bottle
changing the way we recycle
U.S. DEPARTMENT OF ENERGY

Bio-Optimized Technologies to keep Thermoplastics out of Landfills and the Environment

DOE Bioenergy Technologies Office (BETO) 2023 Project Peer Review

BOTTLE 6 – Characterization

April 3, 2023

Technology Session Review Area: Plastics Deconstruction and Redesign

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U.S. DEPARTMENT OF ENERGY | Office of ENERGY EFFICIENCY & RENEWABLE ENERGY
BIOENERGY TECHNOLOGIES OFFICE
ADVANCED MATERIALS & MANUFACTURING TECHNOLOGIES OFFICE

Overview: Characterization and its role in BOTTLE

Characterization is central to BOTTLE's mission

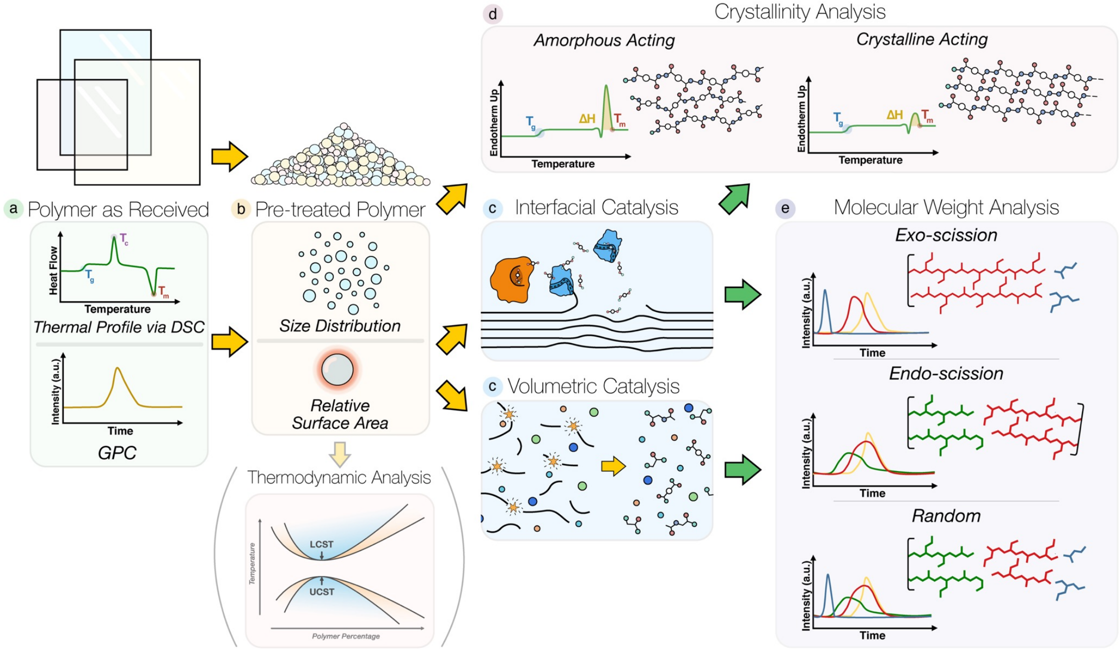
- Develop robust processes to upcycle existing waste plastics, and
- Develop new plastics and processes that are recyclable-by-design
- Detailed characterization is critical for developing efficient and effective processes and understanding the behavior of redesigned polymers

Activities in chemical, physical, and end-of-life studies

- Chemical and physical characterization takes advantage of SLAC and other BOTTLE partner core capabilities
- End-of-life testing capability is critical for redesigned polymers

Framework for characterization of polymer deconstruction proposed in 2021 review[1]

- Enables reproducibility across studies and research groups
- Finalizing characterization of research substrates for the community

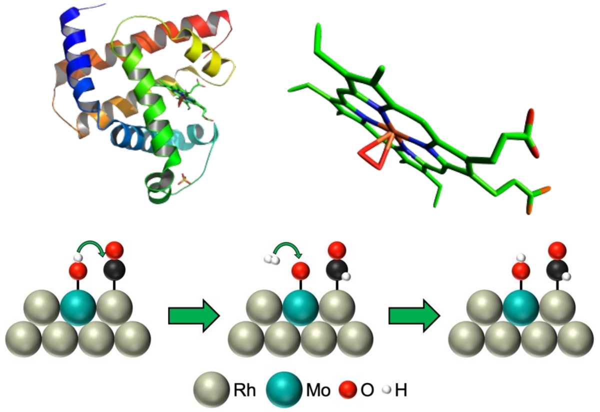


Approach: Mechanisms, structure, and EOL

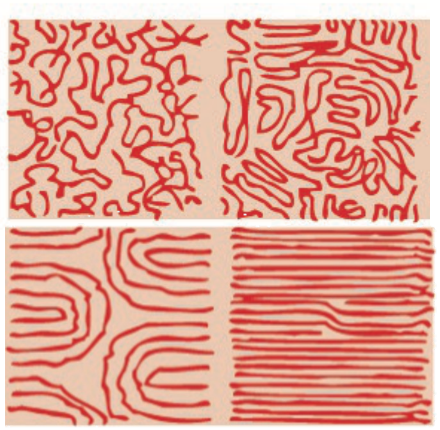
Three cross-cutting activities:

- **Mechanism determination**, quantification of products, and elucidating kinetics enables us to improve process efficiency, product selectivity, and process compatibility
- **Polymer structure** characterization used to determine relationships between polymer chemistry, and its associated structure and performance to improve redesigned polymers and design deconstruction processes
- **End-of-Life (EOL)** determines environmental impacts of redesigned polymers in natural (soil and fresh water) and engineered environments (landfill, composting, and anaerobic digester)

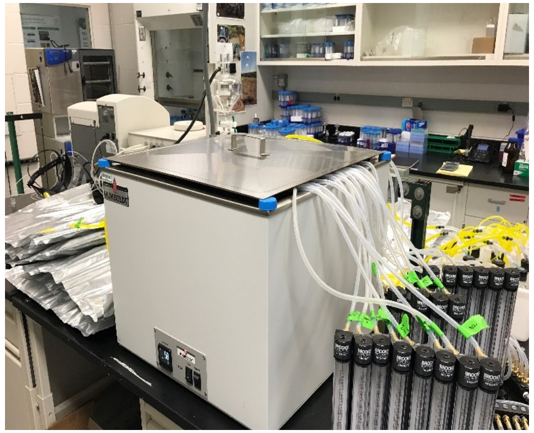
Mechanism Determination



Polymer Structure



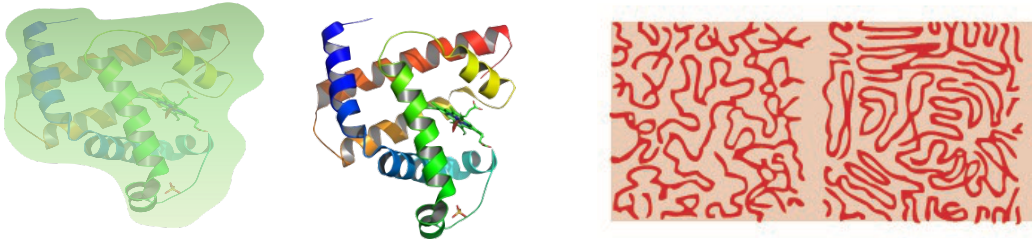
End-of-Life



Approach: Advanced characterization capabilities

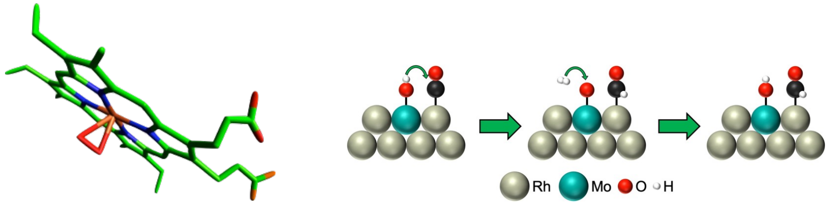
X-ray scattering

Multiple beamlines to characterize polymer substrate evolution during deconstruction, substrate-catalyst interactions, and structure-property relationships in redesigned polymers



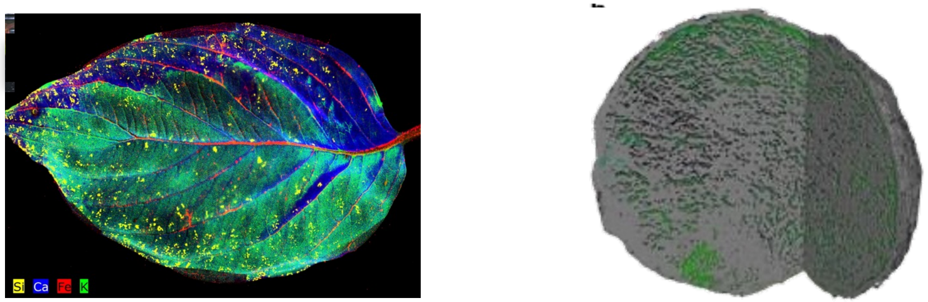
X-ray spectroscopy

Multiple beamlines to characterize the structural trajectory during reactions with fs resolution; elucidate mechanisms and describe the substrate-catalyst active electronic coupling



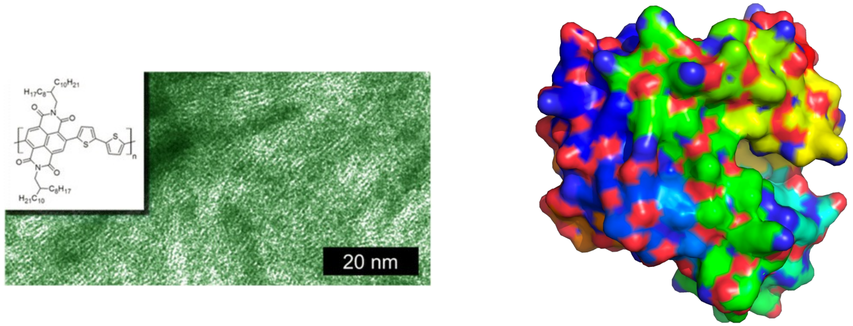
X-ray imaging

Imagine capabilities with resolution from nm to µm scales. Full spectral-tomographic reconstruction to visualize catalyst-substrate interaction to inform process design



Cryo-EM

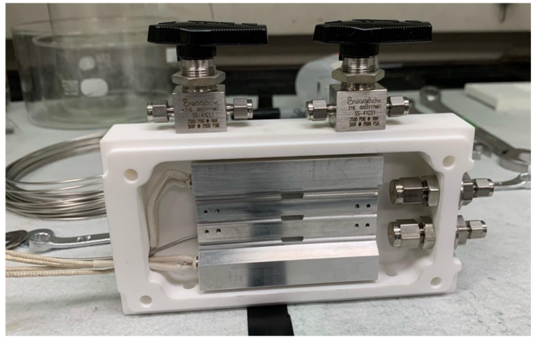
World-class cryo-EM capabilities with atomic resolution to determine structure in non-crystalline samples; imaging of amorphous polymers and structural work on biocatalysts



Progress and outcomes: developing sample environments

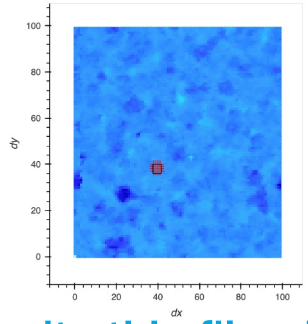
High-pressure reactors

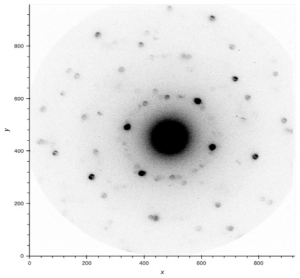
Developed reactors to characterize physical and chemical deconstruction mechanisms in realistic systems

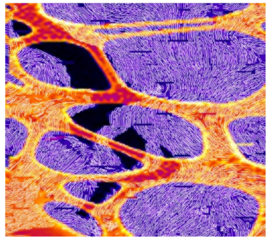


4D STEM

Developed data processing and interpretation pipelines to use 4D STEM to characterize molecular orientation of polymers over large length scales and understand the impacts of morphology to deconstruction chemistries and thermo-mechanical properties

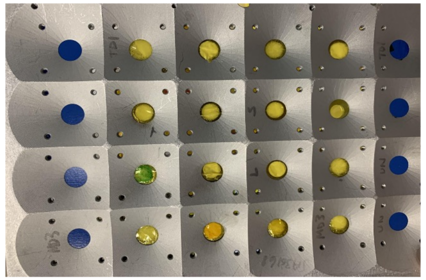


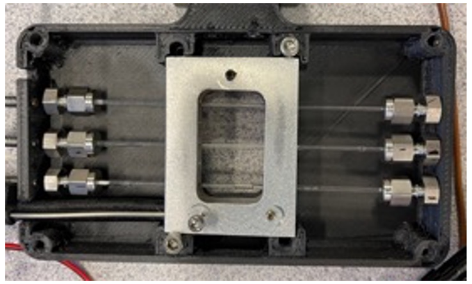




High-throughput measurements

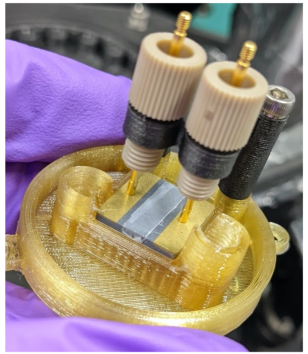
Developed sample environments to enable high throughput measurements of either ex-situ samples of commodity or redesigned polymers, or to rapidly scan process spaces

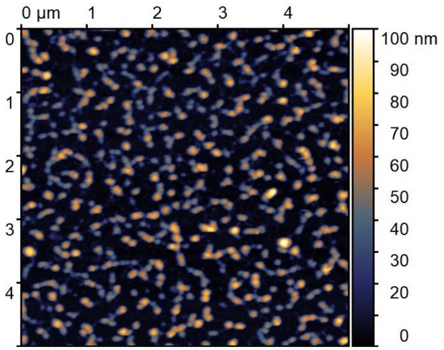




In situ thin film deconstruction

Developed model systems to understand the role of degree of crystallinity, crystallite size, and spacing on enzymatic PET deconstruction

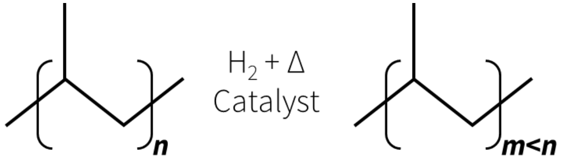




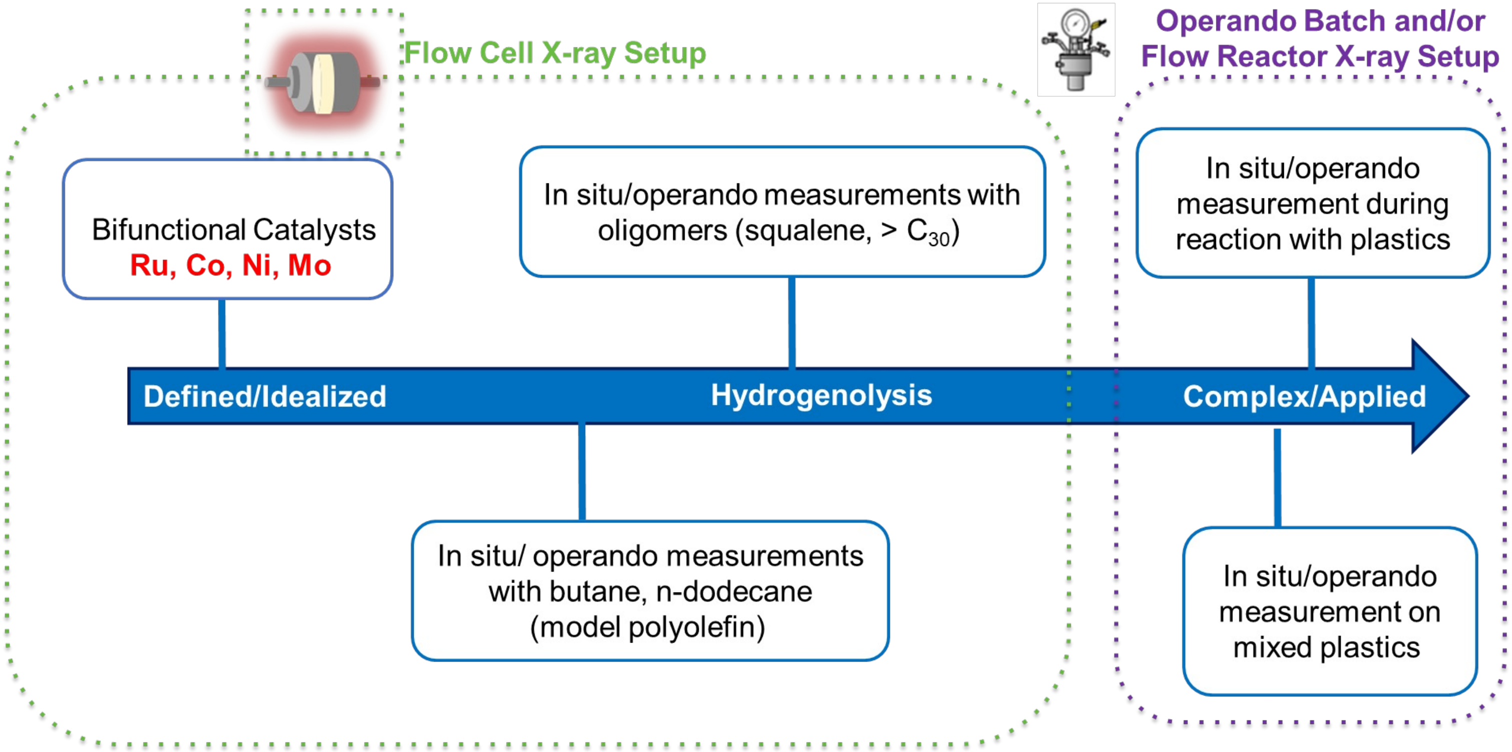
Operando heterogeneous catalytic deconstruction

Goal: Use X-ray Absorption Spectroscopy (XAS) of heterogeneous catalysts for deconstruction to answer key questions:

- What is the catalyst structure?
- What role does the metal play in the catalysis?
- Is the in situ/operando chemical state of the metal different across different samples?
- Does the metal structure change with TOS?



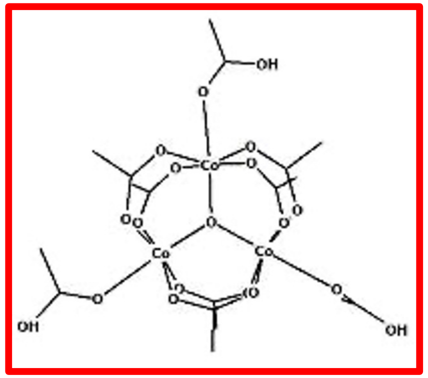
Sample	Onset Reduction Temperature (°C)	
	Hydrogen	n-dodecane + H2
Ni/ZSM-5	180	350
Ni/SiO_2	150	250

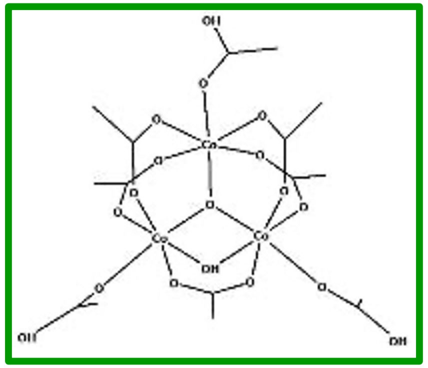


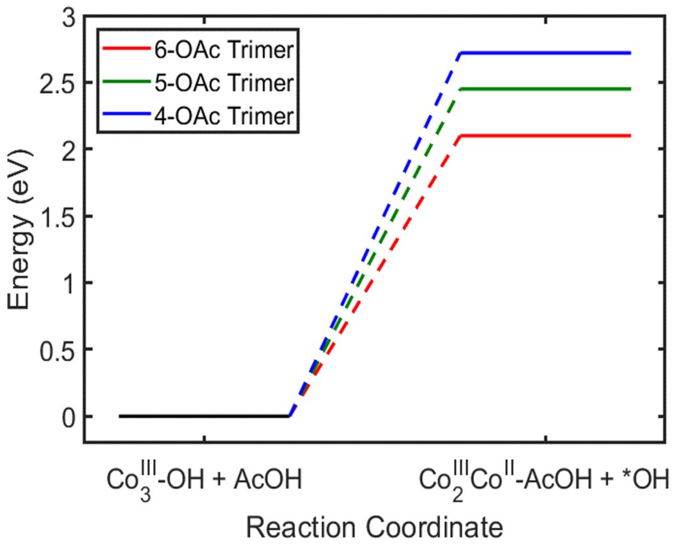
Impact: Understanding heterogeneous catalyst reorganization under reaction conditions is essential to the rational design of catalysts and processes

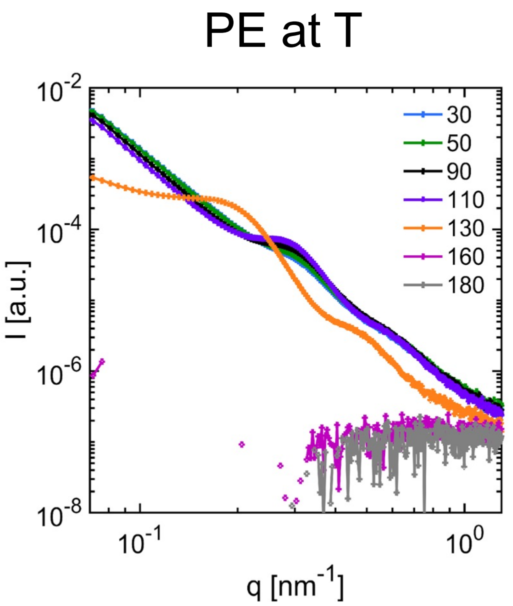
Characterization of oxidative deconstruction catalysis

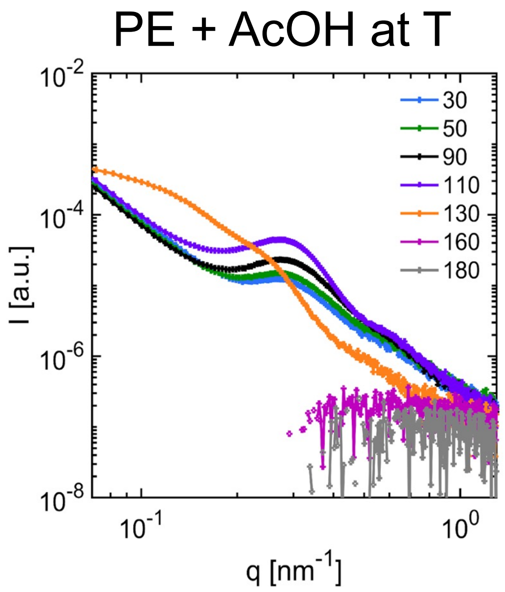
Goal: Identify the physical and chemical processes responsible for high activity observed in autoxidation









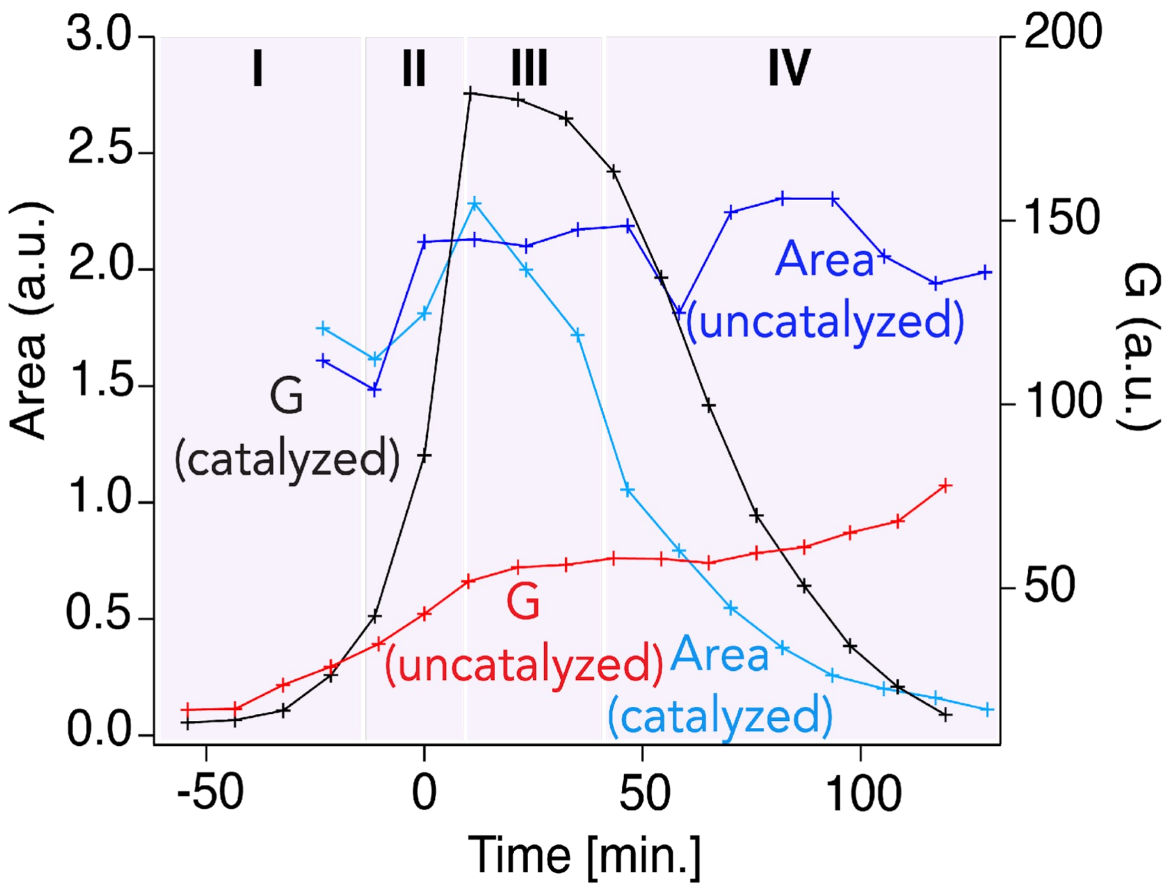


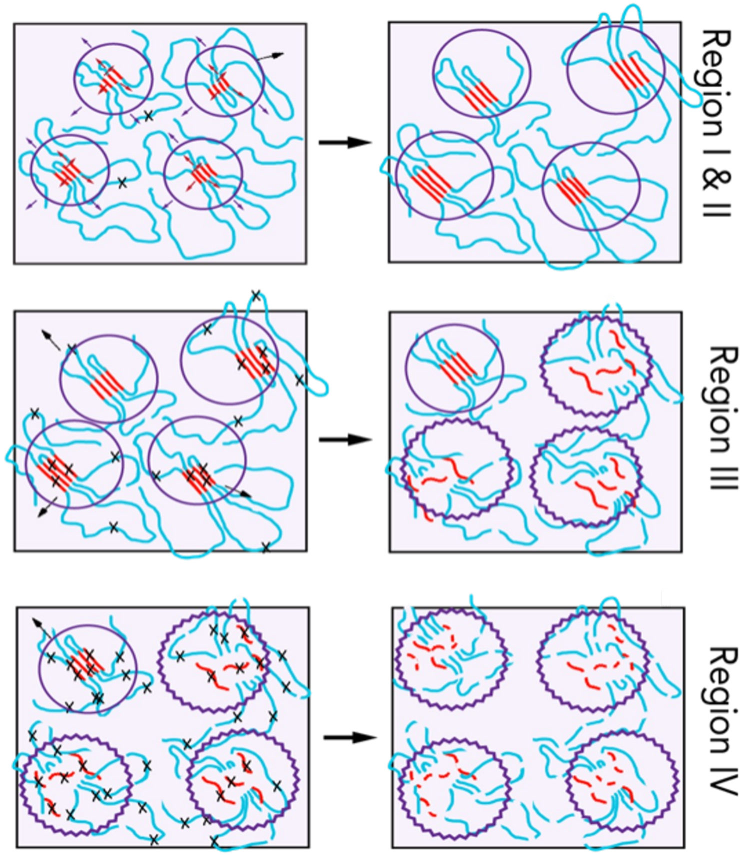
Outcome: Determination of key role played by the Co^{3+}, stabilized by AcOH ligands, in the reaction activity and how water content reduces activity

Outcome: Determination of the key role that acetic acid plays to diffuse catalyst into the solid polymer, turning what should be a surface limited process into a bulk deconstruction process

Characterization of PET glycolysis

Goal: Identify the physical and chemical mechanisms controlling activity in PET glycolysis





Outcome: Determined the key step in the deconstruction process is deconstruction within the amorphous interphase, likely at chain terminations, which then allows catalyst access to crystalline fraction of PET

Rorrer *et al.*, *in preparation*

Developing new methods for end-of-life testing (EOL)

Approach: Scaled-down standard methods used for EOL testing

Method Name	Temperature	Inoculum	Sample Size	Reactor size	Duration	Measurement
ISO 14851 - *Aerobic biodegradability of plastic materials in an aqueous medium*	23 °C ± 2 °C	Activated sludge taken from a WWTP	100 mg	300 mL	Min 28 days	BOD over time or CO_2
ASTM 5988 - *Aerobic Biodegradation of Plastic Materials in Soil*	23 ± 2 °C	Natural Soil	3 g	2 L	Max 2 years	Volume of CO_2
ASTM 5338 - *Aerobic Biodegradation of Plastic Materials Under Controlled Composting Conditions*	58 ± 2 °C	Mature compost	10 g	300 mL	Min 45 Days	Volume of CO_2
ASTM 5511 - *Anaerobic Biodegradation of Plastic Materials Under High-Solids Anaerobic Digestion Conditions*	37 ± 2 °C	Activated sludge taken from a stand- alone food waste digester	6 g	300 mL	15-30 Days	Volume of CO_2 and CH_4

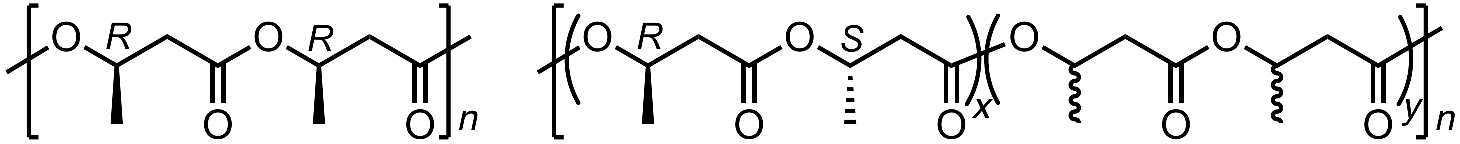
Goal: Determine polymer performance and sample fate in case of leakage into the environment.

Progress: 27 polymer samples have been received and tested

- 22 samples in freshwater
- 24 samples in soil
- 6 samples in compost
- 1 in an anaerobic digester

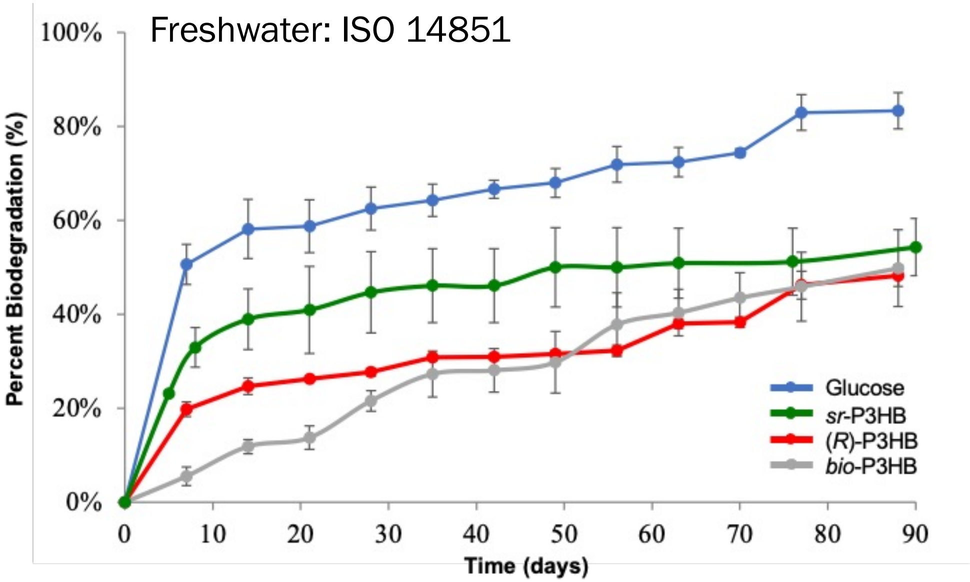
End-of-life (biodegradation) testing on designer PHAs

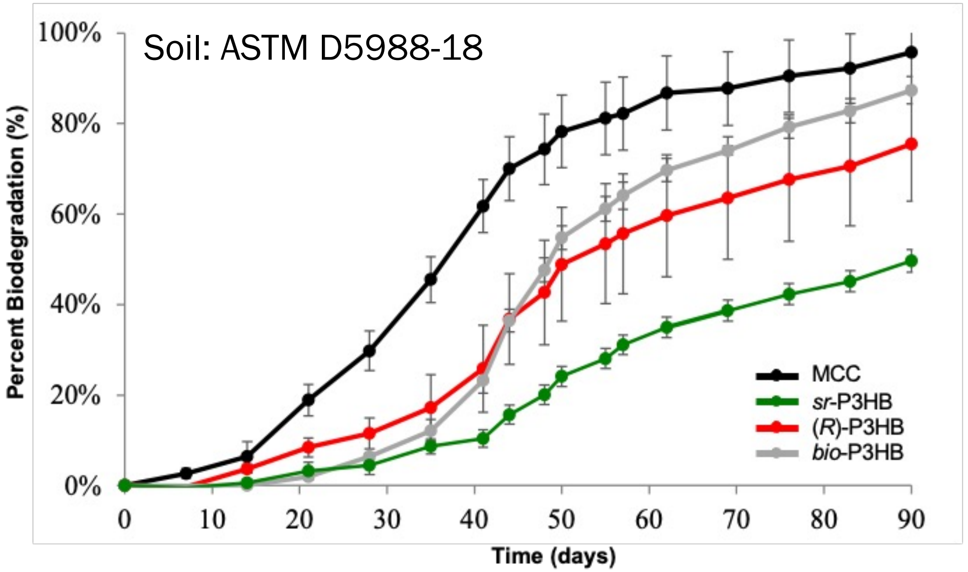
- **Background:** syndio-rich PHA, *sr*-P3HB, shows comparable mechanical properties to HDPE and PP
- **Outcome:** In freshwater: bio-P3HB (282 days) shows slightly higher biodegradation than *sr*-P3HB (383 days) and (*R*)-P3HB (433 days)
- **Outcome:** In soil: bio-P3HB (105 days) shows slightly higher biodegradation than (*R*)-P3HB (145 days) and *sr*-P3HB (268 days)



(*R*)-P3HB and *bio*-P3HB

sr-P3HB ($x > y$)





Quinn et al. *J. Am. Chem. Soc.* 2023

Beyond the standard EOL testing

Methods used to assess changes in polymer structure:

DSC	FTIR	GPC	NMR
SEM	XRD	Viscometry	

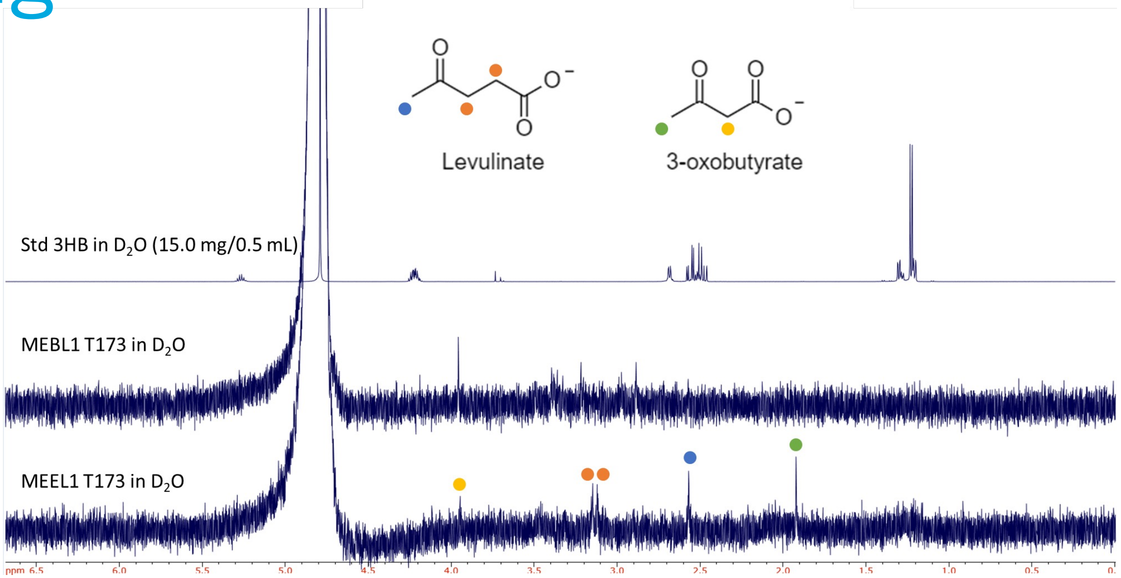
Methods used to assess plastic degradation:

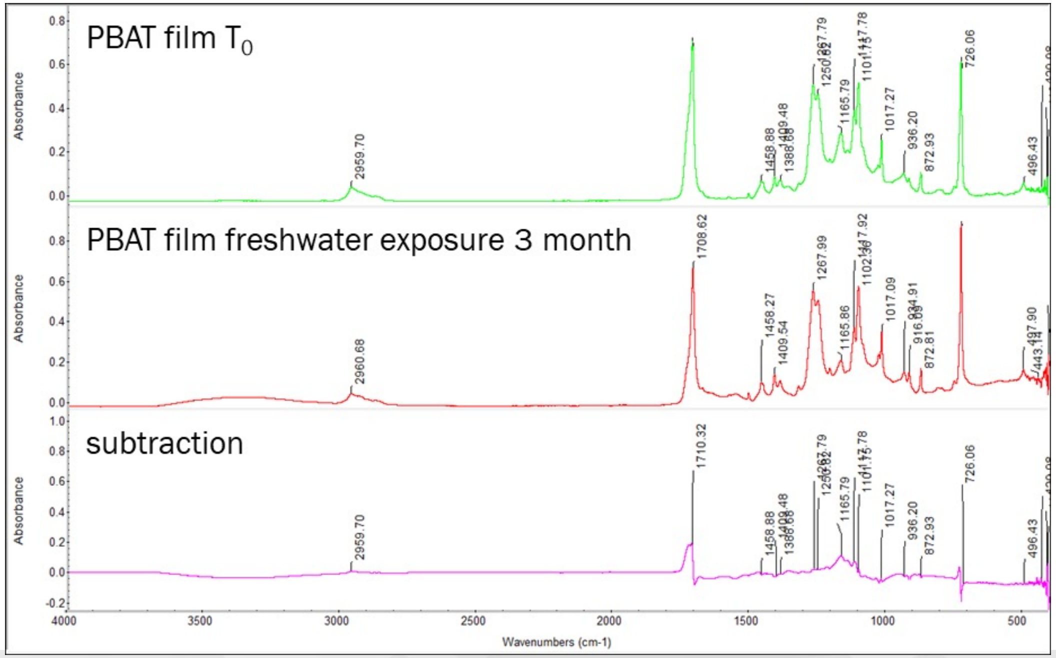
TGA	CO_2/CH_4 evolution

Methods used to assess generation of plastic metabolites:

GC	SEC	HPLC
LC-MS	FTIR	

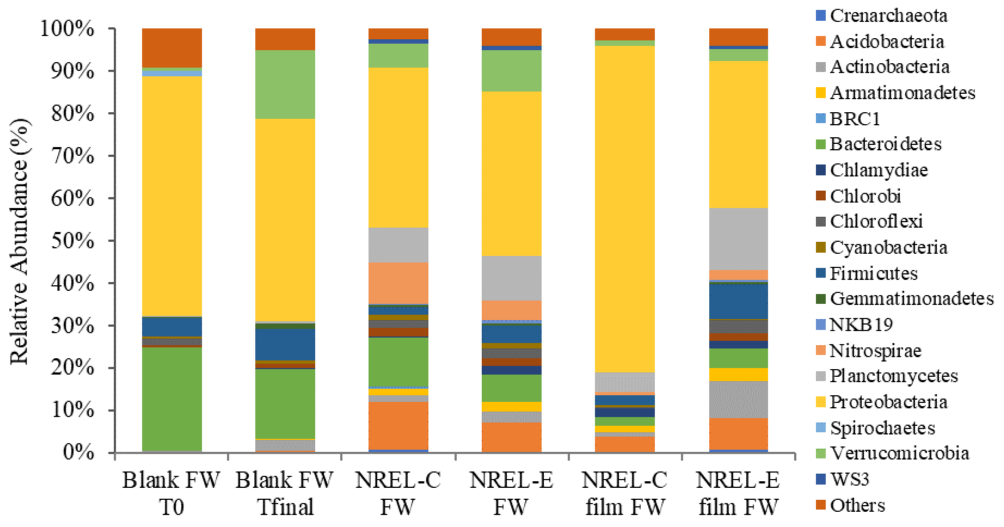
Future Work: combine methods used for changes in polymer structure, plastics degradation, and detection of metabolites

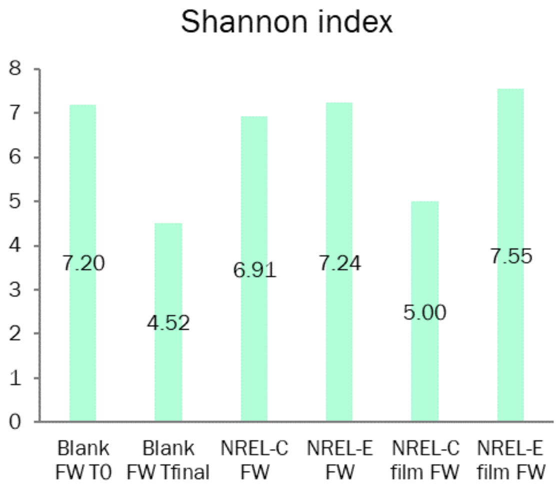




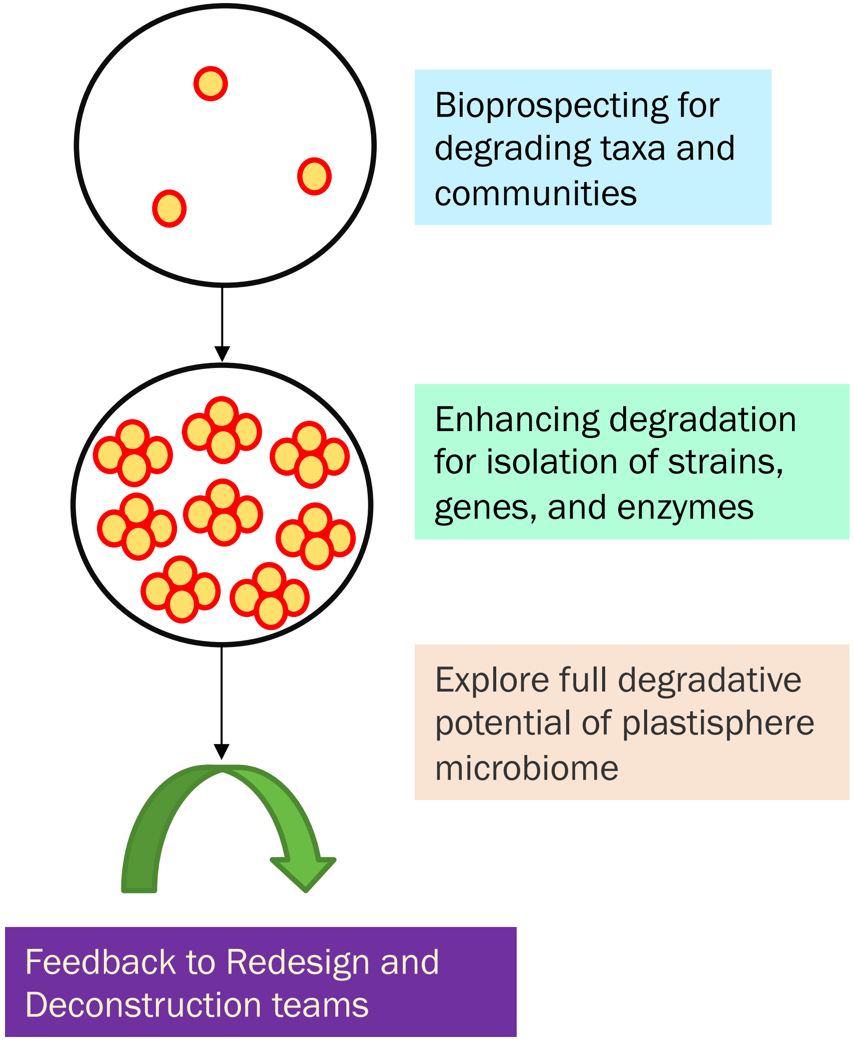
Enhanced polymer degradation and pathway discovery

- Elucidate dynamics and core constituents of microbial communities from samples showing biodegradation – 16 s RNA analysis





- Assess diverse plastisphere communities: abundance, functionality, spatial and temporal dynamics (pioneer species, colonization stages)
- Understand complex interactions between the degrading microbes and their corresponding genes, along with environmental conditions



Impact

Inform

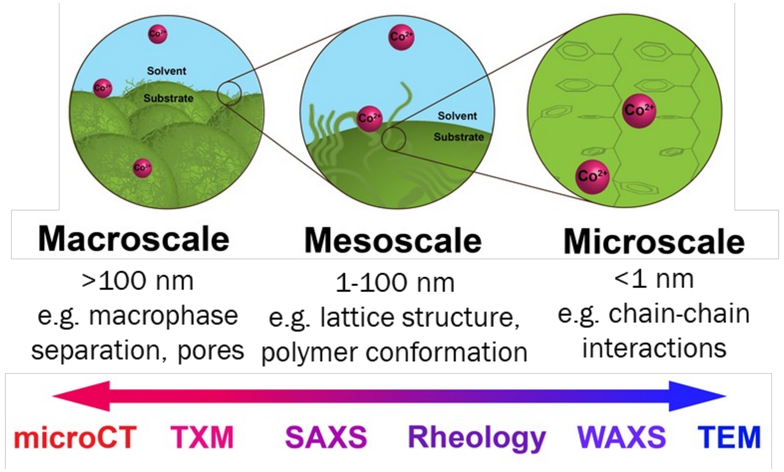
- Develop understanding of physical, chemical, and biological mechanisms that control deconstruction outcomes
- Determine the pathways into and the impact to environment for conventional and RBD polymers

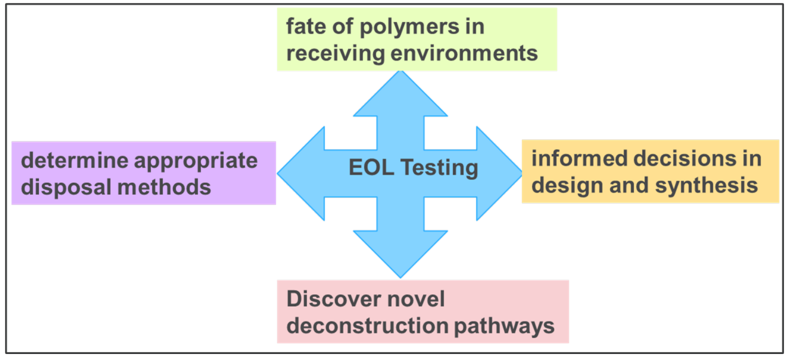
Guide

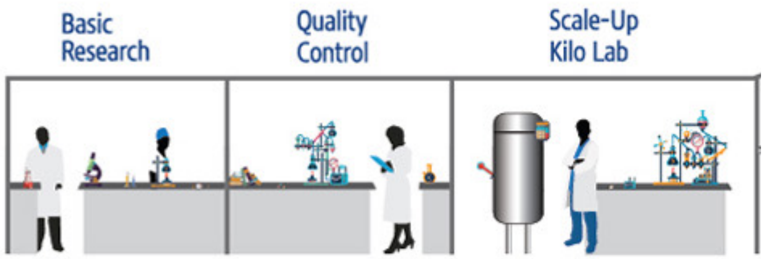
- Rationalize design of deconstruction processes, mitigations to contaminants, and scale considerations
- Bring EOL behavior as a property to design RBD polymers

Enable

- Scale-up of deconstruction chemistries, while accounting for real world post-consumer plastic waste streams
- Design of high performing polymers with minimum environmental impact
- Discover plastisphere microbiome and their genomes for deconstruction of plastic waste streams







Thank you!

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Publications

In preparation

Amy A. Cuthbertson, Clarissa L. Lincoln, Joel Miscall, Lisa Stanley, David T. Moore, Brenna A. Black, Nicholas A. Rorrer, Gregg T. Beckham, Characterization of polymer properties and identification of additives in commercial research plastics, in preparation.

Shaik Afzal, Avantika Singh, Scott R. Nicholson, Taylor Uekert, Eric C.D. Tan, Abhijit Dutta, Robert M. Baldwin, Gregg T. Beckham, Techno-economic analysis of mixed plastic waste gasification for the production of methanol and hydrogen, in preparation for *Energy Env. Sci.*

Rosie Graham, Erika Erickson, Richard K. Brizendine, Davinia Salvachúa, Zhongping Tan, Gregg T. Beckham, John E. McGeehan, and Andrew R. Pickford, Enzymatic deconstruction of poly(ethylene terephthalate) is not improved by the use of carbohydrate-binding modules at industrially-relevant solids loadings, in preparation for *Chem Catalysis*.

Julie E. Rorrer, Amani M. Ebrahim, Ydna Questell-Santiago, Clara Troyano-Valls, Arun S. Asundi, Simon R. Bare, Christopher J. Tassone, Gregg T. Beckham, Yuriy Román-Leshkov, Selective hydrogenolysis of polyethylene and polypropylene waste to liquid hydrocarbons over bifunctional Ru/acid catalysts, in preparation.

Geetanjali Yadav, Avantika Singh, Abhijit Dutta, Scott R. Nicholson, Kylee Harris, Calvin Mukarakate, Joshua A. Schaidle, Cody J. Wrasman, Yuriy Román-Leshkov, Robert M. Baldwin, Gregg T. Beckham, Techno-economic analysis of pyrolysis of mixed plastics waste, in preparation for *Energy Env. Sci.*

Submitted

Houqian Li, Robert D. Allen, Xianglan Bai, Gregg T. Beckham, et al., Expanding plastics recycling technologies: chemical aspects, technology status and challenges, submitted to *ACS SusChemEng.*

Publications

In review

Richard K. Brizendine, Erika Erickson, Stefan J. Haugen, Kelsey J. Ramirez, Joel Miscall, Davinia Salvachúa, Andrew R. Pickford, Margaret J. Sobkowicz-Kline, John E. McGeehan, Gregg T. Beckham, Effect of particle size and substrate crystallinity on enzymatic depolymerization of poly(ethylene terephthalate), in review at ACS SusChemEng.

Kevin P. Sullivan, Allison Z. Werner, Kelsey J. Ramirez, et. al., Tandem chemical oxidation and biological funneling for upcycling of mixed plastic waste, in review at Science.

In revision

Anelia Milbrandt, Kamyria Coney, Alex Baggett, Gregg T. Beckham, Quantity, distribution, market value, and energy value of plastic waste in the United States, in revision at Resources, Conservation, and Recycling.

Allison Z. Werner, Rita Clare, Thomas D. Mand, et. al, Tandem chemical deconstruction and biological upcycling of poly(ethylene terephthalate) to b-ketoadipic acid by Pseudomonas putida KT2440, In revision at Metabolic Engineering.

In press

Scott R. Nicholson, Julie E. Rorrer, Avantika Singh, Mikhail O. Konev, Nicholas A. Rorrer, Alberta C. Carpenter, Alan J. Jacobsen, Yuriy Román-Leshkov, Gregg T. Beckham, The critical role of process analysis in chemical recycling and upcycling of waste plastics, in press at Ann. Rev. Chem. Biomolec. Eng.

Publications

2023

Taylor Uekert, Avantika Singh, Jason S. DesVeaux, Tapajyoti Ghosh, Arpit Bhatt, Geetanjali Yadav, Shaik Afzal, Julien Walzberg, Katrina M. Knauer, Scott R. Nicholson, Gregg T. Beckham, and Alberta C. Carpenter, “Technical, economic, and environmental comparison of closed-loop recycling technologies for common plastics,” ACS Sustainable Chemistry & Engineering (2023) 11, 3, 965–978.

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Presentations

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Presentations

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High throughput test tools for industrially relevant microbial chassis, SIMB 2022, August 2022.

Circular polymers and biodegradable plastics, Circular Polymers and Biodegradable Plastics International Research Training Group, University of Muenster, July 2022.

Engineering P450s to alleviate a bottleneck to lignin demethylation, Intl. Conference on Porphyrins and Phthalocyanines, July 2022.

Difficult to recycle plastics, Sustainable Packaging Coalition Engage Meeting, July 2022.

Selective chemical recycling of mixed plastics waste, Polymer Physics Gordon Research Conference, July 2022.

Plastics recycling and upcycling in the BOTTLE Consortium, NASEM Committee on Repurposing Plastic Waste, July 2022.

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Multi-Material Flexible Packaging Coalition SPC, February 2022.

Presentations

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Development of chemical recycling approaches for plastic waste, Enzyclic Consortium (via webinar), January 2022

Development of chemical recycling approaches for plastic waste, UIUC, December 2021

Design Principles and Synthetic Methodologies for Circular Polymers with Intrinsic Recyclability and Tunable Properties, Pacifichem Conference, December 2021

New building blocks for performance-advantaged renewable and recyclable polymers, Pacifichem (via webinar), December 2021

Discovery and characterization of PET degrading enzymes, University of Rochester microplastics workgroup seminar series, December 2021.

Design Principles and Synthetic Methodologies for Intrinsically Circular Polymers and Biodegradable Plastics, Columbia University, November 2021

Selective Hydrogenolysis of Polyethylene and Polypropylene to Liquid Alkanes over Tunable Ruthenium-Based Heterogeneous Catalysts, 2021 AIChE National Conference, Boston, MA, November 2021.

Plastics recycling and upcycling, ACS Converge (via webinar), October 2021

Presentations

Genetic tools and microbial engineering for biological production of sustainable fuels and chemicals, Presented to Weekly Seminar for DOE CCI/SULI Students. October 2021

Heterogeneous Catalytic Deconstruction and Upcycling of Waste Polyolefins, Biodesign Institute at Arizona State University, SM3 Seminar Series, October 2021.

Domestication of diverse non-model microbes for plastics upcycling and sustainable fuel and chemical production, Biological Sciences Departmental Seminar, Michigan Technical University. October 2021.

Catalysis for valorization of lignin and plastics, Great Plains Catalysis Society (via webinar), June 2021

The critical role of economic and environmental analysis to guide research in lignin valorization and plastics upcycling, Keynote Invited Lecture, ACS Green Chemistry and Engineering (via webinar), June 2021

Towards Intrinsically Circular Thermoplastics and Reprocessable Thermosets, Dow Chemical Company, virtual seminar, May 2021

Recent progress in performance-advantaged bioproducts and plastics upcycling, Arizona State University (via webinar), April 2021

Recent adventures in biomass conversion and plastics upcycling, Rutgers University (via webinar), April 2021

Presentations

Recent adventures in biological plastics upcycling, MIX-UP Consortium (via webinar), April 2021

Framing challenges and opportunities for chemical recycling of waste plastics, ACS Presidential Symposium on Chemistry and the Future of Plastics (via webinar), April 2021

Recent updates in plastics upcycling from the BOTTLE Consortium, ExxonMobil Research and Engineering, April 2021

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Heme and non heme iron enzymes and renewable carbon, University of San Antonio Texas, April 2021

A flexible kinetic assay efficiently sorts potential biocatalysts for BHET hydrolysis, Symposium on Biomaterials, Fuels, and Chemicals, April 2021

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Design Principles for Circular Plastics with Tunable Properties, CellPress LabLinks: The Circular Plastics Economy: Linking Across Scales, virtual event with 440 registered attendees. March 2021.

Process analysis for enzymatic PET recycling, Global Research and Innovation on Plastics annual meeting (via webinar), March 2021

Presentations

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Biological processes for lignin and plastics conversion, University of California Riverside (via webinar), January 2021