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

BIOENERGY TECHNOLOGIES OFFICE

BIOPROCESSING SEPARATIONS CONSORTIUM

Volatile Products Recovery

BETO 2023 Project Peer Review

Performance-Advantaged Bioproducts and Bioprocessing Separations

April 6, 2023

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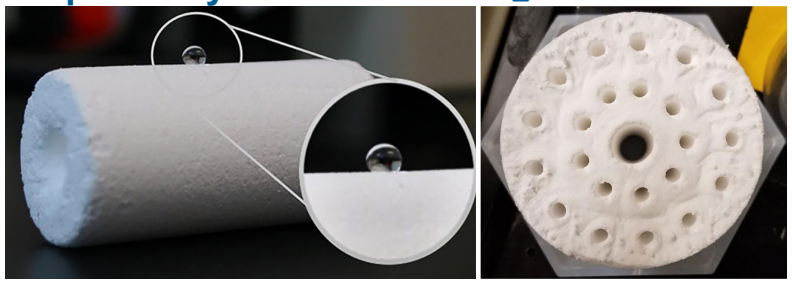
Project Overview

- Context and Bioprocessing Separations Challenge
  - Volatile product capture technologies continue to challenge biomanufacturing as current recovery methods are time/thermally intensive.
- Project goals and specific research question answered
  - Deploy advanced adsorbent materials in fermenter off-gas streams to recover a wide range of volatile product classes
  - Minimize energy use and maximize product purities by tuning materials surface chemistries to passively – and specifically – capture products (at culture temperatures)
  - Demonstrate near-quantitative capture of bioproducts from industrial-derived SAF-producing bioprocesses with product purities allowing for immediate use
  - Leverage prior successful technology deployment in the consortium to focus on scaling and industry engagement.
  - Validate at pilot-scale – and beyond – advanced, cost-effective materials synthesis and process integration
  - Can volatile bioproducts be approached economically in a biomanufacturing setting?

Specificity in humid air | **Accommodating high gas-flow rates**



- Integration within BETO mission space, broader BETO project portfolio and new SAF industrial interactions
  - Leveraging bioconversion systems from the other BETO consortia (Agile BioFoundry, Co-Optima, and BOTTLE) as well as SAF strains and processes from SOT projects and candidate processes leveraging C1 feedstocks

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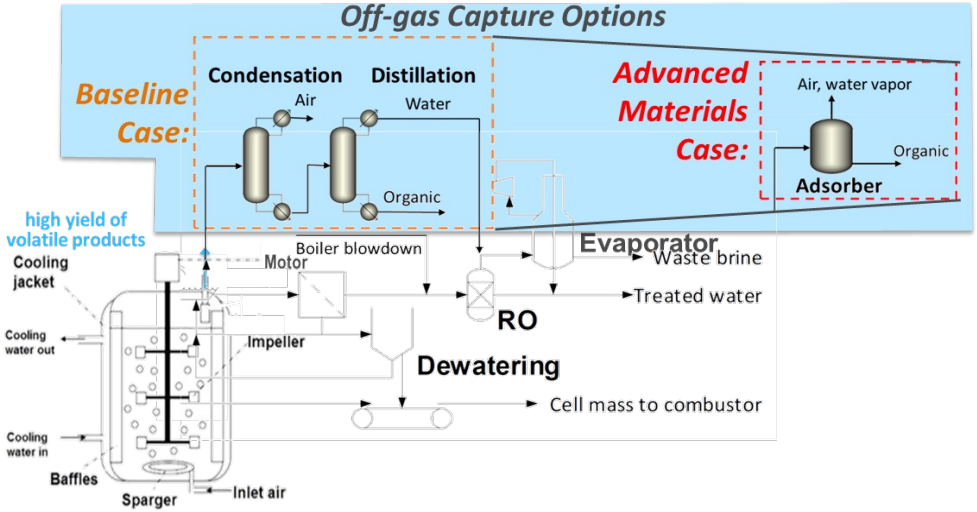
State of Technology

- Alternative methods are costly, and time/thermally intensive – requiring additional unit operations to achieve required purities.
  - Condensation/distillation requires an additional dewatering step in order to achieve purities needed for immediate use
  - Solvent overlay costs/operations versus volatilization and recovery
  - *In house* deployments will readily compare adsorbent approach to internal overlays and solvent stripping
  - To fully assess the technology space, we plan to deploy and compare competing technologies in house, strengthening publication impact and potential for new IP.

Streamlined process deploying advanced materials



Capture of volatile bioproducts from bioreactors from off-gases (blue) using the baseline (orange) or more efficient adsorbent-based recovery (red) technology.

- Economics or key metrics for justification over baseline case(s)
  - Reduce cost by demonstrating adsorbent reuse over ≥ 100 cycles.
  - Maximize recovery to >98% capture across product types – critical for TEA & LCA inputs to justify advantages over baseline technology(ies).
    - Expand baseline comparisons based on industrial interactions/conversations to include solvent overlays and chilled column capture, etc.

Approach

Strategy: Reduce operating costs and complexity of volatile product recovery via highly specific and tunable gas-phase adsorbents:

- Utilize selective nanostructured adsorbents
- Passive, non-thermal, adsorptive capture
- Facile product recovery via compression
- Eliminates continuous condensation and solvent stripping
- Extracts pure product without dewatering or distillation

Xerogels

Key Challenges:

- Designing materials with high specificity for product of interest while maintaining high gas flow
- Designing scalable and economically viable processes that incorporate advanced materials capture

These high-surface-area materials deployed as platform for testing of processes.

Key Metrics:

- Product capture efficiency, adsorbent capacity, product purity, and adsorbent longevity
- Process economics must demonstrate operational and capital costs less than 50% of baseline case(s)

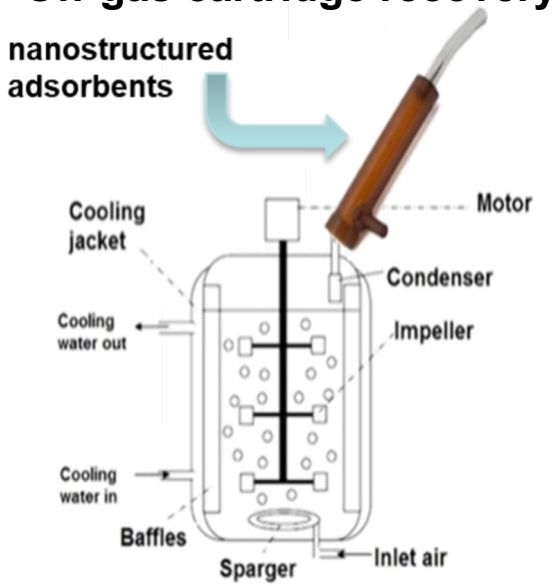
Volatile Product Recovery Task Management

- Frequent meetings and staff exchanges amongst performance sites to harmonize experimental methodologies and coordinate product and process selection and technology transfer
- Effective/recurring interactions with integrated analysis teams to understand and coordinate research with economic analyses and environmental assessments as drivers for process and product downselection decisions
- Computation-guided optimization of xerogel designs and surface chemistries
- Incorporate risk analyses from industrial perspectives, like those available from the LANL team to streamline experimental approaches and foci

Risk	Severity	Mitigation
Surface chemistry options are too limited to enable specific separation	Moderate	Survey additional surface modifications with chemical combinatorics, using search algorithms, to efficiently expand parameter space.
Accurate assessment of scalability for off-gas capture requires robust and industrially-representative fermentation processes to ensure high data fidelity	Moderate	Assess multiple products and processes in parallel and leverage fermentation processes from elsewhere in the BETO portfolio and from industry partners.

Volatile Capture Approach and Cartridge Designs

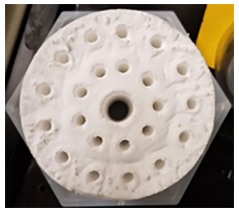
Off-gas cartridge recovery

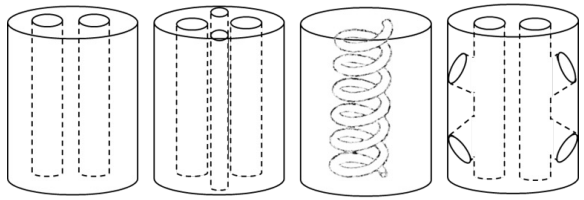


Advanced materials designs

Engineered xerogels accommodate high gas flows and bioproduct recovery

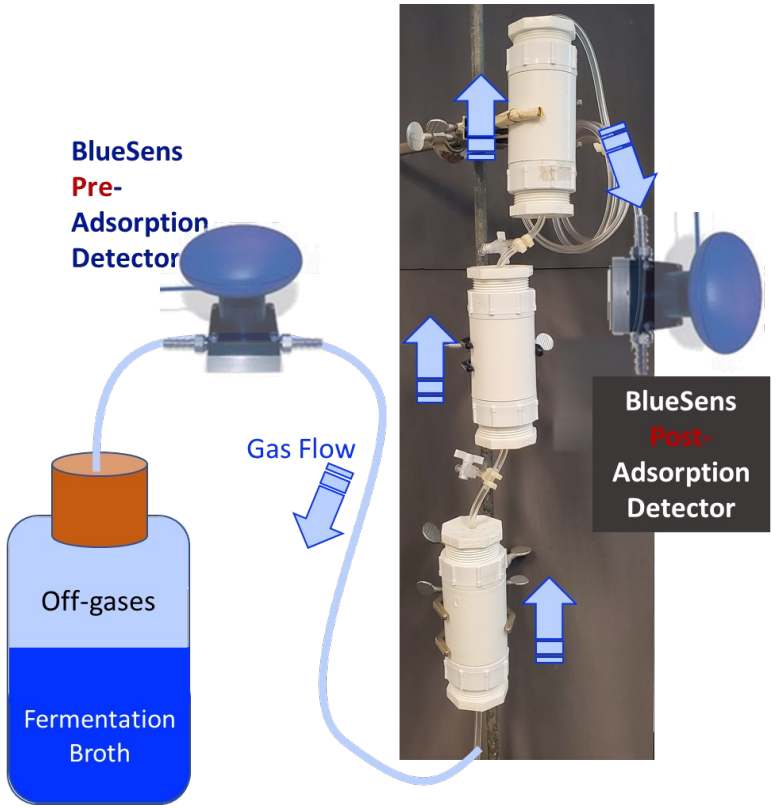
- Capacity for volatile products unaffected by presence of water vapor
- Water recovery insignificant

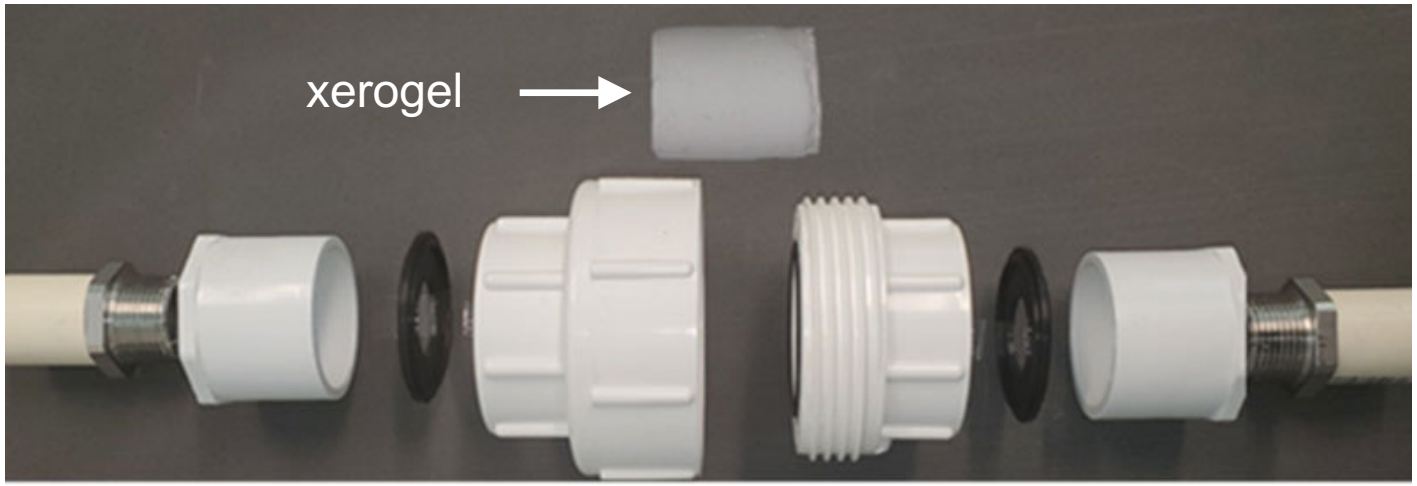




3D-printed macroporous structures under evaluation

Multi-stage Designs



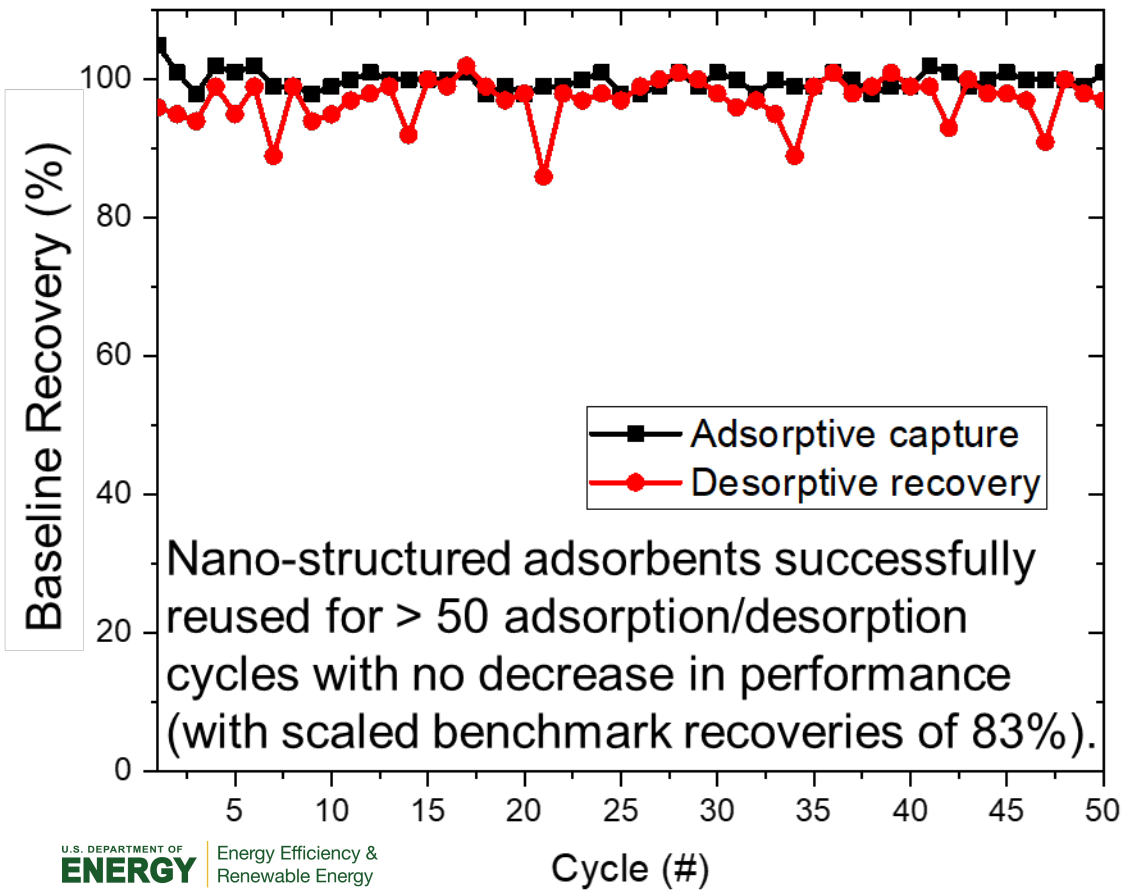


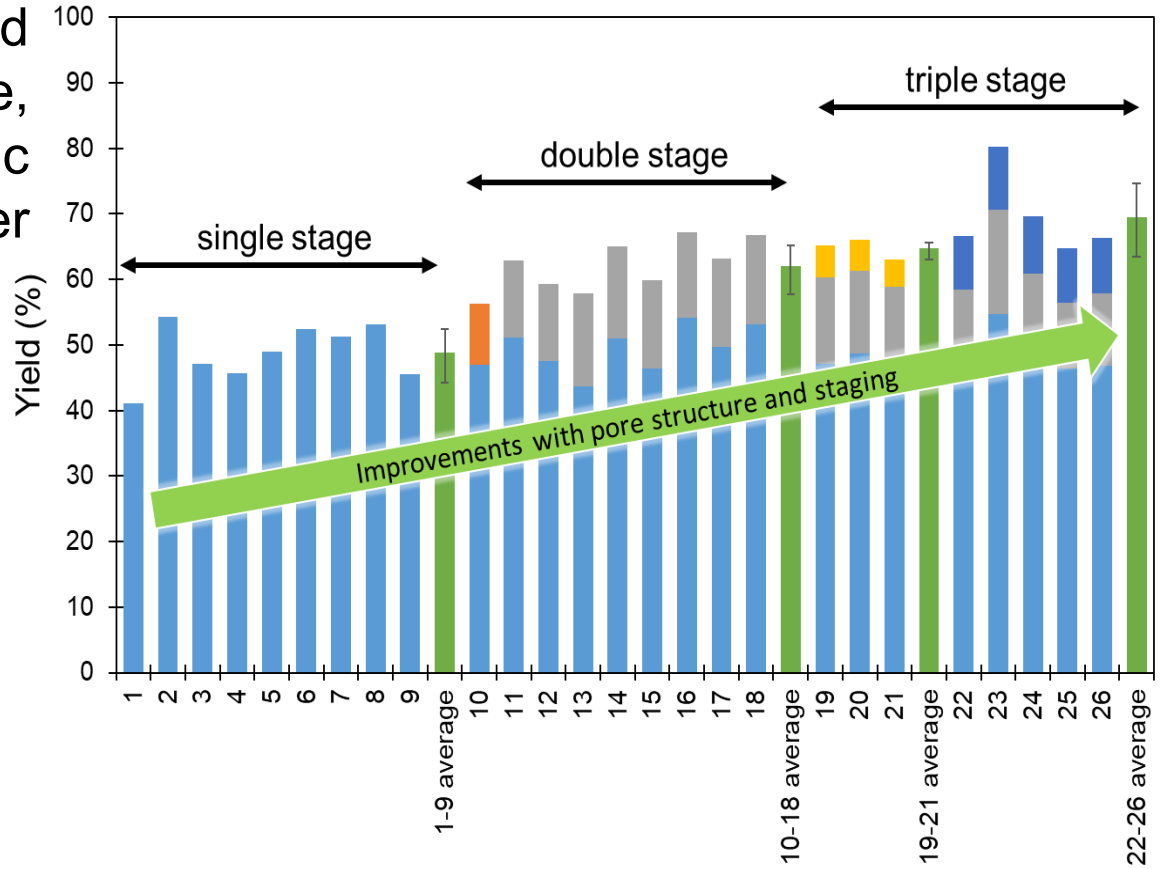
Initial cartridge prototype used in testing

PVC-based cartridge prototype and in-line sensor systems ready for use in materials evaluation and process scaling.

Progress and Outcomes

Demonstrated successful use of advanced materials with tunable surfaces in scalable, inexpensive capture cartridges, to recover specific volatile products with little-to-no water



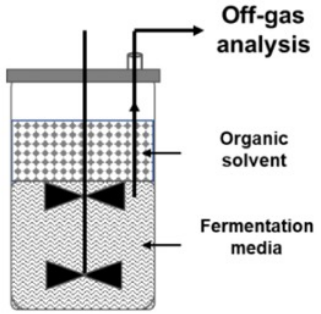


Optimized mechanical desorption processes to confirm material longevity in recovery cycling (and automated variant planned for FY23-Q3)

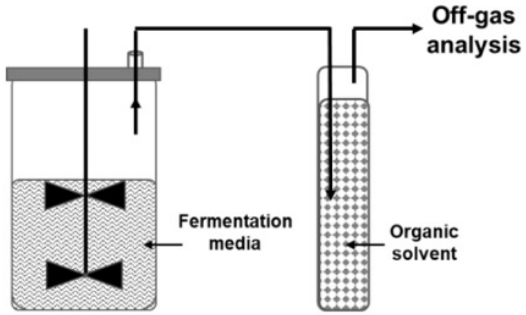
Progress and Outcomes

Developing fermentation test cases: key learnings

- Most volatile / hydrophobic products are toxic to the biocatalyst
- Extractive fermentation is the current SOT for toxic products
- Internal overlays impose separation challenges
- Volatilization from overlays is substantial
- For certain products volatile capture can eliminate overlays
- Losses from off-gas capture are an unresolved challenge
- Product volatilization does not always scale linearly

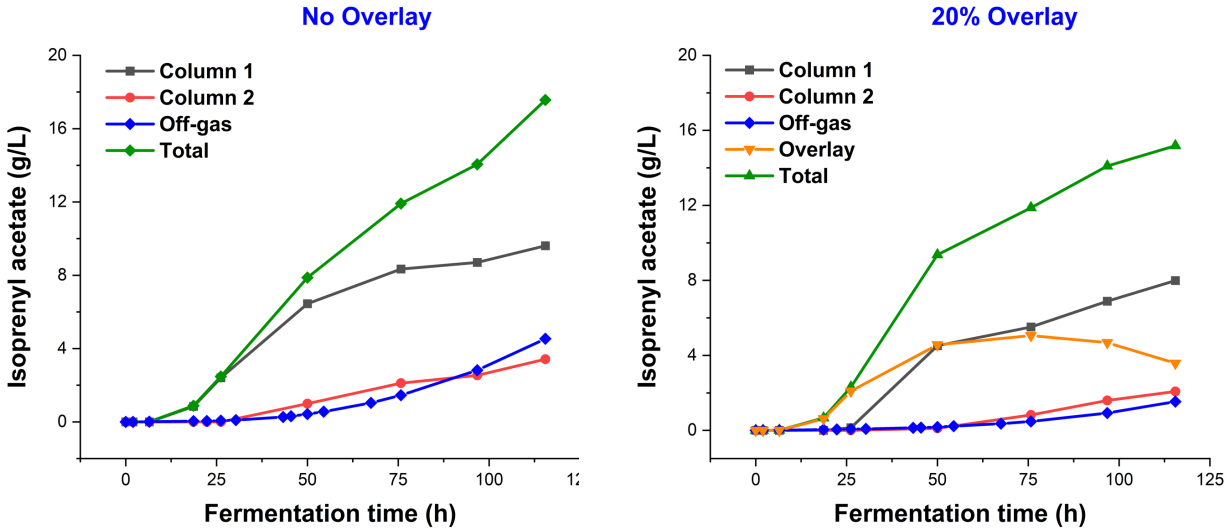


Extractive fermentation approach (baseline)

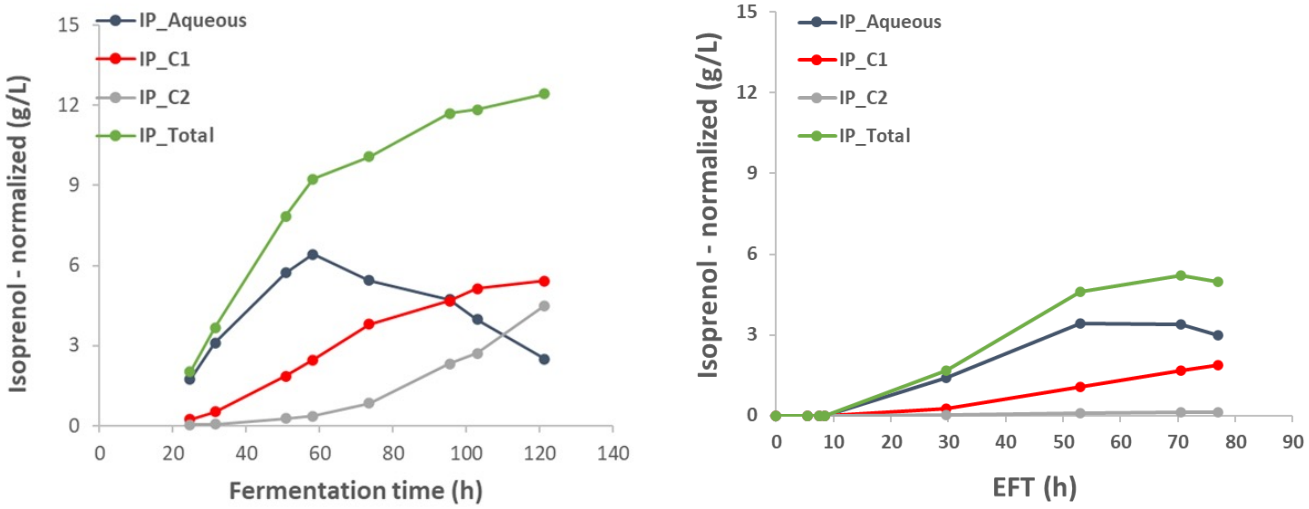


Volatilization approach

Isoprenyl acetate: 2L scale, 0.5 vvm aeration

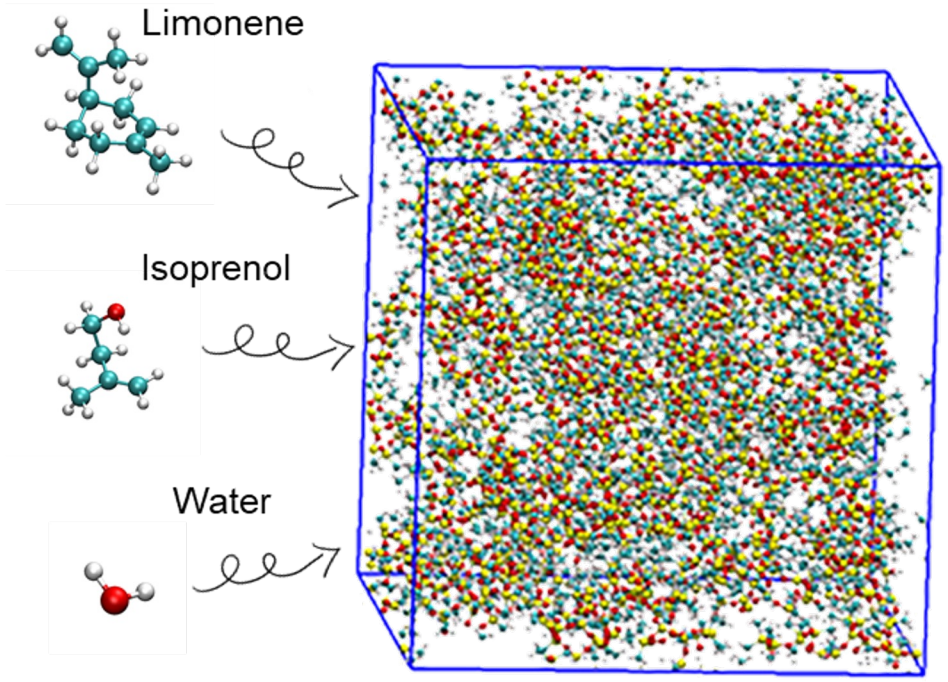


Isoprenol: 2L scale vs 300L scale, 0.5 vvm aeration

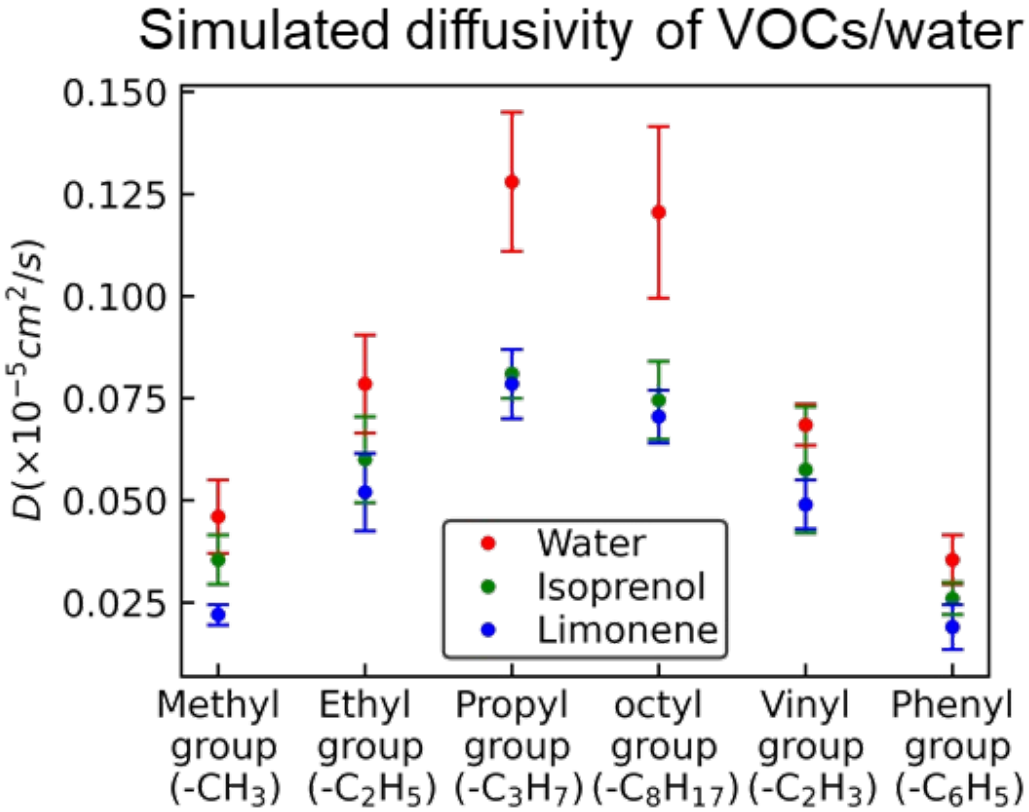


Strong integration with Computational Team

Model of xerogel structure used for calculation of binding energies and product diffusion throughout material



A polysiloxane model (6^3 nm^3)



A better understanding of surface interactions has led to improved materials capacities and streamlined processes, maximizing economic and environmental gains

Intertask teaming to improve industrial utility

- Expansion further into other SAF molecules/precursors
  - broaden class of fuel attributes and specificity requirements
- Integration with other BETO processes
  - leveraging strength of other consortia
  - utilize production strains and strategies found in SOT projects
  - Identify strong use cases for processes using C1-derived feedstocks
- Deploy next to other competing technologies for apple-to-apple comparisons (true benchmarking)
- Expand interactions with interested industrial collaborators
  - Amyris
  - Lanzatech
  - Industrial Microbes
  - Dow/DuPont
  - Praj
- Scaled demonstrations with three or more processes
- Broadcast availability to users with unique needs at the ABPDU

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amyris

LanzaTech capturing carbon. fueling growth.

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Benefit extensively from regular/inquisitive interactions with the IAB of the Separations Consortium

Planned milestones and Go/No-Go Decisions

Description	Criteria	End Date	
	Arrange for strain and process streams acquisition and transfer of processes from at least one BETO SOT project and one industrial collaborator.	12/31/2022	✓
	Compare capture efficiencies, energy expenditures, and costs for advanced materials capture versus condensation distillation and solvent overlays for two volatile SAF processes at 2 L scale or larger.	3/31/2023	*Underway*
Automate desorptive recovery from xerogel-based capture cartridges	Demonstrate automated xerogel cycling with inline desorptive processing demonstrating >75% recovery yields and >90% bioproduct purities.	6/31/2023	*Underway*
	Optimize modular cartridge designs to recover volatile SAFs or SAF precursors from > 200 L- bioreactors such that recoveries are >80% with prolonged production runs of at least 3 days	09/30/2023	*Underway*
	Quantify relative performance of internal overlay baseline vs. off-gas stripping approach for 2 or more SAF or SAF-precursor producing bioprocesses and demonstrate 20% increase in performance metrics (through increases in titers, rates or yields) with adsorptive technologies	06/30/2024	

Type	Description	Criteria	End Date	
Go/No-Go		Run 20 L or larger fermentations with advanced materials capture of 85% of total volatile products and desorption of at least 75% of product with < 5% water. Process should demonstrate 50% cost savings over condensation/distillation approaches.	03/31/2024	*Underway*

Impact: Direct response to an industrial need

- Advances over industrial separation technology
  - Planned demonstration of cost advantages over baseline technologies with recovery of multiple SAFs (or SAF precursors) at purities that would allow for immediate use
- Demonstration
  - Optimized concerted fermentation/recovery regimes leveraging full product removal via aeration to minimize costs and GHG impacts for aerobic bioprocesses and removal of toxic products at an integrated biomanufacturing level
- Industrial application
  - Leverage success with liquid-liquid product capture with volatiles, expanding industrial engagement and involvement with SAF foci
  - Enable *in situ* removal for toxic products and eliminate costly internal overlays
  - Evaluate approach for polishing/scavenging from other types of dilute off-gas streams from bioconversion processes
- Current/planned industrial engagement and demonstration
  - Industrial and government collaborations (e.g., Amyris, LanzaTech, iMicrobes, Dow/DuPont, and US Navy)
  - Additional processes tested at the Advanced BioProcessing Demonstration Unit at LBNL by industry partners
  - Leverage discovery within Energy i-Corps program and evaluation within LaunchPad Program at ANL (industry evaluation akin to Cyclotron Road at LBNL)
  - Technology additionally used/vetted in proposals submission to TCF program; recent interactions with US Naval Air Weapons Station China and Captis Aire.
  - Additional outreach planned for BIO IMPACT and the annual SIMB meeting.

Facile vapor phase recovery would broaden the suite of product classes available for biomanufacturing

Biomanufacturing interest:

DMC · Visolis (Carbon Negative Materials) · iMicrobes · RHO Renewables, Inc. · amyris · DuPont

Summary

- Our work in this area has solidified the Separations Consortium as a thought leader in addressing this emerging challenge.
- Validated novel technologies are now capable of being deployed for a wider range of volatile product classes, using engineered adsorbents with tunable surface chemistries to passively capture products from bioreactor off-gases.
- The xerogel approach, with scaled recoveries > 85%, works at bioreactor temperatures to capture bioproducts in high purity with minimal energy use.
- Success with the advanced materials approach now opens new opportunities to contribute to the success of biomanufacturing and to realize aerobic fermentation efficiencies by leveraging aeration for *in situ* product removal (in prolonged runs).
- Industrial interactions (>12 thus far) and government collaborators (3 currently) continue to grow/expand, especially those emphasizing SAF production/pathways.

Quad Chart Overview

Timeline

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

	FY22 Costed	Total Award
DOE Funding	(10/01/2021 – 9/30/2022) $500K	(FY23-FY25) $2025K
Project Cost Share *	N/A	N/A

TRL at Project Start: 3
TRL at Project End: 6

Project Goal

Validate at pilot-scale, and beyond, advanced, cost-effective material to capture volatile product(s) directly from bioreactor off-gases

End of Project Milestone

Demonstrate near-quantitative capture of bioproducts from ≥ 3 industrial-derived SAF-producing bioprocesses with purities allowing for immediate use, highlighting cost advantages of ≥ 50% over baseline technologies

Project Partners

- ANL
- LBNL
- Amyris
- Lanzatech
- Industrial Microbes
- Dow/DuPont
- Praj

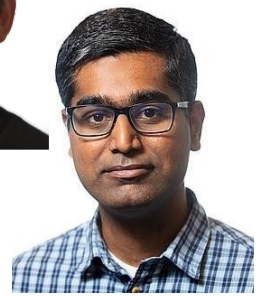
Diversity and collaboration within Volatile Product Recovery










- Expanding LCA team

Previous and planned DEI activities

In FY22, this team hosted two student interns and participated in five outreach events. In addition, outreach events are scheduled for grade and high school in FY23, FY24 and beyond through an ANL program; Recent 1:1 industry interviews that compared DEI in BETO consortium to DEI in industry – stressed early interactions; the volatiles team will interact in all phases of the Bioenergy-to-Bridge Program in all summers that it is available within the Separations Consortium.

Acknowledgements

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- Expanding LCA team

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- Pradeep Gurunathan
- Charlie Freeman
- Jian Liu

DMC

RHO Renewables, Inc.

Thank you!

VISOLIS CARBON NEGATIVE MATERIALS

Additional Slides

Responses to Previous Reviewers' Comments

- The exclusive focus on xerogel adsorbents was questioned.

The Volatile Product Recovery team has extensive familiarity with xerogel-based adsorption technology from projects using a similar approach for recovery of bioproducts directly from fermentation broths (liquid-liquid extraction). We decided to leverage this experience and the patented xerogel platform as the test case for adsorbent-based recovery of volatile bioproducts, while working with industry stakeholders and the SepCon analysis team to develop a systematic understanding of key use cases and alternative technologies for volatile product capture. The xerogel approach takes advantage of the unique properties of xerogel-based adsorbents (adjustable porosity, exceedingly high-surface-area, flexible solids with tailorable surface chemistries) to build out first-generation process schemes to test out the approach. It is highly likely that other adsorbents (e.g., those based on rigid organic frameworks or resin beads) will work as well, but these technologies would require additional solvents and/or distillation in desorption and recovery processes. Our soft materials offer the means of facile desorption and recovery from the adsorbent materials with simple compression, pressure, and/or temperature swings, while allowing recovery of the product in high concentration without the use of additional solvents.

- Recyclability of adsorbents should be considered in near time frame

Regarding cyclability/material longevity, we agree that the success of the xerogel-based adsorbent approach will require materials to be used in many cycles of adsorption/ desorption to amortize the cost of their syntheses over larger volumes of recovered bioproduct. While our initial efforts focused on capture and desorption efficiency, we are confident that our current xerogel-based materials could be cycled 10s to 100s of times based upon previous demonstrations with liquid-liquid recoveries. As a critical part of the baseline comparison efforts, the longevity of the xerogels in the gas-liquid recovery scenario will be determined and used to better understand tradeoffs between synthesis cost and xerogel longevity. These results will inform synthesis formulations, cartridge configurations, and desorption strategies that minimize synthesis cost while maximizing the lifespan of the adsorbent.

Publication, Patents, Presentations, Awards, and Commercialization

- The earliest studies of baseline technology applied to the isoprenol case is nearing completion and will form a stand-alone publication. The materials approach with integrated computation modeling is nearly the final draft stage and will wait to incorporate a few straggling experiments on optimized recovery and scaled validation (currently underway at ANL and LBNL).
- A utility patent for the use of xerogels in bioproduct recovery has been awarded: P. Ignacio-deLeon and P.D. Laible. SURFACTANT-TEMPLATED SYNTHESIS OF NANOSTRUCTURED XEROGEL ADSORBENT. USPTO 11,052,374 B2. Issued July 6, 2021.
- An invention report on volatile product recovery using an adsorbent-based cartridge has been submitted: P.D. Laible and N.P. Dylla. ANL IN-20-152. Adsorbent system design for capture of volatile products in biomanufacturing. November 30, 2020.
- Discussions held with and/or presentations given by Eric Sundstrom (Volatile Products Recovery Co-Lead) to ~20 prospective industrial partners on the use of xerogels in recovery of volatile bioproducts from their processes, facilitated by interactions at the Advanced BioProcessing Demonstration Unit at LBNL.
- An invention report describing the use of 3D printing to incorporate microporous structure into xerogel designs to facilitate recovery of product from fermentation off-gas streams at high flow rates is in the final stages of preparation.