cost of production

Ct-O: Selective separations of organic species

Ct-D: Advanced bioprocess development

Project Goal

The goal of this task is to inform research direction and go/no-go decisions by identifying the separations challenges that most influence the cost and sustainability of producing fuels in BETO priority pathways. The cross-cutting computational task applies modeling to assist in the down selection and optimization of material properties.

End of Project Milestone

Complete TEA and LCA for all projects in Consortium and document results.

Complete the revised separations challenge stream analysis, identifying top streams based on the economic potential of effective product separation. Submit as a final report or journal manuscript.

Funding Mechanism

Merit-reviewed AOP-based Consortium

Acknowledgements

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Gayle Bentley, Nichole Fitzgerald

Ben Simon, Clayton Rohman

BIOPROCESSING SEPARATIONS CONSORTIUM

Steering Committee **Jennifer Dunn, Taraka Dale, Gregg Beckham**

OAK RIDGE National Laboratory

- **Andy Sutton**
- **Eric Muckley**
- **Ilia Ivanov**
- **Jim Parks**
- **Josh Thompson**
- **Matt Olumide**
- **Michelle Kidder**
- **Tim Theiss**

Separations Consortium Members and Partner Laboratories

BERKELEY LAB

Los Alamos NATIONAL LABORATORY EST. 1943

Argonne NATIONAL LABORATORY

NREL NATIONAL RENEWABLE ENERGY LABORATORY

Pacific Northwest NATIONAL LABORATORY

ChemCatBio Chemical Catalysis for Bioenergy

NREL Biochem Team

Nancy Dowe, Min Zhang, Rick Elander

Publication and Patents

Publications on BDO Task:

S-PCM membrane separations of BDO (in preparation, Jan 2021)

Publications in FY20 based on previous tasks:

1. Aimee L. Church, Michael Hu. Novel Porous Ceramic Tube-Supported Polymer Layer Membranes for Acetic Acid/Water Separation by Pervaporation Dewatering., *Separation and Purification Technology* Volume 236, 1 April 2020, 116312

2. Aimee L. Church, Michael Z. Hu, Suh-Jane Lee, Huamin Wang, Jian Liu. Selective adsorption removal of carbonyl molecular foulants from real fast pyrolysis bio-oils. *Biomass and Bioenergy*, Volume 136, May 2020, 105522

3. A Combined Experimental and Computational Investigation of Acetic Acid Ketonization over Ca-doped CeO2 Catalyst (ORNL & ANL, submitted to Applied Catalysis)

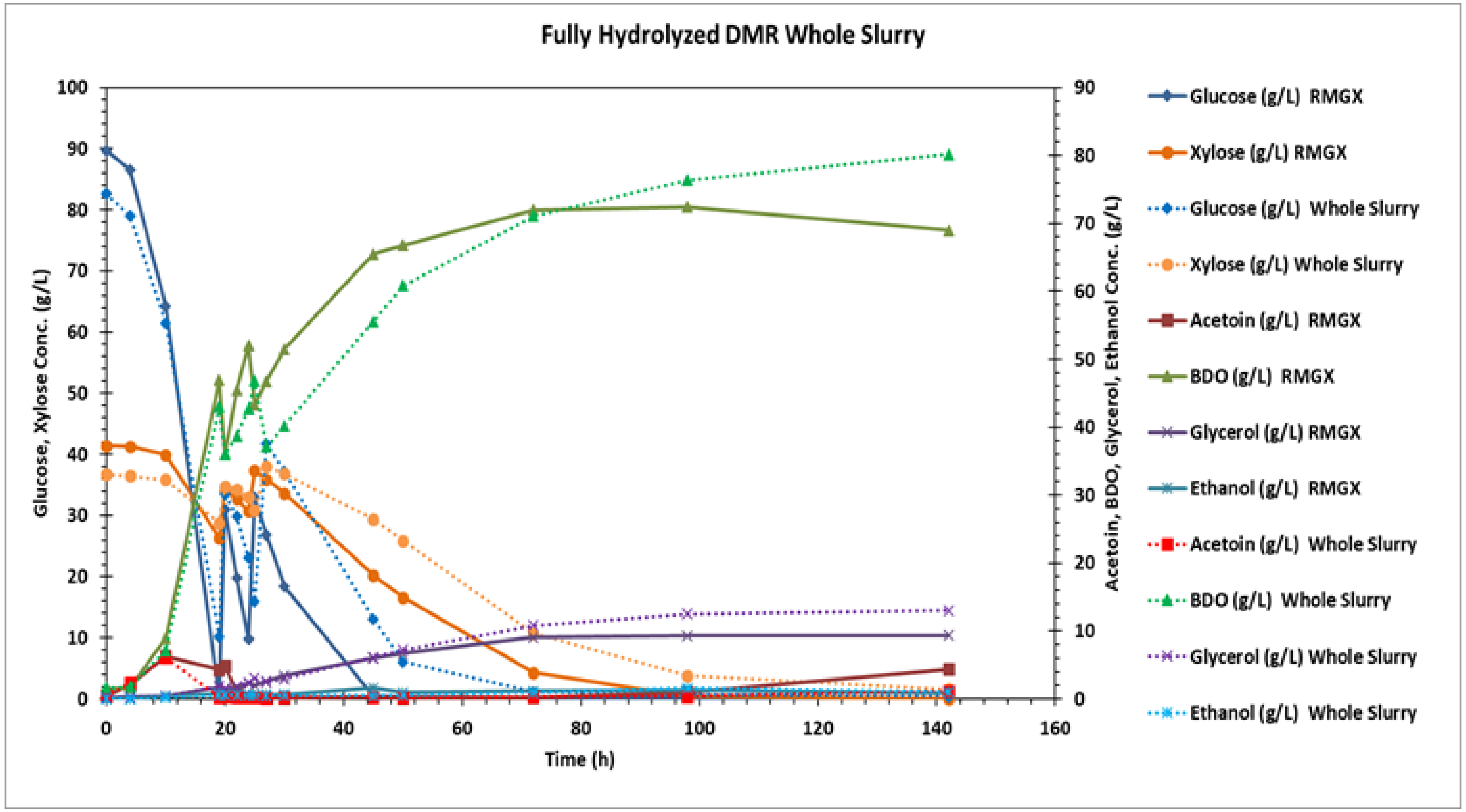
4. Denitrogenation computation & experiments (ORNL & PNNL, in preparation)

Patents

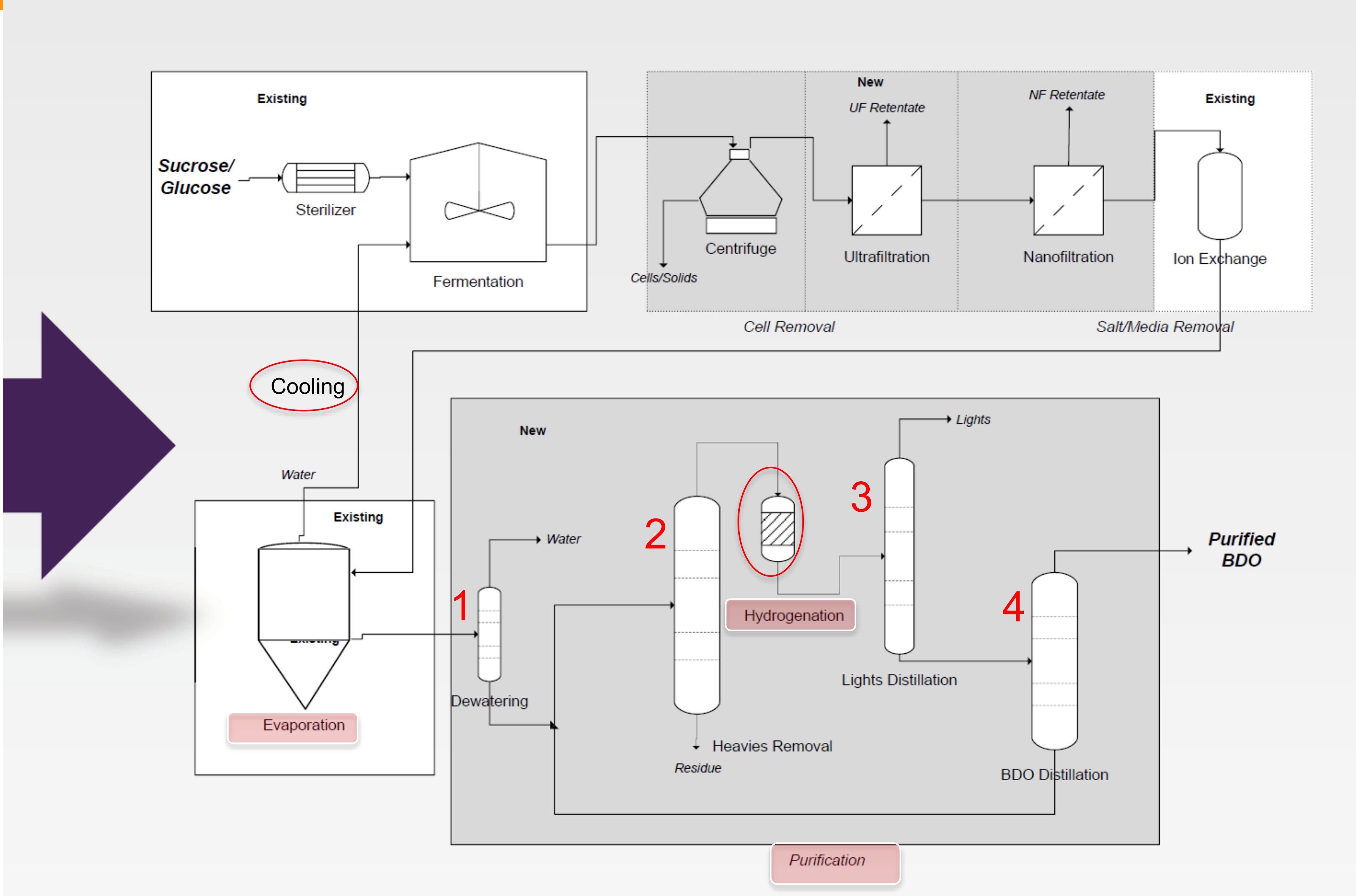
Invention filed on membrane materials, provisional patent filing is in process

Extra slides

Discussion with fermentation team (Nancy Dowe, Min Zhang, Rick Elander)



2016 Genomatica-Novamont commercial plant process: -- purifying 2,3 BDO as final product



Source: extract from CEO's presentation

MPV can operate in industrial scale

Hydrophilic membranes on ceramic substrates

- Energy saving
- Azeotrope separation is perfectly possible
- Decrease usage of cooling water
- Enhanced product quality also through milder conditions
- Reduced formation of side products
- Higher plant availability
- Chemical resistant
- Stable up to high temperatures

Major applications

- Removal of water from organics.
- Removal of organics from water.
- Separation of organic mixtures.
- Concentration of aqueous solutions.

Organophilic membranes on polymeric or ceramic substrates

- Selective of high value products like aromas, flavors, fragrances
- Increased efficiency of fermentation processes
- Stable up to high temperatures and broad pH range

Main advantages

- Since only the properties of the membrane determine the distribution of a component in the permeate phase, mixtures which at normal distillation form azeotropes and/or require a large number of theoretical stages (like the dehydration of acetone), can easily and economically be separated even without the use of entrainers. Therefore, high product purity is obtained (no entrainer required) and no environmental pollution occurs (no entrainer emitted).
- Multi component mixtures even with just small differences in boiling points can be dehydrated effectively and economically.
- The feed mixtures to be treated may be supplied in either liquid (→ pervaporation) or vapor (→ vapor permeation) form.
- Low energy consumption for pervaporation and vapor permeation processes.
- Significantly reduced energy consumption for hybrid systems (pervaporation and vapor permeation in combination with rectification/distillation).
- Due to the modular design of the membrane system even small units can operate economically.
- High degrees of flexibility regarding the feed mixtures that may be accommodated (multi-purpose systems, various feed mixtures can be treated in one unit), throughputs, and final product qualities.
- Modularly, compactly designed, and factory-preassembled systems simplify their adaptation to suit the desired performance parameters and shorten the time required for system installation and start-up.
- Pervaporation and vapor permeation systems are simple to operate and can be started up and shut down rapidly.

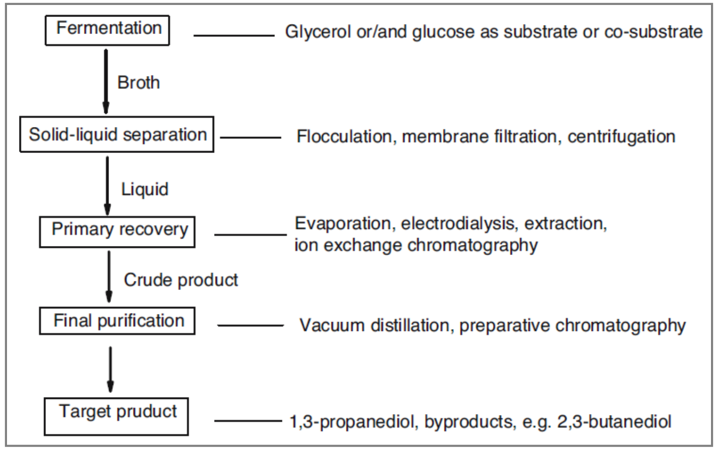
https://pervaporation-membranes.com/products/membranes/

Literature SOT Overview:
Separations from fermentation broth to diols (e.g., 2,3-BDO, 1,3-PDO)

Methods	Purposes	Drawbacks
Distillation, Evaporation	Remove water, diol purification	**Large energy consumption**
Electrodialysis	Desalination before evaporation	Low product yield, Serious membrane pollution
Pervaporation (Membranes)	Energy efficient water molecular separation	**Low flux** of zeolite membrane, impurity effect
Chromatography, sorption	Combine with resins, desal., separation and diol recovery	Slow throughput due to mass-transfer limitation
Extraction (Solvent, L-L, salting/sugar-out)	Recovery of diols	No effective extractant for hydrophilic PDO, **solvent** recycle, low **selectivity**
Reactive extraction/removal	Diol recovery by reaction, extraction, hydrolysis, etc.	Complicated multi-steps, require proteins/salt removal
Hybrid Extraction-Distillation (HED)	Recovery of 2,3-BDO from its fermentation broth	**High capital cost**, Organic solvent use, distillation need

An example: Multicomponent **multi-step** separations

Xiu&Zeng, 2008



Separations Challenges:

Expensive and energy-intensive separations for pure diols production

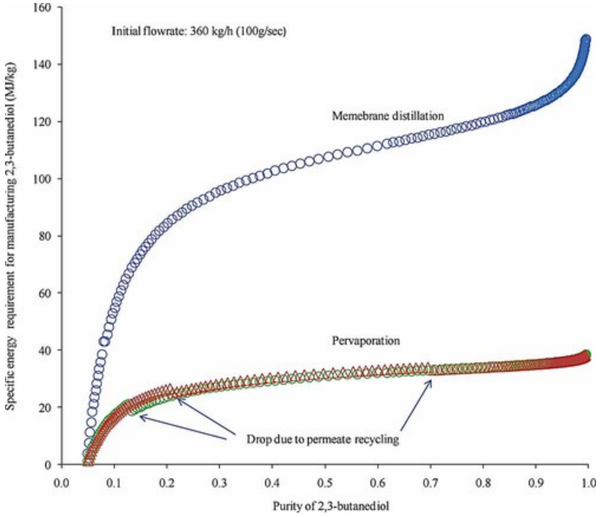
- **Low energy efficiency**: Conventional distillation is not desirable as BDO has a high b.p. (183°C), causing over half the cost of its microbial production.
- **Lacking efficient separation** technologies in the RO-NF regime [400-1000Da to nano]
- **Not large enough processing throughput (flux), low selectivity**
- **Operational stability**
- **High costs (capital/operation)**
- **Environmental concerns**

Literature MPV SOT

Table 4. Summary of PV performance of various types of membrane for butanol/water mixtures.

Membrane Type	Feed Concentration [wt %]	Feed Temperature [K]	Separation Factor	Flux [kg·m^{-2}·h^{-1}]
PDMS	1.5	328	43	0.67
PDMS	1	333	51	1.08
ZIF-7/PDMS	1	333	66	1.69
ZIF-8/PDMS	1.5	353	82	4.85
ZIF-8/PMPS	1	353	40	6.40
Silicalite-1/PDMS	1	313	92	0.13
Ge-ZSM-5	5	303	19	0.02
Silicalite-1	1	343	150	0.10
Silicalite-1	1	318	465	0.04
Silicalite-1	1.5	353	207	0.22
*BEA	1	318	229	0.62

Membrane Pervaporation provides four times energy savings over the vacuum membrane distillation



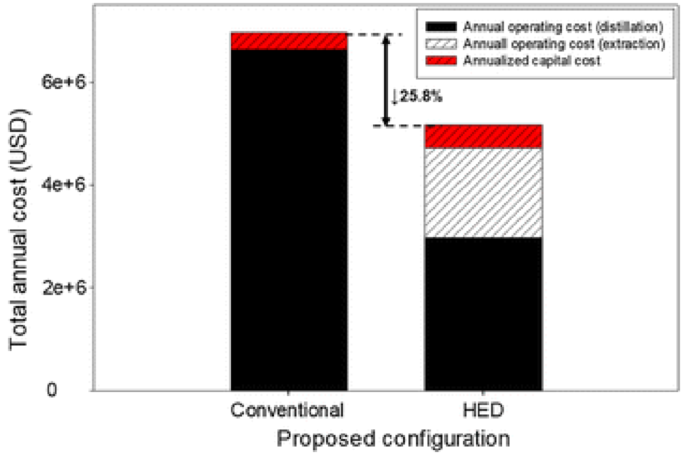
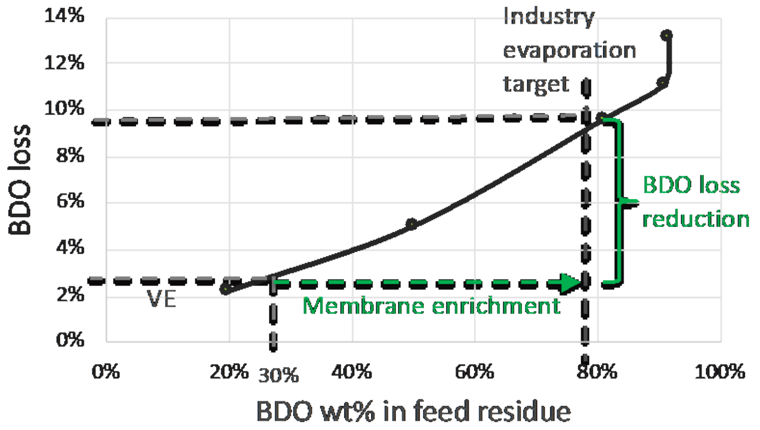


Table 1
Performance of various membranes for recovering 2,3-butanediol from aqueous solutions.

Membrane	Feed (wt.%)	Permeate (wt.%)	Separation factor
PDMS (250 μm)	1.1	0.3	0.3
PE[a] (55 μm)	1.4	0.5	0.3
PP[b] (30 μm)	1.1	0.5	0.5
PVC[c] (120 μm)	1.4	0.5	0.3
PE with carbon black (80 μm)	4.2	1.5	0.3

[a] Polyethylene.
[b] Polypropylene.
[c] Polyvinylchloride.

Vacuum Evaporation of 10 wt % BDO



Membranes **2019**, *9*(7), 86; https://doi.org/10.3390/membranes9070086

Shao, P., & Kumar, A. The CanadiaHarvianto, G.R., Haider, J., Hong, J. et al. Biotechnol Biofuels 11, 18 (2018)
n Journal of Chemical Engineering, 89(5), 1255-1265. (2011).

Wiped Film Evaporation (WFE)/Distillation hybrid unit

What is WFE and its advantage

- Medium vacuum (to 0.1 torr) and a high surface area external condenser are utilized
- Evaporation with short residence time, lower temperature, without degradation of heat-sensitive material
- Readily scalable

Current applications

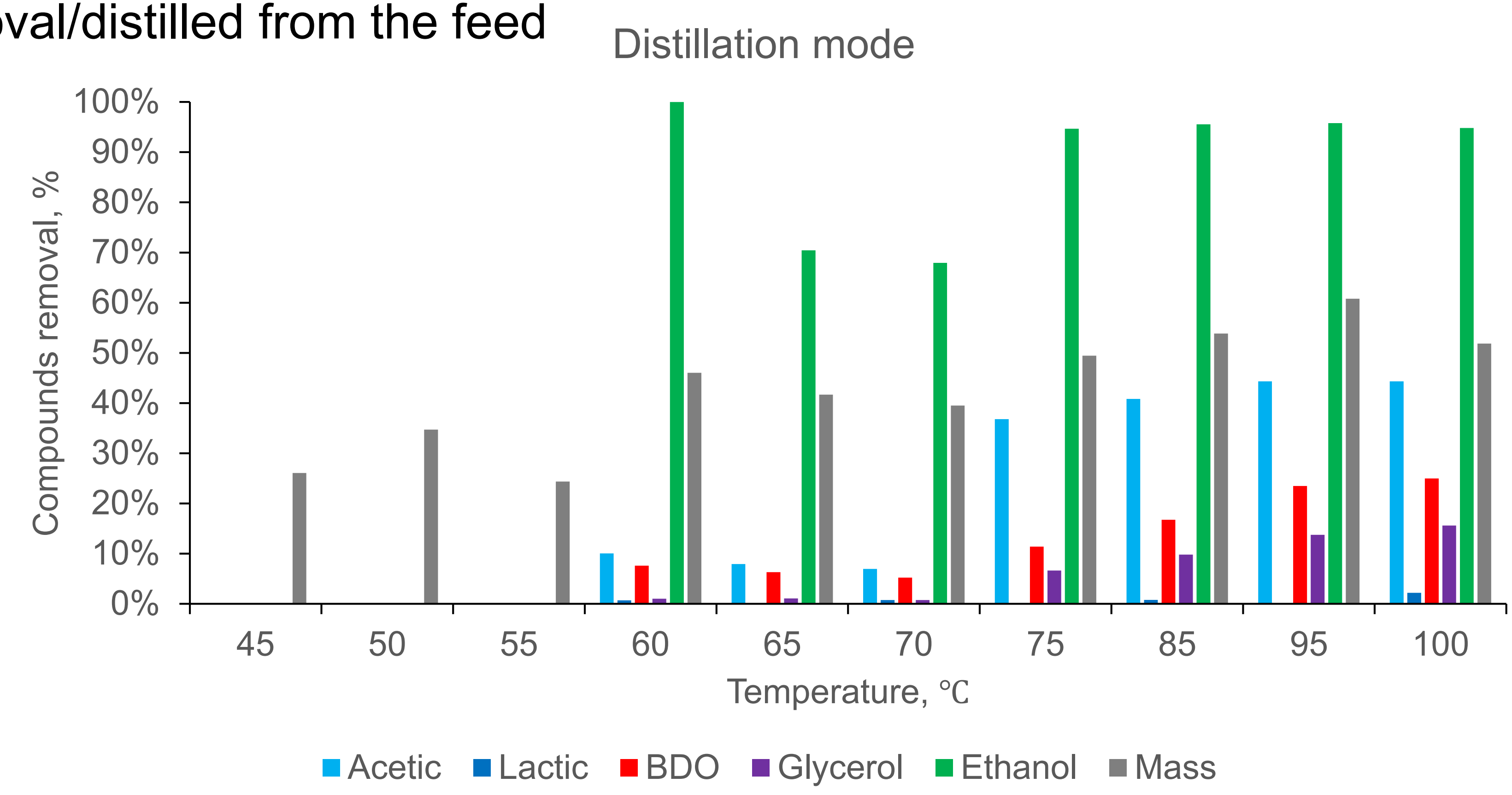
- Moisture removal
- Edible oil deodorization and deacification
- Vitamin and nutritional supplement purification
- Extract and biomaterial concentration
- Food, flavors and fragrance isolation
- Polymer devolatilization
- Wax and silicones fractionation
- Decolorization
- F.A.M.E. Fractionation (Also F.A.E.E.)
- Polyglycerol Distillation
- Omega – 3 Fatty Acid Distillation

2'' model @ LBNL: 50 mL/hr

20'' model: 350 l/hr

Progress update – vacuum evaporation

Compound removal/distilled from the feed



- No measurable acids, glycerol, ethanol observed in the distillate under 60 ℃
- Up to 25% BDO was removed at 95 ℃
- Water and BDO, are the major compounds in the condensate along with a small amount of acetic acid and glycerol