PERFORMANCE-ADVANTAGED BIOPRODUCTS, BIOPROCESSING SEPARATIONS, AND PLASTICS

TECHNOLOGY AREA

CONTENTS

INTRODUCTION

The Plastics and Performance-Advantaged Bioproducts Technology Area is one of 12 technology areas reviewed during the 2023 Bioenergy Technologies Office (BETO) Project Peer Review, which took place April 3–7, 2023, in Denver, Colorado. A total of 48 presentations were reviewed in the Plastics and Performance-Advantaged Bioproducts session by 8 external experts from industry, academia, and other government agencies. For information about the structure, strategy, and implementation of the technology area and its relation to BETO's overall mission, please refer to the corresponding Program and Technology Area Overview presentation slide decks (www.energy.gov/eere/bioenergy/2023-project-peer-review).

This review addressed a total U.S. Department of Energy (DOE) investment value of approximately $68.3 million, which represents approximately 12% of the BETO portfolio reviewed during the 2023 Project Peer Review. During the Project Peer Review meeting, the presenter for each project was given 20–30 minutes to deliver a presentation and respond to questions from the review panel.

Projects were evaluated and scored for their approach, impact, and progress and outcomes. This section of the report contains the Review Panel Summary Report, the Technology Area Programmatic Response, and the full results of the Project Peer Review, including scoring information for each project, comments from each reviewer, and the response provided by the project team.

BETO designated Coralie Backlund as the Performance-Advantaged Bioproducts Technology Area review lead, with contractor support from Jessica Phillips of Boston Government Services. In this capacity, Coralie Backlund was responsible for all aspects of review planning and implementation.

PERFORMANCE-ADVANTAGED BIOPRODUCTS REVIEW PANEL

Name	Affiliation
Michael Mang*	Danimer Scientific
Vera Schroeder	Safar Partners Venture Capital
Alper Kiziltas	Amazon
Debbie Mielewski	Dione Solutions

* Lead Reviewer

PLASTICS DECONSTRUCTION AND REDESIGN REVIEW PANEL

Name	Affiliation
Sharon Haynie*	Independent Consultant
Vera Schroeder	Safar Partners Venture Capital
Michelle Seitz	American Association for the Advancement of Science Fellow
Margaret McCauley	U.S. Environmental Protection Agency
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* Lead Reviewer

PERFORMANCE-ADVANTAGED BIOPRODUCTS AND PLASTICS REVIEW PANEL SUMMARY REPORT

Prepared by the Performance-Advantaged Bioproducts and Plastics Deconstruction and Redesign Review Panels

INTRODUCTION

The review panel members brought a range of experience in different technology areas, from basic science to application development and finance, which provided an excellent cross section of perspectives to the assessments. The panelists' disciplines include biocatalysis, chemical synthesis, chemical engineering, environmental engineering, and materials science. The review panel's research and work experiences cover the sustainable synthesis of monomers and polymers, venture capital funding, environmental assessment, metabolic engineering, polymer processing, and polymer characterization.

The review process was well coordinated, and the panel was able to review a large number of programs and projects quite efficiently. The tools available for the review process worked well. The presentation template used facilitated the flow of each project, making it particularly easy to assess the projects' objectives and progress. The presentations incorporated previous suggestions about including technology readiness levels (TRLs) and process diagrams, making them more effective. The in-person format allowed for good question and response periods, and good discussion was had throughout.

In a relatively short time, the technology manager actively engaged with the projects and exerted the requisite technical knowledge and managerial skills to oversee the work in this relatively large and complex portfolio. The 3-day Project Peer Review session was coordinated well. Each project team was provided with a presentation slide deck to ensure the presence of some key unifying elements among the presentations—specifically, the quad chart, strategy, approach, progress, and impact slides. The in-person meeting allowed adequate time for the review panel (and the general public) to engage in a Q&A with the presenters.

The projects presented to the review panel showed impressive breadth and depth across a broad range of technology areas, demonstrating effective implementation of the program strategy and good scientific and technological progress across the effort. The portfolio has been shaped by a road map formulated from roundtables and then refined with input from many stakeholders from various sectors (advocacy, academia, government, and industry). The value chain of industry partners includes producers, suppliers, waste collectors, and processors. The project themes follow the recommendations that emerged from the roundtable, and projects fall into the categories of deconstruction, upcycling, recycling by design, and scaling and deployment.

STRATEGY

The Performance-Advantaged Bioproducts and Bioprocessing Separations strategy has been to define and develop new fuels and chemicals that provide a genuine environmental benefit over incumbent petroleum-based products, such as an improved carbon footprint. By considering an integrated approach from feedstock to application, the program is able to develop technologies that have a higher likelihood of industrial success and that will meet the strategic goals of the program. The specific targets as they relate to bio-based aviation fuels, new chemical products, and plastics are clearly articulated and relevant to current technological problems. The structure of the Separations Consortium is an excellent example of a focused technology area that exhibits how the strategy is intended to be deployed.

The program design considers input from stakeholders from national laboratories, academia, and industry to design the program strategy and direct the management of the program to the greatest effect. A greater level of broad industry and academic input at the beginning of projects—with the aim of improving the target selection for industrially practical topics—would be valuable, especially in the bioproducts area. In the current program, once the topics are established, there are overall good interactions with industry partners, which helps maintain a focus on commercial acceptance and implementation. There is a good portfolio approach to the program with projects at various stages of development as well as technical risk. The use of techno-economic analysis (TEA) and life cycle analysis (LCA) is valuable, but some effort to standardize how those analyses are carried out will improve their utility in future project designs and assessments. The program is using appropriate funding mechanisms for the various projects with combinations of lab calls (as part of the annual operating plan) and funding opportunity announcements (FOAs).

This technology area has a clear and focused strategy with its own specific goals and technical targets. The strategy is well developed with input from all key stakeholders (industry, advocacy, academic, and government). Since 2021, the plastics technology area has expanded its objectives and sharpened its metrics. First, a specific objective around greenhouse gas (GHG) emissions has been added: designing recycling strategies that mitigate ≥50% of GHG emissions relative to virgin resin or plastic intermediates. Second, addressing the end-of-life fate now has a quantitative target metric and will address end-of-life fate for ≥90% of all plastics (replacing “most”).

This technology area funds a mix of early-, mid-, and later-stage programs; the split between the FOA and annual operating plan funding mechanisms seems appropriate. A casual analysis of project descriptions suggests that there is a reasonable number and diversity of projects in the key goals outlined for the strategy for plastics innovation: deconstruction, upcycling, recycling by design, and scaling and deployment; however, one reviewer recommends that the managing agencies (DOE/BETO) take an additional step and map the current portfolio by keyword metrics. For example, for project focus, that could include new material development or deconstruction and upcycling process development, type of polymer, application, and waste stream. Scrutiny of the projects in this manner may reveal whether there are funding gaps and whether the appropriate mix of activities is being pursued by BETO. Moreover, if this analysis included other DOE technology areas that fund plastic circularity activities (e.g., Advanced Materials and Manufacturing Technologies Office [AMMTO]), BETO could seek and monitor mutual activities (communications, collaborations) across these technology areas that may be beneficial.

STRATEGY IMPLEMENTATION AND PROGRESS

The technology area is funding a strong cross section of projects that strongly ties to the strategic goals of the program. There are three primary technology objectives outlined for the program in aviation biofuels, new chemical products, and polymers. The funded projects effectively address some aspects of each objective. The adsorptive denitrogenation project was rated highly by the review panel and has an excellent approach with excellent alignment with the aviation biofuel objectives. The effort to broadly include TEA and LCA is particularly valuable. Any advances made in the broad area of bioproducts require three elements: technical performance, economic viability, and environmental benefit. Including the TEA and LCA helps ensure that the technical work does not proceed too far without proper consideration of the economic and environmental performance. Further integration of those methods will help ensure that future work is being done to most effectively further the strategic goals of the technology area. Artificial intelligence (AI) and machine learning (ML) may be technologies that are too immature to provide value to current efforts.

The Separations Consortium is well structured to advance the strategic objectives of the technology area, with an excellent collaborative nature and very good interactions with industry partners. The impact of the industry advisory board’s (IAB’s) input is apparent, with good alignment of projects toward industrially relevant technology objectives. The biobutanol project is a particularly good example of this, showing how the integration of separation technologies with front-end biological manufacturing can demonstrate considerable technical value along with reduced costs and lower emissions.

The projects funded by the technology area represent a broad portfolio of project maturities, from early stage to the small pilot scale. The portfolio approach helps mitigate the technical risk associated with the early-stage projects. The work across projects can be described as leading edge and is consistent with high-quality technical efforts throughout. One good example is the volatile products recovery project, which scored highly on both approach and impact.

The projects individually showed good progress. Some early-stage projects had only a few months of effort, but progress was apparent, and the later-stage projects (some of which were essentially completed) showed that the individual project objectives had been met. Because of the design of the portfolio and the way risk mitigation strategies have been employed across the program, progress against strategic objectives has a good probability of success in both the short term and the medium term. The longer-term industrial acceptance of many of these products and technologies is still uncertain, but the perspective carried forward with the integration of TEA and LCA with input from industry partners will increase the likelihood of success.

The projects are also well managed both individually and at the program level to provide beneficial outcomes for the performer and the government. The overall program approach to project management is very active, with regular review, industry and academic partner input, and good use of go/no-go points as checks against progress keeping the projects on track toward completion. There is better industry engagement with the Separations Consortium than with the Performance-Advantaged Bioproducts (PABP).

The projects are well aligned with the strategic objectives of the technology portfolio. Only a single project (Highly Recyclable Thermosets for Lightweight Composites) was flagged as one that may better fit with another technology office. The reviewers were impressed with most projects. The scores and the individual comments supported project ratings where strengths exceeded (or far exceeded) project weaknesses. In general, the range in panel review scores aligned with comments on a particular project, and the panel comments were more complementary than divergent. Most projects in the plastics portfolio with scores trending below the Project Peer Review session average were early-stage projects. Although the diversity, equity, and inclusion (DEI) activities across projects in this portfolio were not uniform in their depth and breadth, most project teams did an adequate job of addressing and describing DEI activities.

The portfolio is examining the deconstruction of a diverse array of plastic waste compositions (e.g., polyester [polyethylene terephthalate, or PET], polyurethanes [PUs], and polyolefins [polyethylene, or PE, and polypropylene, or PP]). The material forms were also diverse: multilayer films, single-material plastic, composites, waste flake, and fabric (post-consumer waste and industrial waste). The reviewers commend the addition of PET fabrics to the waste stream, as this was an important target gap that was highlighted in the 2021 Plastics Project Peer Review. The deconstruction strategy featured projects with diverse technologies, including thermal, pyrolysis, chemical, and biological options—a multipronged strategy that allows greater leeway to choose the most promising and efficient path. Within the plastics portfolio, there are early-stage and novel technologies represented with an opportunity to demonstrate proof of concept. The Bio-Optimized Technologies to Keep Thermoplastics out of Landfills and the Environment (BOTTLE™) Consortium is a crown jewel in the BETO portfolio, and the panel was aligned in assigning high scores for each area (management, approach and implementation, and impact) for nearly every BOTTLE Consortium presentation. The review panel applauds the quick sunsetting of the technologies that did not fulfill expectations (e.g., the photochemical and electrochemical deconstruction approaches described in BOTTLE 3).

LCA/TEA and industry engagement were key focal points in this Project Peer Review. The strong consensus among the reviewers is that the integration of LCA and TEA early on in a program increases the probability of better project outcomes. Better engagement of industry members (through IABs) was a recommendation from the 2021 Project Peer Review panel. This recommendation is validated because this 2023 Project Peer Review panel sees a correlation between impact and a potential path to commercialization when there is early, active engagement by an industry partner.

This Project Peer Review panel feels that the current portfolio of projects is likely to meet most of the stated near-term and medium-term goals in the four goal areas under the plastics strategy. The detailed project commentaries support this overall view of the progress in this portfolio. In this high-level summary, we offer five projects that are representative of the spectrum of progress toward the goals. The review panel was enthusiastic about the progress made in the Lubricating Oils From Upcycled Plastics (LOUPs) project, which converts single-use polyolefins into higher-valued lubricating oils. The LOUPs team was noted for performing parallel LCA/TEA, and the team's early success has yielded a spinoff company. Additionally, the LOUPs project team had a clear vision and addressed nearly all the elements in the value chain (additives, processing considerations, and end applications). For the goal of recyclability by design, the panel recognizes the new chemistry achievements of the Responsible Innovation for Highly Recyclable Plastics (ResIn) project. The project team addressed the concerns raised in the 2021 Plastics Project Peer Review and is on track to deliver bio-based PU derivatives that are moving toward the portfolio target metrics. In addition, the tunable PU derivatives bring functional value to engaged industry partners. Another recyclable-by-design project that is on the leading edge of science discovery work is supported in the BOTTLE redesign and modeling work—in particular, the redesign by Eugene Chen and colleagues to create chemically circular melt-processable polyhydroxyalkanoates with physical properties similar to those of other polymer classes. This project has related BETO-funded work that was recently featured in *Science* (https://doi.org/10.1126/science.adg4520) and rebroadcast in other technical trade journals. The IBM-led project to upcycle PET via the volatile catalyst (VolCat) process is a higher-TRL development project with a clear commercial goal. Its target product, bis(2-hydroxyethyl terephthalate) (BHET), can plug into existing infrastructure. This program has tested real PET industrial waste streams (flake and fabric) and is focused on the right elements to pilot the process within the next year. For a project targeting a long-term deconstruction goal, the panel was intrigued by the novelty and potential of AMO.01—which deploys microbial spores that were evolved to survive higher temperatures and to grow on a thermoplastic polyurethane (TPU). Although the presence of the evolved spore strains has not shown any significant impact on TPU degradation rates, the team has demonstrated considerable progress in their biological work. The progress to date is consistent with the expectation that advancing biological systems for deconstruction is a long-term goal (up to 10 years).

These final three comments around strategy implementation cut across the portfolio:

- The review panel suggests two additions to the presentation content. The reviewers noted that there were gaps or a lack of clarity around how the industry partners were engaged in many projects. The panel felt that it would benefit the review process if BETO were to establish a rubric for categorizing industry participation (e.g., advisory, collaboration, funding, material or prototype suppliers). This industry engagement descriptor could be placed on the quad chart. Second, the reviewers urge that all project teams (a) supply a Gantt chart with tasks, milestones, go/no-go decision points, and project quarters; and (b) include an absolute timescale on the chart (e.g., overlay with the program years).

- An alternative view was offered on the cooperative research and development agreement (CRADA) template for industry engagement with the BOTTLE Consortium. Specifically, the reviewer stressed that exclusive licenses are a barrier to pushing technologies out for broad deployment and could compromise the achievement of objectives. It was suggested that BETO should explore alternative licensing models and not offer exclusive licenses to single companies if the work is partly government-funded.

- A reviewer raised a concern that polymer processing considerations in early projects within the portfolio were largely absent. Chemistry-push projects should be integrated with expert guidance on polymer processing, as it is important to consider whether the new materials can feed into existing infrastructure or if new infrastructure will be required.

RECOMMENDATIONS

Recommendation 1: Independent assignment of TRL.

We recommend that an entity outside the team—e.g., the technology manager—assess/assign the TRL for each project in the portfolio. There was general skepticism among the reviewers about the accuracy of the self-assessed TRLs. The panel disagreed with some individually assessed TRLs for projects and felt that the review would have been more effective if those TRLs were assigned by an external party or parties. The TRL assignments did not align with the reviewers' sense of the project's technology status. We recommend that the person with this role offer a concrete example as a benchmark. We recognize that there will be some subjectivity in the assessment, but a single assessor would generate more consistent assignments across the entire portfolio. Engaging the principal investigator (PI)/research team in a dialogue to reconcile the TRL would also be a healthy educational opportunity. This could be part of the regular management and review process and would allow progress to be assessed on a consensus basis.

Recommendation 2: Encourage project teams to perform LCA and TEA early in the project.

We suggest further developing a consistent framework for TEA and LCA during the life cycle of a project. There should be a standard applied to early-stage projects that becomes increasingly detailed and more probable as a project matures. The framework should be broadly communicated so that it is clear what calculations have been applied and how likely they are to be correct for each project. The reviewers strongly encourage integrating this from the beginning to steer choices; it should be used in the individual go/no-go decisions, as it will become adopted by teams if it is institutionalized. If BETO encourages cross-project sharing of LCA/TEA boundaries and assumptions, this will enhance consistency and the ability of reviewers and the technology manager to compare across projects. It may be useful to connect this framework to the TRL.

Recommendation 3: Encourage engagement of teams across the portfolio.

We recommend aiming to build bridges (communication, collaboration) between projects that appear to have natural synergies or interests. A hypothetical example is the all-PET multilayer film project engaging with other PET recycling projects to understand key end-of-life issues. Consistent use of industry and subject matter experts with more involvement from IABs from the onset of projects and throughout will substantially improve the relevance of the programs. There is considerable variability in the application of external reviews, with excellent involvement in the Separations Consortium but less in some of the other project areas. A more formal approach to industry engagement and cross-project collaboration may be helpful here.

PLASTIC DECONSTRUCTION AND REDESIGN PROGRAMMATIC RESPONSE

INTRODUCTION

The program thanks the reviewers for their dedication and thoughtful review of this diverse portfolio. BETO greatly appreciates the reviewers' efforts and expert recommendations, which will significantly contribute to the success and effectiveness of our initiatives. The program agrees with the reviewers' assessment that the plastics portfolio continues to benefit from industry engagement, though inconsistency with reporting this could be streamlined to facilitate the review process. Future development of industry engagement could be strategically used to shape new material design via expert guidance on polymer processing. Specific recommendations and feedback will be discussed and considered when working on future project selection and program design, as future appropriations allow. For each recommendation, BETO has provided a general response, followed by some specific examples of how the feedback will be integrated into the technology area covered in this session.

Recommendation 1: Independent assignment of TRL.

The program thanks the reviewers for their insightful review and valuable recommendations regarding the assignment of TRLs for projects in our portfolio. As we are committed to continuously improving our processes to ensure accuracy and consistency in TRL assessments, this is an excellent suggestion; it will allow for more realistic cross-portfolio analysis.

Based on your suggestion, we recognize the importance of having an entity outside the project teams, such as the technology manager, assume the responsibility of assessing and assigning TRLs. This approach will help eliminate potential biases and provide a more objective evaluation of the TRL.

To address the concern of skepticism among reviewers about self-assessed TRLs, we acknowledge the need for a concrete example as a benchmark to guide the assessment process. Providing a clear reference point will enhance the reliability of TRL assignments and ensure that they better align with the reviewers' perception of the project's technology status.

BETO understands that there might be some subjectivity in the TRL assessment process, but having a single assessor for the entire portfolio will indeed promote greater consistency across projects for the reviewers. We are keen on implementing this approach to maintain uniformity and fairness in our evaluations.

In addition, we agree that engaging the PI and research team in a dialogue to reconcile the TRL will be a valuable educational opportunity. This open discussion will foster better understanding and communication among all stakeholders and will lead to more accurate and meaningful TRL assignments.

Recommendation 2: Encourage project teams to perform LCA and TEA early in the project.

BETO thanks the reviewers for recognizing the importance and effectiveness of detailed LCA and TEA for the success and impact of projects.

We completely agree with your recommendation to encourage project teams to perform LCA and TEA early in the project's life cycle. By integrating these assessments from the beginning, we can better steer our choices and ensure that our decisions align with the most sustainable and economically viable options. We have begun requiring these as milestones within each budget period for our more recently funded projects.

The reviewers' suggestion to utilize LCA and TEA in individual go/no-go decisions is an excellent approach that is currently being adopted by project teams. By making them an integral part of our decision-making process, we have begun to institutionalize their importance and ensure they become standard practice across projects.

Moreover, we recognize the potential benefits of promoting cross-project sharing of LCA/TEA boundaries and assumptions. Enhancing consistency in these aspects will not only streamline the review process but also empower technology managers and reviewers to make more accurate comparisons across different projects, leading to better-informed decisions throughout the life of the project and across the portfolio.

Recommendation 3: Encourage engagement of teams across the portfolio.

The program thanks the reviewers for their insightful suggestion to encourage the engagement of teams across our project portfolio. This suggestion perfectly aligns with our goal of fostering collaboration and communication to maximize the potential synergies and shared interests among projects.

Building bridges between projects is an excellent strategy to promote effective communication and collaboration. Your hypothetical example of the all-PET multilayer film project engaging with other PET recycling projects to understand key end-of-life issues is a compelling illustration of the potential benefits that can arise from such interactions.

To implement this approach, we will actively encourage project teams to identify areas of natural synergy and shared interests with other projects. By fostering open dialogue and knowledge exchange between teams, we can enhance the overall effectiveness and impact of our portfolio initiatives.

Further, we recognize the significance of sharing best practices and lessons learned among projects. Encouraging such cross-pollination of ideas will not only strengthen individual projects but also contribute to the advancement of the entire portfolio.

We genuinely appreciate your valuable input and will make concerted efforts to promote engagement and collaboration among teams across our portfolio.

UPCYCLING OF CFRP WASTE: VIABLE ECO-FRIENDLY CHEMICAL RECYCLING AND MANUFACTURING OF NOVEL REPAIRABLE AND RECYCLABLE COMPOSITES

Washington State University

WBS:	2.2.3.400
Presenter(s):	Jinwen Zhang
Project Start Date:	10/01/2019
Planned Project End Date:	06/30/2023
Total Funding:	$2,051,949

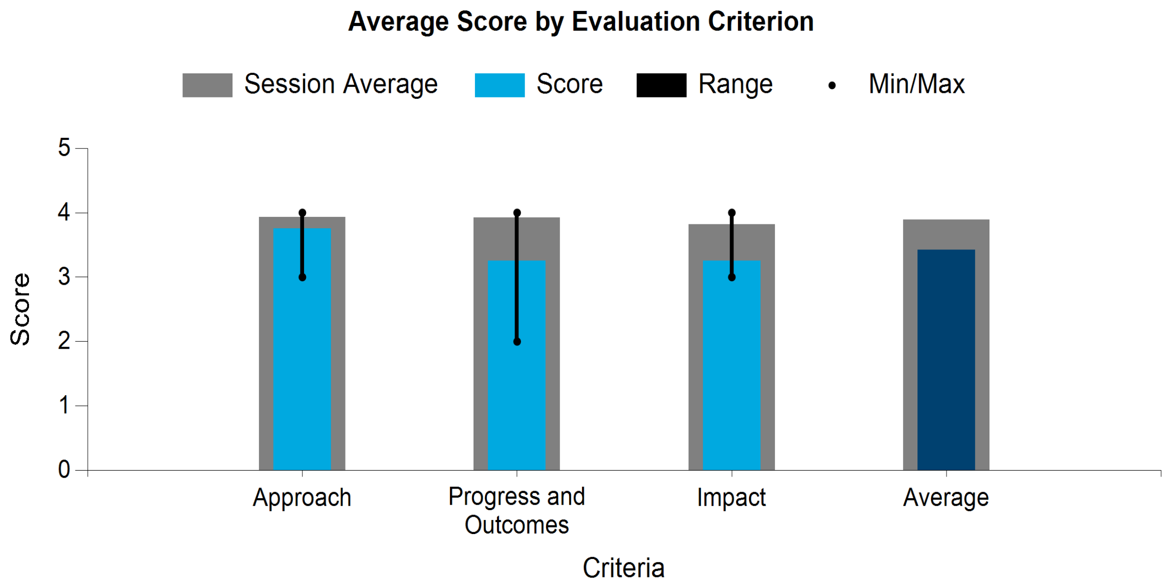
PROJECT DESCRIPTION

A team of researchers led by Washington State University will collaborate on a project entitled "Upcycling of Carbon-Fiber-Reinforced Polymer (CFRP) Waste: Viable Eco-Friendly Chemical Recycling and Manufacturing of Novel Repairable and Recyclable Composites," sponsored by the DOE Office of Energy Efficiency and Renewable Energy (EERE). In this project, the researchers aim to develop a viable chemical recycling technology for carbon-fiber-reinforced epoxy composite (CFEP) waste that is eco-friendly, energy-efficient, and cost-effective in breaking down the matrix polymer structure and makes use of both recovered carbon fiber and decomposed matrix polymer in new advanced composite manufacturing.

The rapid growth of the polymer composite market also propels researchers to find value-added applications for out-of-date prepregs, manufacturing scraps, and end-of-life components. At present, most polymer composite wastes are disposed of by burning or landfilling. To make use of the residual value and reduce the burden to the environment, various mechanical, thermal, and chemical approaches have been attempted to recover the fiber, matrix, or both; however, these current practices exhibit a lack of low-cost effectiveness, are energy-inefficient, generate secondary waste, and bring new pollution problems. In this project, the research team will develop a new CFEP recycling platform that addresses all the existing problems with current recycling methods and will introduce high-value polymer materials based on the recyclates. The key innovation is in the integration of the mild chemical recycling of CFEP and the preparation of new composites.

The success of this project will address the most significant cost/technology barriers for thermosetting composite recycling. With this technical research success, the developed technology will move from the lab scale to the small pilot scale in collaboration with commercial partners. We expect to advance the technology from the current TRL of 2–3 to TRL 4–5 by the end of this project.

The project is led by Washington State University professor Jinwen Zhang, and the major team members include Tuan Liu and Michael Wolcott, both from Washington State University; Long Jiang of North Dakota State University; and Kevin Simmons of Pacific Northwest National Laboratory (PNNL).



COMMENTS

- Recycling of carbon-fiber-reinforced plastics is energy-intensive, not cost-effective, and requires harsh conditions and secondary waste. The team is aiming to develop an eco-friendly, energy-efficient, and cost-effective chemical recycling technology to address these issues. There has been great progress in this review period. The project is on track, and the team is ahead of schedule. There is always a question about carbon fiber recycling in terms of LCA. The team should run an LCA and compare their results with other recycling methods and carbon fiber from lignin. Cost is still a challenge for implementing the recovered carbon fiber in composites applications, and therefore the team should focus on cost reduction items in their approach for scalability and transferability. The mechanical and thermal properties of recovered carbon fiber were not presented. It would be great to see the retention or decrease in the mechanical properties of the recovered carbon fiber. The PI did not present the DEI strategy and progress.

- The project has made good technical progress in defining process conditions to recover both carbon fiber and resin from carbon fiber automotive, wind, and aerospace composites. The PI has gone beyond the statement of project objectives and developed a one-step swelling-decomposition process and an atmospheric pressure process. These are both important for reducing the environmental impact. Is this process too messy to scale up industrially? It would be interesting to understand the cost implications of the vitrimer composites. Not much was shared on the vitrimer composite work. Further work should include active participation of industry partners and understanding of the durability and recyclability of the vitrimer composites.

- This project provides a good approach to a difficult recycling problem and advances the goals of the program with good progress toward the project milestones. There has been good collaboration among academia, the labs, and industry to complete the project, with appropriate application of TEA to validate the costs of the process. It would be useful to have more complete data on the reusability of the recovered carbon fiber to determine whether the recycling process deleteriously affects the physical properties of a new composite made with recycled fibers. The impact would be significantly greater if the fibers were not being downcycled during this process, and it is not apparent that they retain their performance. The disposition of the degraded resin should be described better, as it was not clear what would become of that material (whether a recovery of chemical value for new resins or just as fuel value).

- The team needs to analyze the environmental impact of their process to be able to judge if it is indeed eco-friendly. I would suggest preparing a TEA and LCA that compares the process to virgin material (is it cheaper/better for the environment to use the recycling process versus starting with new material?). The team did not build a prototype of its process to scale beyond the bench scale with their current system. I appreciate that the team is talking to Boeing and Toray about this upcycling method and is continuing the conversation to further scale up this method. I would suggest that the team further engage its industry partners to help with scale-up as well as verify that its recycled materials can be reused in the targeted applications.

PI RESPONSE TO REVIEWER COMMENTS

- We appreciate the comments from the reviewers. We did not propose an LCA study in the original proposal. During the implementation of the project, we also obtained permission from the project manager to not pursue the LCA, as the chemical recycling proposed in this project is unprecedented and we did not have any reference for doing the LCA. The recovered carbon fiber retained approximately 91% of the original carbon fiber's strength and exhibited almost identical thermal stability. We have recently identified a new solvent system that is eco-friendly, inexpensive, and can be used for chemical recycling of CFRP below 200°C at ambient pressure. We are very hopeful that this new resin system will make the recycling process more economic. We will continue to engage with related industrial companies for potential collaboration for technology transfer. Our industry partner in this project, Global Fiberglass Solutions, is a minority-owned small company in Washington state. We have also trained an American Hispanic student for his master's study on this project for 2 years.

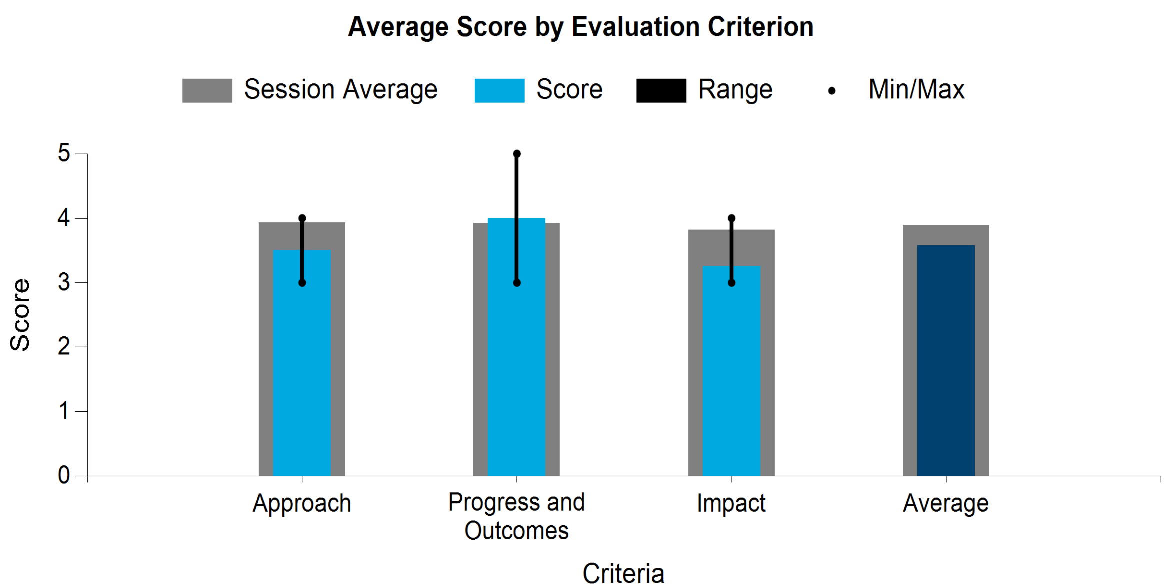
DESIGN AND DEVELOPMENT OF BIO-ADVANTAGED VITRIMERS AS CLOSED-LOOP BIOPRODUCTS

University of California, Berkeley

PROJECT DESCRIPTION

WBS:	2.3.2.219
Presenter(s):	Jay Keasling
Project Start Date:	10/01/2018
Planned Project End Date:	03/31/2023
Total Funding:	$2,497,327

Plastics in use today are predominantly single-use and are rarely recycled. The linearity of their life cycle is not only wasteful from a resource and energy perspective but has also resulted in environmental stresses with more than 6 billion metric tons of plastic waste. The goal of this project is to elucidate design rules by which life cycles for plastics become circular and therefore sustainable. We focus our efforts on a new class of dynamic covalent polymer networks, known as vitrimers, which combine the processing and recycling ease of thermoplastics with the performance advantages of thermosets. Regarding circularity, most vitrimers are differentiated from classical thermosets in that they can be chemically de-polymerized, typically into small molecules or short oligomers, including dimedone, ß-keto-d-lactone (BKDLs), and diacids in this project. For decades, the microbial production of commodity chemicals has been limited in the diversity of the molecules produced by natural or modified enzymes. Our technology of recombining the Type I polyketide synthases demonstrates a promising strategy for the synthesis of diverse molecules, including BKDLs and diacids. With computational materials genomics of vitrimers and TEA and LCA for bioproducts, we can design and develop infinitely recyclable and therefore closed-loop polymeric bio-based materials for potential commercialization.



COMMENTS

- The team's technology of recombining the Type I polyketide synthases demonstrates a promising strategy for synthesizing diverse molecules, including BKDLs and diacids. Using this approach, the team can design next-generation sustainable plastics and composites. The team also incorporated computational materials genomics of vitrimers and TEA and LCA for bioproducts. They selected the right polymer to work on this proposal. There is a lack of research in PU foams in terms of recyclability/circularity of plastics recovered. There has been some progress in this review period. The project is still in the development stage. The TRL should be updated at the end of the project. The PI and the team need to meet and have a serious discussion with their industry partners for the target applications. It is very difficult to pass the requirements for automotive seating. They should focus more on under-the-hood and under-the-carpet foam applications. Achieving PU foams with >75% biomass content and meeting all the durability and design requirements for the final PU applications is extremely difficult. The PI did not present the DEI strategy and progress.

- The concept of infinitely recyclable poly(diketoenamine)s, as vitrimers, replacing petrol-based chemicals like PUs is innovative and addresses an unmet need for the circularity of cross-liked polymers. As such, the team has a difficult road ahead in changing materials and systems that have long been in place, scaled, and optimized. PU foams for the applications identified (automotive seating and mattresses) have an exceedingly difficult list of properties to meet, including density, odor, cell size, compression modulus, compression set, fogging, and tear. The characterization and material properties of the isocyanate, polyol, and novel foams were not provided. Although original equipment manufacturers have shown interest, they should provide specifications for seating foams as a control so that materials can be tailored to meet these requirements. There should be identified partners throughout the supply chain (Tier 1, 2, 3). It is unclear how much this technology could supply the 21 million ton/year PU market as far as scalability. It might be better for the project to focus on foams that have lower performance requirements, such as automotive headliner foams.

- Very good progress has been made on this well-managed project to provide an alternative recycling process for PET. The project has made appropriate use of TEA and LCA to inform the work and to demonstrate potential viability for such a process. There has been excellent collaborative work between academia and the labs to achieve the progress that has been made to date. The application of *in situ* product recovery is an innovative approach to recovering valuable products from the process, and there is a strong sense of industrial practicality in the upstream process steps (including extrusion) that were investigated.

- The team has fulfilled the performance metrics as laid out in its proposal. I appreciate the team's approach to creating novel, recyclable vitrimers. To enable translating this technology into industry, I would implore the team to quantify the processability of their plastics as well as the foaming of their plastics. The proposed material is economically and environmentally viable at 99% recyclability. To further this project, the team should investigate with industry partners ways to realize these very high recycling numbers.

PI RESPONSE TO REVIEWER COMMENTS

- Thank you for the very positive comments. Lawrence Berkeley National Laboratory is negotiating a license to this technology with multiple companies. We are looking beyond seating for automobiles to several other important applications.

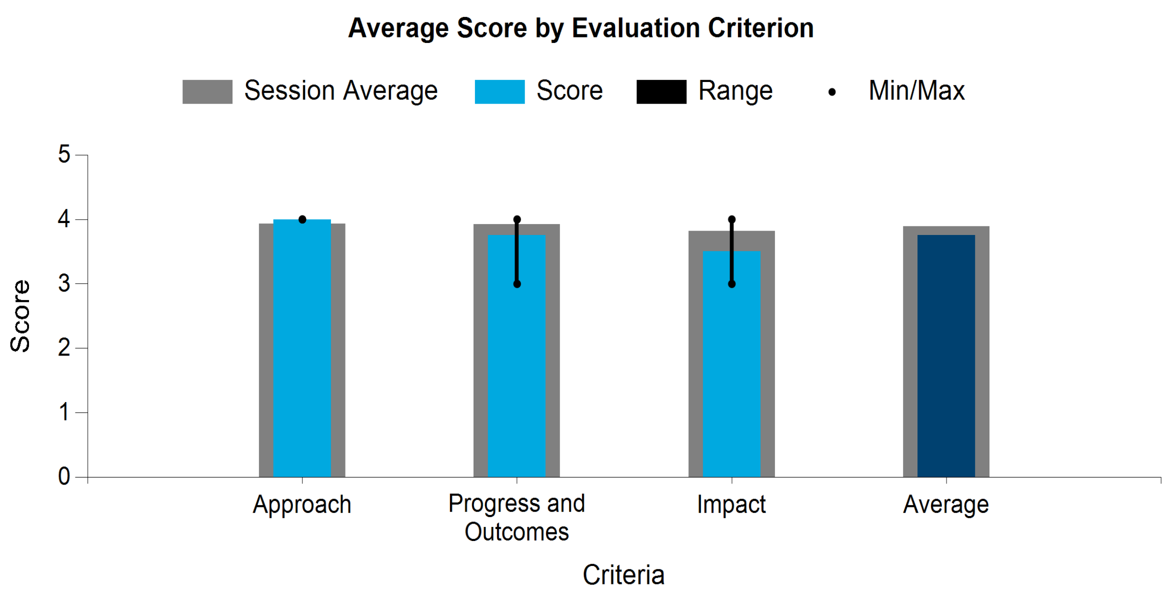
BIOCONVERSION OF HETEROGENEOUS POLYESTER WASTES TO HIGH-VALUE CHEMICAL PRODUCTS

University of Massachusetts Lowell

PROJECT DESCRIPTION

WBS:	2.3.2.224
Presenter(s):	Margaret Sobkowicz-Kline
Project Start Date:	10/01/2019
Planned Project End Date:	06/30/2023
Total Funding:	$1,921,237

This project aims to discover, evaluate, and develop pathways for the economic biochemical recycling of waste polyesters into small-molecule products used in the chemicals and materials industries. A major hurdle in recycling plastics to high-value chemicals is the energy intensity of the process, which could be improved using microbial deconstruction. The aims of the project are to (1) study efficient pretreatment strategies to prepare diverse polyester wastes for deconstruction, (2) develop optimal enzyme combinations for the production of terephthalic acid (TPA) from PET waste, and (3) scale up reactor design for high-efficiency product recovery. The project will enable industry to deploy high-performing drop-in chemicals as an alternative to conventional unsustainable sources.



COMMENTS

- The team tried to develop a biochemical conversion process for the microbial production of specialized degradation enzymes for recalcitrant polyesters as well as bioconversion of the degradation products to high-value-added chemicals, guided by TEA. The team also evaluated different PET waste streams and characteristics for degradation. There were improvements in the minimum selling price and environmental impact resulting from lowered electricity costs for pelletizing versus cryogrinding for recycled terephthalic acid (r-TPA). There has been some progress in this review period. The project is on track. The PI and the team should look at other challenging post-consumer PET wastes, such as automotive waste (headliner and seat fabric, under-body shield and wheel liner, carpet), carpets, and colored textile wastes. If they can focus on these wastes that are not easily recycled (contamination and used chemicals for coloring), that could be a true game-changer. As far as I know, the enzymes digest the PET and leave the rest. The team should also increase their engagement with their industry partners to understand materials and cost requirements. A detailed DEI plan will be great.

- The project has made progress toward the goal of addressing the enzymatic recycling of PET mixed waste to TPA. The PI has a strong DEI plan, including active bystander training. Progress has been made in quantifying the mechanical properties of a wide variety of PET waste streams, identifying appropriate enzymes, and exploring pretreatment through melt processing. Real-life PET mixed waste streams will include a vast selection of dyes, colorants, and other additives. How these will affect both the extrusion process and the enzymes and yield of TPA should start to be addressed. Recycled materials with colorants (generally black because of mixed colors) are of much lower value than a purified product that can be recolored. It is unclear whether there is a higher level of engagement between the researchers and the companies that provided PET samples. Understanding their needs and challenges could help refine the project goals to ensure TRL 4–5 at the end of the project.

- This project provided a very innovative approach to developing high-value products from waste streams. It made excellent use of computational methods and molecular biology to produce new molecules that could have a significant impact on a developing bioproduct market. The project made excellent progress toward its milestones and showed a very good collaborative effort across the labs and academia. The progress through scale-up in fermentation was excellent, and the TEA and LCA were appropriately applied. This project could have benefited from more interactions with an industry partner. The suggested end use for the technology (flexible foams) is a noble target but one for which practical advice on the probabilities of reaching that target would have been a benefit.

- The team has presented a viable strategy to recycle polyester waste. I appreciate that the team has already tried feed streams other than bottle flake, including mylar film and textiles. I suggest that the team work with material recovery facilities (MRFs) and source real-world recycling streams to prove the translatability of their approach and test the influence of additives and dyes on their enzymes. On the commercialization side, the team is ready to move to work on reactor design, scale-up, and system integration. I suggest that the team reach out to industry partners to help with scale-up and integration as well as with potential offtakers of their products (TPA, etc.).

PI RESPONSE TO REVIEWER COMMENTS

- Thanks to the peer reviewers for their thoughtful comments on our project. We identified two primary topics that the reviewers brought up, and we will respond to them here. On the subject of diverse PET waste sources: We agree that this technology would be particularly useful for mixed and contaminated waste streams. The project team has preliminarily evaluated some diverse PET sources for feedstocks in the enzymatic depolymerization process, including textile waste, film, and metallized film. There are two complications that prevent more in-depth investigations in this area. First, our extrusion pretreatment process cannot handle low bulk density materials without a densification step. The extrusion of textiles or fibers is only possible at a very small scale because the material does not flow freely in solids handling equipment. The other complication is the influence of unknown contaminants on enzyme activity. This would be a very interesting area to explore in depth, but because so many factors could be influenced, model contaminants should be explored in a systematic way first. Thanks to the peer reviewers' suggestions, we plan to run some experiments in standard reactor conditions with PET textile feedstocks containing colored contaminants in the final months of the project. On the subject of industry partners: We appreciate the suggestion to look toward commercial partners for this technology. We will discuss reactor scale-up with our partners at the National Renewable Energy Laboratory (NREL) to understand how our innovations could be translated. We also plan to meet with Unifi, our source for PET textile grade scrap, to discuss their interest in supporting this project through the study of more diverse waste sources.

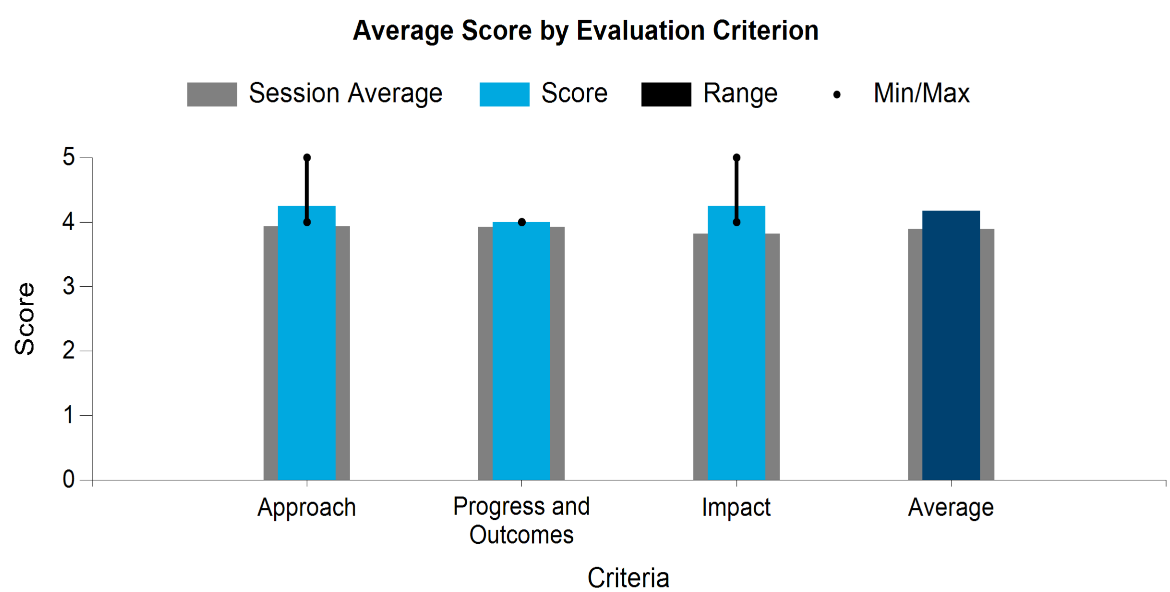
HIGH SOLIDS IN SITU PRODUCT RECOVERY; THE NEXT GENERATION OF ARRESTED ANAEROBIC DIGESTION TECHNOLOGY

Quasar Energy Group, LLC

WBS:	2.3.4.210
Presenter(s):	Xumeng Ge
Project Start Date:	10/01/2021
Planned Project End Date:	04/30/2025
Total Funding:	$4,380,000

PROJECT DESCRIPTION

This project aims to scale the high-solids *in situ* product recovery (ISPR) system (patent application no. 63/020,598) developed by NREL to produce >1.5 kilograms of volatile fatty acids (VFAs) from high solid food waste and subsequently to produce >1 kilogram of sustainable aviation fuel (SAF). Wet waste streams are available at zero or even negative cost and generate significant GHG emissions when landfilled, making them an attractive feedstock for producing cost-advantaged, carbon-negative biofuels. Arrested anaerobic digestion (AAD) can convert wet waste into VFAs, a chemical intermediate for SAF production via ketonization and hydrodeoxygenation; however, this process needs an advanced separation system to continuously extract soluble VFAs from sludge-like fluids with solids present. Due to separation challenges in handling solids and controlling energy consumption, AAD technology has seen limited commercial success in biofuel production. The ISPR system can potentially address these two hurdles, allowing the use of high solid food waste with AAD technology and improving energy efficiency by more than tenfold. This project will scale this system for a first-of-a-kind demonstration to produce VFAs, and subsequently SAF, from high solid food waste with a positive energy balance. We have developed a process model with cost analysis between the proposed system and the risk mitigation strategies. We have carried out pilot-scale system construction and commissioning with 80% completion in build-out and 60% completion in equipment commissioning. We have done feedstock characterization with the preliminary evaluation of biological conversion.



COMMENTS

- The project is on track. The team is trying to separate VFAs from digesters and produce an SAF product using NREL-patented high-solids separation technology. Previous and planned DEI activities look good.

- The DEI plan does not mention the number of interns that will be recruited; however, the team itself is diverse. The objective of the project is to demonstrate NREL's patented process for high-solids food waste separation to separate VFAs that can be turned into SAF. There are two approaches that will be researched: (1) rotating ceramic disk filter followed by hollow tube filter and (2) vibrating membrane followed by emulsion separation membrane. The TEA data and construction demonstration build-out of both processes are close to complete, along with feedstock characterization. The process models are complete and on schedule. The TEA and LCA show significant potential value (lower-cost separation and ~5/gallon VFA) and a carbon-negative footprint. Good progress has been made, and this is a good project.
- This scale-up project is well managed and has a thoughtful approach that should lead to the successful completion of the project objectives. Reducing the costs associated with the separations required for SAF production will have a significant impact on achieving the department objectives. The project shows good collaborations between labs and includes industry partners in useful ways. Risks that may require more attention to mitigation include ensuring that the separations reflect the actual feed, whether it is live broth or some other source, because small changes in that feed could have a deleterious impact on the process. This is exacerbated by the variability of the wet waste feed stream. The post-bioprocessing step appears to be less developed than the basic separation, and the team should consider decoupling the effort to ensure that the proper focus is kept.
- I appreciate that the team is working closely with their industry partners, who are supplying the equipment parts of the separation system. I appreciate that the team has identified a downstream partner, Alder, that is planning to offtake the product of their separation. I appreciate that the team is working on its TEA and LCA during the whole lifetime of the project. I would love to learn more about how resilient the process is for waste streams from different sources and whether the team can find an industry partner to produce food waste. This team's assessment of the starting TRL as 3 is accurate, and their target of TRL 5 seems realistic.

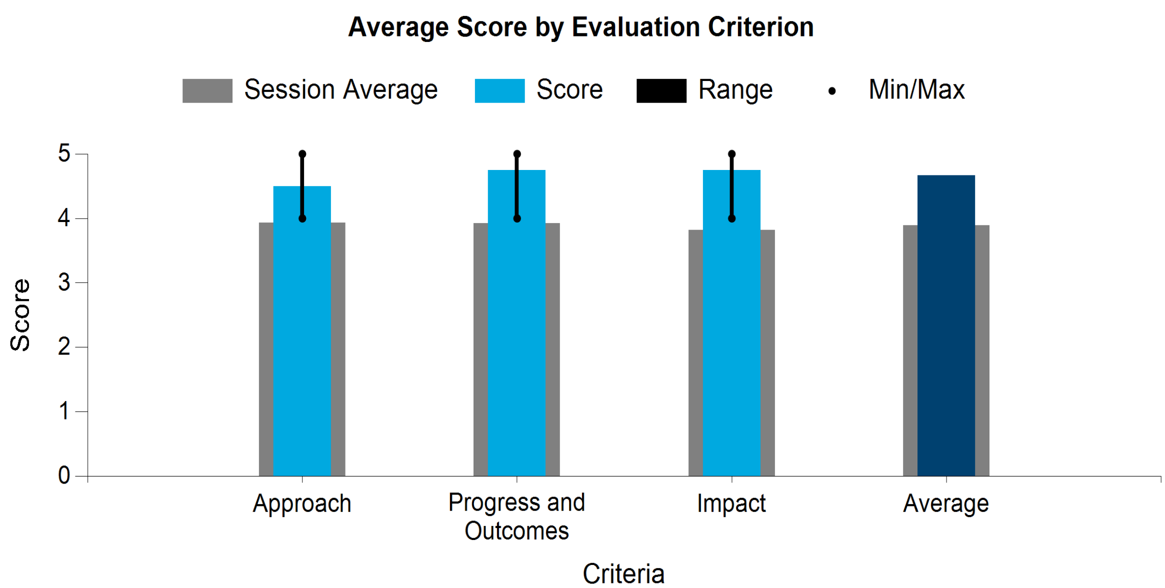
PI RESPONSE TO REVIEWER COMMENTS

- We thank the review panel for the positive comments and constructive input. We are incredibly excited about the potential for this project to be successful in the coming months as activities ramp up in all the tasks. In response to the comment about the number of interns that will be recruited, Quasar Energy Group (QEG) plans to hire two interns from local institutes in Budget Period 3 (BP3). Regarding the comment on using actual feedstocks—we completely agree on the need to use real feedstocks during commissioning to ensure a successful demonstration and accurate integration, especially between the anaerobic digestors and the separations train. This has been planned and will be happening in the coming months. The need to start building out and commissioning all the unit operations in a parallel manner led to initially using materials other than the actual digestate for the early commissioning of the separation's unit operations. In addition, we are leveraging prior NREL results from an initial lab-scale demonstration using food waste at high solids. In terms of decoupling efforts between the post-bioprocessing and product separation steps, as noted in the approach section of the presentation, novel separation technologies are being assessed in this project. The pilot-scale vibrating membrane system developed by project partner New Logic Research has recently been commissioned and will be evaluated as an alternative to the rotating ceramic disk filter to serve as a solid-liquid separation unit. Although initially proposed to be coupled to the emulsion separation membrane system as an option, potential permutations between the two process configurations will also be considered. Regarding the comment on using different sources of waste streams—we completely agree on the need to understand the resilience of the process; however, our first objective is to demonstrate the proposed process at the pilot scale, and using a single source of waste will allow us to focus on the bioconversion-separation interface during the integration efforts. After that, and if the period of performance allows, we will use samples from the feedstock tanks of QEG's digester plants, which receive food waste streams from different sources. As for industry partners that generate food waste, QEG contracts with many of them (mainly in Ohio) and routinely works with them to treat their food waste.

CONTINUOUS BIOBUTANOL FERMENTATION INTEGRATED WITH MEMBRANE SOLVENT EXTRACTION

Archer Daniels Midland Co.

WBS:	2.3.4.212
Presenter(s):	Erik Hagberg; Jesse McVay
Project Start Date:	10/01/2021
Planned Project End Date:	09/30/2024
Total Funding:	$4,341,844



COMMENTS

- There has been excellent progress in this review period. The project is on track, and the team is ahead of their schedule. A business justification and a single-source material for feedstock should be addressed, and the team should look at alternative feedstocks other than pea starch. Based on the plan and progress, the team will be ready to present the infrastructure architecture that would demonstrate the process to produce 100 kilograms with 100 hours of continuous operation at the end of the project. The industry, national lab, and university collaboration is excellent, and the DEI plan is very clear and well executed.

- The project plan looks very solid and is integrated with the participation of partners at each step. A strong industry partner, Archer Daniels Midland (ADM), is collaborating with the team. Decision points are well defined. The DEI plan is well defined and broad (nine internships, teacher workshops, and researcher training). It may be interesting to study starches other than pea starch. Is the pea starch crude or purified (washing)? Pea starch is a growing industry, but many companies using it are struggling financially, and the amounts available pale in comparison to starches like corn. Also, pea starch is viewed as a healthier, low-glycemic-index, and non-genetically modified organism material for use in gelling foods or as a gelatin replacement (for vegetarians). There may be resistance from a media standpoint in using more precious food sources for aviation fuel and chemicals. Pea starch is also high in amylose, making it more resistant to digestion. This could be a benefit for human health as well. I wonder if you would see any effects or efficiencies in your process (hydrolysis) using other starches?

- This project has the potential to be very impactful in reaching the department goals for SAF, and it illustrates an excellent collaborative relationship with industry partners. Each party's role is understood, and the division of labor is very sensible. One weakness in the approach is the focus on pea starch. Volumes were not presented, but the amount of pea starch available for fuel production relative to the demand should be considered in order to assess the value of the technology. To meet the fuel volume target, many more sugar sources will be required, not just pea starch. That risk should be relatively low, as changing the feedstock should not have a large impact on the fermentation process.
- The graphic on slide 5 showing which industry partners are supplying which materials and equipment/expertise is extremely helpful. I would suggest that all projects with milestones around partnering, sourcing real-world feedstocks, or working with offtakers should present an equivalent slide. I appreciate that both Gevo and ADM have plans to expand their SAF collaboration and deploy capital. The team is strongly partnering with Gevo, which will be the offtaker of isobutyric acid to use it as a starting material in SAF production. How much pea starch is available? I would suggest that the team explore other feedstocks to make the process more widely useable.

PI RESPONSE TO REVIEWER COMMENTS

- We would like to thank the peer reviewers for their thoughtful comments. All reviewers recognized the feedstock, pea starch, as a potential weakness in the project's approach due to its relatively low volume as well as its potential human nutrition applications. The approach for selecting pea starch, a protein production residue, was centered around three basic assumptions: (1) Alternative, plant-based proteins will grow significantly in the future (reaching 65 million tons per year by 2035, based on some estimates); (2) protein produced from legumes and grains will generate carbohydrate byproduct streams as their protein is recovered (from 0.5–2 kilograms carbohydrate per kilogram protein, depending on source); and (3) pea starch is a reasonable representative carbohydrate stream from these alternative proteins. The focus on one carbohydrate byproduct from protein production allowed the team to investigate whether individual challenges from fiber, residual protein, and non-hydrolysable solids could be overcome with a relatively stable and homogenous feedstock. Initial findings showed that hydrolysis could be achieved economically and that the resultant fermentation feedstock meets the organism's needs and provides adequate nutrition. These results indicate that, in principle, starch streams from additional protein sources could be addressed as well, including from legumes (for example, lentils and chickpeas) and other low-value carbohydrate streams not appropriate for human nutrition, such as soy protein processing byproducts (soy molasses) and wheat processing byproducts (B-type starch). Traditional sources of dextrose for fermentation, such as corn—processed in either dry grinds or wet mills—could also likely be used for this fermentation with little technical risk, based on the initial results.

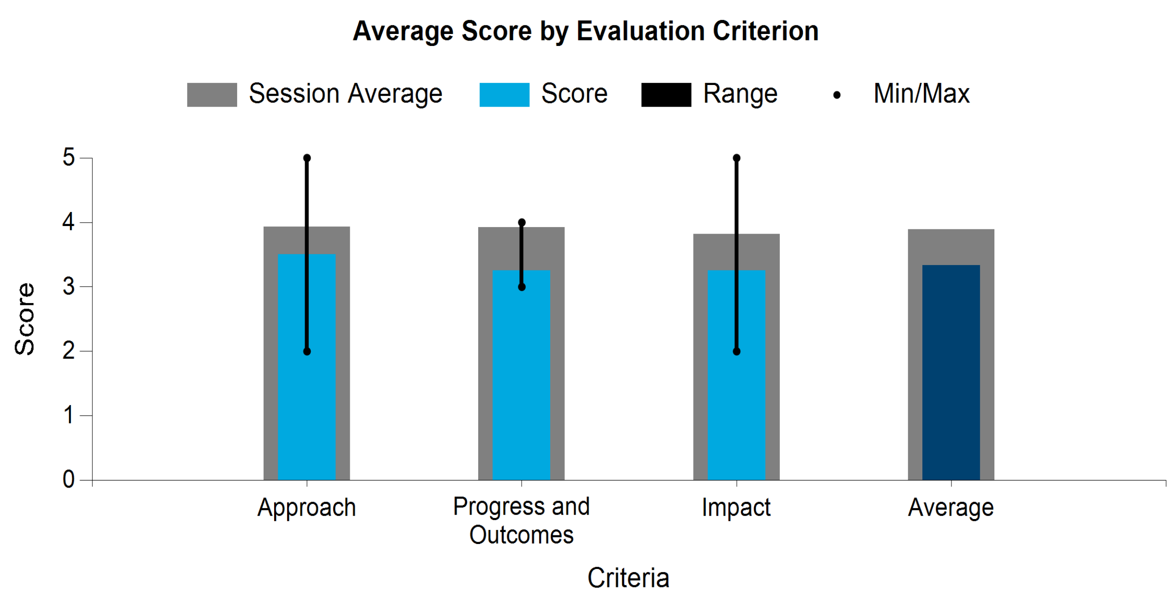
TROJAN HORSE REPEAT SEQUENCES FOR TRIGGERED CHEMICAL RECYCLING OF POLYESTERS FOR FILMS AND BOTTLES

Iowa State University

PROJECT DESCRIPTION

WBS:	2.3.4.400
Presenter(s):	Eric Cochran
Project Start Date:	10/01/2020
Planned Project End Date:	03/31/2024
Total Funding:	$2,722,420

This BOTTLE project (Topic Area 1a) will develop highly recyclable (at least 50% and up to 100%) bio-based polyesters (at least 50 wt % non-food starch-based terephthalate) that are functionally equivalent or superior to, and compatible with, PET. ADM will develop scalable pathways for processing non-food starches obtained as coproducts of vegetable protein production from peas, wheat, beans, and other crops to upgradable furan- and phthalate-based building blocks, including the furan dimethyl esters and terephthalates ADM has developed previously from corn sugars. Iowa State University will use these building blocks to design highly recyclable PET/"trojan horse" copolymers through trojan horse repeat sequences that enable quantitative chemical depolymerization triggered by specific yet mild conditions, enabling facile raw material recovery and repolymerization to virgin material. Diageo and 3M will holistically evaluate PET/trojan horses as bottles and films from the perspectives of performance, aesthetics, compatibility with existing infrastructure and recycling streams, regulatory considerations, and life cycle impacts. Promising candidates will be brought to the multikilogram scale by 3M and processed to bottle (Diageo) and biaxially oriented film (3M) prototypes for further evaluation. Diageo will evaluate bottle prototypes for suitability as packaging for its products. 3M will evaluate prototypes as carrier films, release liners, and optical films that are used for adhesive tapes and electronic devices. TEA will demonstrate the economic potential of the materials, and LCAs will model the carbon and energy savings of the PET/trojan horse copolymers over the entire life cycle. The research partnership comprises entities representing the entire supply chain of the proposed plastics, ensuring that the research and development (R&D) efforts account for all aspects of bringing a new polymer to market.



COMMENTS

- This presentation was notable for communicating in-depth scientific information in a manner that works for a broader audience (me). It also clearly noted limitations and decisions not to proceed with certain material(s), which to me provided credibility to the larger effort. My notes indicate that one of the commercial partners is now working in the kilogram range, which indicates that commercialization is being explored in accordance with the lab findings.

- The concept of trojan horse linkages for the controlled depolymerization of PET is elegant. The project team includes strong industry participation spanning the value chain, including material manufacturing (3M), packaging design and recycling (Diageo), and bio-based feedstocks (ADM). The team has clear roles and a clear communication plan. The project team is diverse and has successfully recruited students from minority-serving institutions (MSIs). It would be useful to pay continued attention to how members with different identities thrive during the project so that true inclusion can be achieved. My main concern with this project is that the risk mitigation in inadequate. Of particular concern to me are the toxicity/rational selection of trojan horse linkages from the start; benchmarking against current methanolysis/glycolysis recycling routes, as they go back to monomers rather than the expected oligomers; interaction with existing polymer processing and recycling streams; and the melt stability and moisture sensitivity of these systems. It is good that the project has ambitions to evaluate these materials' suitability for film and bottle processing, but jumping there without first screening for food contact compliance and melt stability aspects creates a large risk that the material will not be fit for application in the end. Including TEA to understand the commercial viability of this approach would be advisable early in the project. Additionally, the LCA shown focused on the impact of bio-based TPA and did not include the trojan horse linkages. This must also be included. Also, given the commercialization of several chemical recycling routes for PET (methanolysis, glycolysis, etc.) that are coming online, it would be helpful to understand the comparison with this technology and those routes as the system depolymerizes to monomers rather than the envisioned oligomers. I am also concerned about how these materials will interact with standard mechanical PET recycling streams. If they would negatively affect mechanical recycling and require sorting from that stream, it could have globally negative consequences on the recycling system for PET.

- The team has made satisfactory progress. There is a very transparent description of DEI and organization. The regular team meetings of all stakeholders (including the industry partners) are important in providing guided technical decisions. The scale-up goals for 2023/2024 indicate strong interest by industry partners and promise in the technology. The physical properties of the PETs with varying percentages of the trojan horse molecule look good. Does the team have a hypothesis-driven plan to address the barrier inferiority of the PET/trojan horse? If there is a food/liquid-contacting surface, the team should work with partners to identify appropriate in-use stability testing requirements of these new PET/trojan horses.

- I appreciate the close connections the team has to its industry partners. They are working with 3M, which wants to use their plastics; ADM as the biomass feedstock provider; and Diageo as a bottle end user, which has a bottle manufacturing pilot line and will make some bottles at the end. I would implore the team to investigate the toxicity of their plastics as well as the processability of their plastics. The easy decomposition of the plastic under the influence of water can result in decomposition of the plastic during processing. As this is a novel compound that will be in contact with food, it will have to undergo U.S. Food and Drug Administration (FDA) approval before coming to market. The team should develop a regulatory plan in order to enable commercialization.

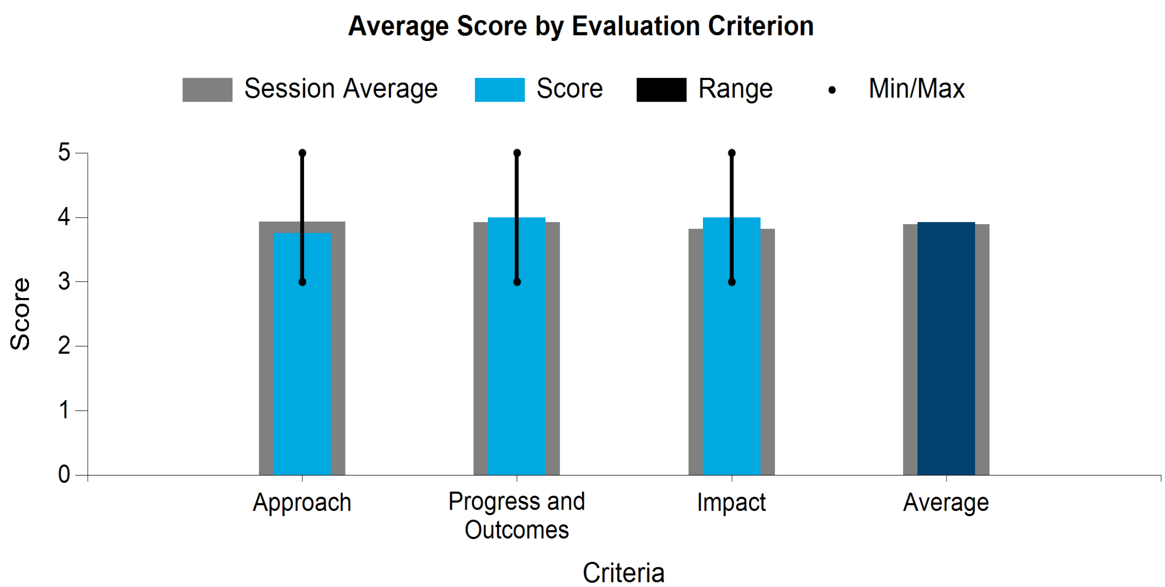
PI RESPONSE TO REVIEWER COMMENTS

- We appreciate your time, expertise, and constructive feedback on our project, "Trojan Horse Repeat Sequences for Triggered Chemical Recycling of Polyesters for Films and Bottles." The unique perspectives and insights each of you have provided are invaluable to the success of our R&D efforts. Reviewer 1, we gratefully acknowledge your positive comments about our project's concept and the diversity of our team, including the significant industry participation across the value chain. Your remarks about our attention to inclusion within the project team are well taken. We will continue to ensure that all team members, regardless of their identities, feel valued and thrive in our project environment. We also recognize the concerns you raised regarding risk mitigation, especially the need for a rational selection of trojan horse linkages from the start to avoid toxicity issues. Benchmarking against current recycling routes and understanding the interactions with existing polymer processing and recycling streams are important aspects that we will address more thoroughly moving forward. These aspects are especially important to project participant Diageo, which has firsthand knowledge of the importance of these issues. Additionally, we will study the interactions of our new materials with standard mechanical PET recycling streams to avoid any negative consequences on the recycling system for PET. The suggestion of including a TEA early in the project to understand commercial viability is acknowledged; we initiated these activities shortly after developing confidence that the trojan horse methodology had technical promise. The project is also working on a complete LCA, including the impact of trojan horse linkages. We recently added a student under Prof. Mba-Wright's advisement to strengthen this part of the project. Reviewer 2, we are glad that you found our presentation to be communicative and credible. We will continue to communicate our scientific findings in a manner that is accessible to a broad audience. Reviewer 3, we appreciate your acknowledgement of our transparency in the description of DEI and organization as well as the regular team meetings with all stakeholders. Regarding the comment relating to the barrier inferiority of the PET/trojan horse, we note that as we continue to collect better data from properly processed films, these properties are, in fact, comparable among the various compositions. We will work closely with our partners to identify appropriate in-use stability testing requirements for these new PET/trojan horses, particularly in the context of food/liquid-contacting surfaces. Reviewer 4, your appreciation of our close connections with industry partners is encouraging. We understand your concerns about the toxicity and processability of our plastics and the need for a regulatory plan for FDA approval before commercialization. We will make sure to investigate these aspects further and develop a comprehensive regulatory plan. In response to the various points raised, we would like to assure you that we will integrate your valuable advice into our project moving forward. Your constructive criticisms not only aid in the identification of potential pitfalls but also guide us toward the necessary adjustments for the success of the project. Again, thank you for your insightful feedback and for your contribution to advancing our research.

UPCYCLING PET VIA THE VOLCAT PROCESS

IBM

WBS:	2.3.4.401
Presenter(s):	Greg Breyta
Project Start Date:	10/01/2021
Planned Project End Date:	09/30/2024
Total Funding:	$2,147,090



COMMENTS

- The presenter stated a definition of closed loop wherein the project does not consider the waste produced; this is a problem either in communication or in vision. Similarly, there was either a problem in waiting until the work was completed to do LCA and TEA or—if those are not just afterthoughts—then there was a problem in the communication of how those analyses are part of the process as decisions are made. I would have appreciated more benchmarks and comparisons, particularly in visual form. Without them, it was difficult to assess the meaning and relevance of the information the speaker was attempting to share.

- This is an extremely strong project with a clear approach to transitioning the technology to pilot-scale demonstrations. Risks are clearly identified with clear mitigation strategies in place, such as reverting to a stirred tank if the extrusion reactor proves unworkable. The management and communication plan was not explicitly covered in the slides, but from the work presented, it appears that there is sufficient interaction among the project participants. My only suggestion is that more focus be put on understanding the BHET benchmarking needed to be suitable for repolymerization (what does "high-quality BHET" mean in terms of performance metrics?). The project has demonstrated substantial progress toward objectives and reducing costs, particularly with regard to the decoloring process. Additionally, the demonstration of successful recovery and decolorization for real post-consumer recycled (PCR) waste flake and fabric trimming is highly encouraging. The focus on evaluating real waste streams is excellent and should remain a focus. The rationale and approach for pursuing polybutylene terephthalate were not clear and not discussed. The project team should consider whether narrowing their focus to core BHET production would ensure faster progress. The potential to target waste streams that do not compete with the mechanical recycling of PET gives this process potential to expand the amount of PET recycled rather than displacing mechanical recycling. This focus and the emphasis on patents is appropriate for this commercialization-focused project. The suitability of BHET as a feedstock for any PET production process currently greatly lowers the barrier for adoption as well as ensures a large potential market with lower barrier of entry for recovered materials. Given the brand pledges around PCR in both fiber and bottle markets, there is a real potential for rapid adoption, and therefore near-term decarbonization impact, due to market pull if VolCat can be shown to be feasible at the pilot scale.

- I am excited by the promise of this project because of the success in demonstrating PET fabric and particles as viable substrates for upcycling. I would like to see more data about the catalyst and understand its stability, resilience, and recovery in the presence of common additives in the crude waste feed.
- The team is doing their TEA/LCA at the very end of the process. It will be too late to pivot then. There has been a baseline TEA out there, and the team is currently feeding information in that model. I would like the team to more actively engage with the TEA/LCA, especially because this project is starting at a relatively high TRL. The stated goal of the project is to develop a TEA/LCA and demonstrate reducing GHG emissions. Without being able to review the results of this analysis, it is not possible to judge the progress made toward this goal.

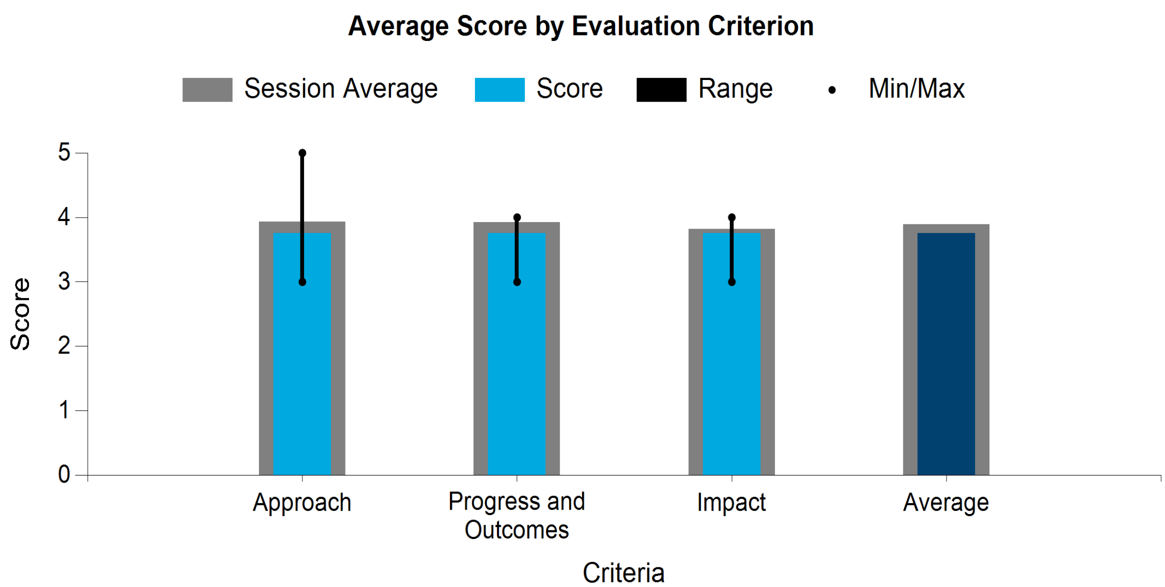
PI RESPONSE TO REVIEWER COMMENTS

- Thank you for providing feedback on my recent project review presentation for PET upcycling via the VolCat process. The comments about benchmarking the BHET have been incorporated into a new task. In this task, we are benchmarking the recycled polyethylene terephthalate (r-PET) prepared from the BHET obtained using the nominal VolCat reaction, which will then be compared with the BHET obtained using all the optimization results, e.g., the modified decolorization process in the scaled process. The production of polybutylene terephthalate is a component of the project for at least two reasons: first, as a demonstration of “upcycling” to a more valuable product than the feedstock polymer type we used to make the monomer and, second, as a demonstration that the VolCat process has broad applicability to polyesters in general (not just PET). Regarding my lack of clarity on the economic analysis/project progress feedback loop: There is already a basis TEA and LCA model prepared from data from the “nominal” VolCat process as it existed prior to the project start. The TEA model will be updated as new data permits, and we will have a final deliverable that incorporates the totality of what we learned during the project. The mid-project interim TEA was not yet prepared, so I was not able to provide a summary at the Project Peer Review. There is a task to quantify the catalyst recyclability along with conducting a purity assessment. This project was not intended to be a survey of a wide range of potential inputs to the VolCat reaction but rather to pick a number of the highest-volume viable inputs to the process that cannot be recycled by existing mechanical means. We are therefore investigating the fate of the catalyst with these inputs as a starting point.

DESIGNING RECYCLABLE BIOMASS-BASED POLYESTERS

University of Wisconsin–Madison

WBS:	2.3.4.403
Presenter(s):	George Huber
Project Start Date:	10/01/2020
Planned Project End Date:	04/30/2024
Total Funding:	$3,125,000



COMMENTS

- My notes indicate that the question of how/why waste plastics escape into the environment arose in the discussion of this presentation. The U.S. Environmental Protection Agency (EPA) has a method—the Escaped Trash Assessment Protocol (ETAP)—aimed at helping people at all levels assess and answer this question for their geographical area of interest. That data set, and other data sets with different parameters (some use weight, some use count of items, etc.), are available for use by researchers. The Q&A indicated that the researchers are not connecting to the main potential markets for their potential products, such as mulch film.

- The approach has merit in terms of identifying potential new bio-based polymers with properties superior to polybutylene adipate terephthalate (PBAT). The active engagement of partners along the entire value chain from monomer production to film production increases the likelihood of commercialization. This is due to a high level of understanding of what material property targets are needed and what monomers can be produced at scale. A particular strength of the project is the focus on the processability and rheology of the materials; however, the crystallization behavior, including rate, should be added as an area of focus. The project is making good progress on hitting the objectives set out, with routes identified to increase biomass content. Incorporating TEA/LCA from the start of the project has proven effective. The involvement of Pyran, which is commercializing 1,5-pentanediol

(PDO) production, increases the likelihood that selected polymers will be able to be produced at scale. Given the choice of PBAT as their reference material to improve upon and its widespread use in mulching films, it would seem logical to target agricultural applications with these new materials. The plan for down-selecting to focus only on the more promising materials for scale-up and evaluation is smart.

- The project has a clearly delineated technical scope coupled with a budget cycle and timelines. I am optimistic that the project has a high probability of meeting polymer physical and process targets for the next budget period verification. I am still curious about the interesting structure/property relationship of the methylene groups in the diol and where the 1,5-PDO falls off that curve. This begs more polymer structure analysis to understand it.
- I am impressed by the team's focus on bringing their plastics to market, thinking through final applications, and lining up partners to help with realizing these. I would recommend that the team test their plastics in a mulch film application as well.

PI RESPONSE TO REVIEWER COMMENTS

- We thank the reviewers for providing their helpful comments about our project. We also thank the reviewers for their very positive comments. We look forward to moving ahead with our new types of biomass-derived biodegradable plastics.
- Comment: The approach has merit in terms of identifying potential new bio-based polymers with properties superior to PBAT. The active engagement of partners along the entire value chain from monomer production to film production increases the likelihood of commercialization. This is due to a high level of understanding of what material property targets are needed and what monomers can be produced at scale. A particular strength of the project is the focus on the processability and rheology of the materials; however, the crystallization behavior, including rate, should be added as an area of focus.
- Response: We have made crystallization an area of focus and have now identified a nucleator to speed up the crystallization of our polymers. Previously, crystallization half-time at 40°C was ~30 minutes. Now, it is ~30 seconds, which is the same value for commercial PBAT at these conditions. We are continuing to explore other nucleators to see if improvements are possible.
- Comment: The project is making good progress on hitting the objectives set out, with routes identified to increase biomass content. Incorporating TEA/LCA from the start of the project has proven effective. The involvement of Pyran, which is commercializing 1,5-pentanediol (PDO) production, increases the likelihood that selected polymers will be able to be produced at scale. Given the choice of PBAT as their reference material to improve upon and its widespread use in mulching films, it would seem logical to target agricultural applications with these new materials. The plan for down-selecting to focus only on the more promising materials for scale-up and evaluation is smart.
- Response: We thank the reviewers for their comment. We are excited for the many industry partners we have on the project who are making crucial contributions.
- Comment: My notes indicate that the question of how/why waste plastics escape into the environment arose in the discussion of this presentation. The EPA has a method—the ETAP—aimed at helping people at all levels assess and answer this question for their geographical area of interest. That data set, and other data sets with different parameters (some use weight, some use count of items, etc.), are available for use by researchers. The Q&A indicated that the researchers are not connecting to the main potential markets for their potential products, such as mulch film.

- Response: We thank the reviewer for providing us with information about ETAP, and we will analyze this in the future. The ETAP tool can be used to quantify the plastic waste across time and habitat type, which provides insight into the trends for plastic waste across the United States. Based on the feedback from reviewers, we are going to focus more on mulch films for the next part of the project.
- Comment: The project has a clearly delineated technical scope coupled with a budget cycle and timelines. I am optimistic that the project has a high probability of meeting polymer physical and process targets for the next budget period verification. I am still curious about the interesting structure/property relationship of the methylene groups in the diol and where the 1,5-PDO falls off that curve. This begs more polymer structure analysis to understand it.
- Response: In work leading up to the current DOE-funded work, Ph.D. student Lei Zheng (University of Massachusetts Amherst, 2022, advised by John Klier) described the effects of PDO structure on macro properties, including thermal transitions, crystalline structure, crystalline behavior, mechanical properties, and biodegradation. We have continued this line of inquiry. We have also found data from other literature sources at higher aliphatic diol contents that suggest more of an even-odd effect than originally thought. We will be adding crystal size to our analysis via small-angle X-ray scattering measurements as well. We will provide this information to DOE in our next report but cannot paste graphics into the response to reviewers.
- Comment: I am impressed by the team’s focus on bringing their plastics to market, thinking through final applications, and lining up partners to help with realizing these. I would recommend that the team test their plastics in a mulch film application as well.
- Response: We are going to focus on mulching film applications in the future as well. Amcor has agreed to produce several mulching films, which we will test.

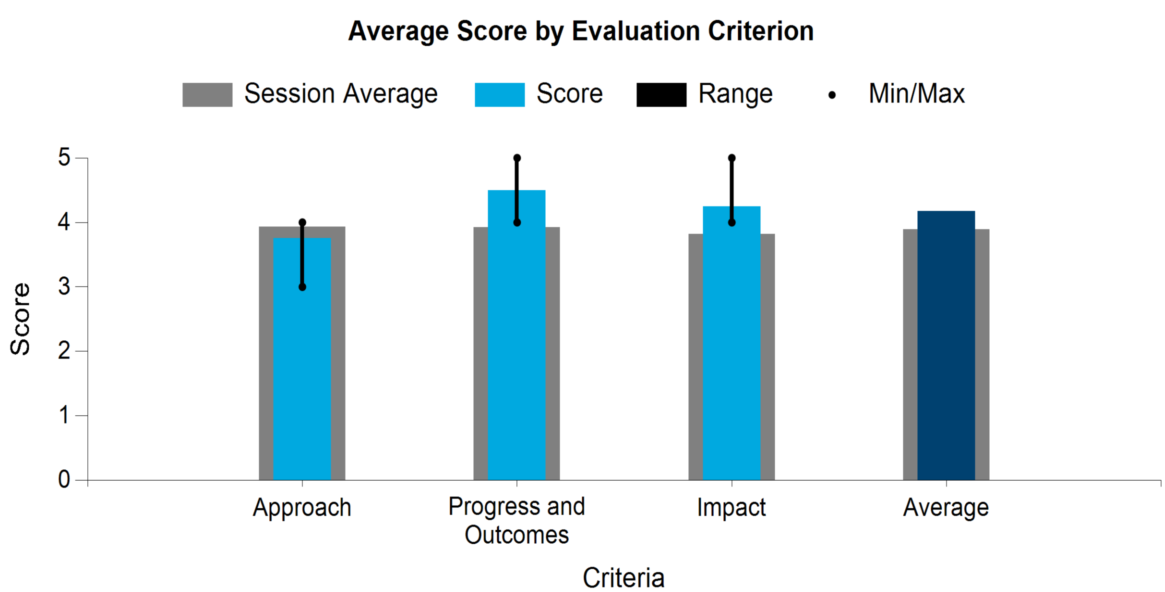
SYNTHESIS AND ANALYSIS OF PERFORMANCE-ADVANTAGED BIOPRODUCTS

National Renewable Energy Laboratory

WBS:	2.3.4.501
Presenter(s):	Gregg Beckham; Laura Hollingsworth; Megan Krysiak; Michelle Reed
Project Start Date:	10/01/2017
Planned Project End Date:	09/30/2023
Total Funding:	$520,000

PROJECT DESCRIPTION

This project focuses on the synthesis and analysis of PABPs. We have established collaborations with other BETO-funded projects and academic and industry collaborators to source new molecules that have promising manufacturing pathways and that could serve as performance-advantaged biochemicals or biopolymers. We conduct synthesis and characterization of biochemicals and biopolymers alongside TEA and LCA to estimate their cost and environmental impacts relative to incumbent materials. As part of the project, Linda Broadbelt and Brent Shanks are developing computational pathway prediction tools to identify optimal production pathways for bio-based compounds via biological and chemo-catalytic transformations. When coupled to the Polymer Inverse Design (PolyID™) tool from the Inverse Design Project, these tools will ultimately enable a narrowing of the design space for PABPs. From FY 2021–FY 2023, we described a framework for benchmarking PABPs, estimated the energy and GHG emissions for commodity organic chemicals, developed performance-advantaged nylons and polyesters from beta-ketoadipic acid (beta-KA), and produced lignin-based plasticizers. We have shown that aromatic amines can be used in performance thermosets and that polyhydroxyalkanoates with cross-linked side chains can exhibit rubber-like properties, along with repair and degradability. We are actively working with industry partners on the scale-up and validation of multiple PABPs.



COMMENTS

- There has been some progress in this review period. The project is on track. The team developed new PABPs for polymers and chemicals with viable, economic, and GHG-advantaged manufacturing pathways. There is cross-collaboration with other teams. It is clear that incorporating beta-KA in PET and PBAT enhances polymer properties, recyclability, and biodegradation. It would be also great to understand the effects of beta-KA on the barrier properties and transparency of the final polymers. Active technology transfer with a startup company (textile major) for nylon-6,6 incorporated with beta-KA is a really important step. The team should investigate and find the mechanism for improving the water absorption behavior of nylon-6,6 incorporated with beta-KA. It is also important to find target applications and industry partners for other polymers with PABPs in the early design process. The PI did not present their DEI strategy and progress.

- There was no structured DEI plan in the presentation. The lignin-derived plasticizer work is promising as an alternative for toxic phthalates. There is a real need for lower-toxicity small-molecule additives such as plasticizers, flame retardants, and per- and polyfluoroalkyl substances (PFAS) replacements. On incorporating beta-KA into nylon 6,6: Is there a need for a higher glass transition temperature (Tg) in textiles? The success of the project may be more likely with small-molecule development versus new polymers, which are very difficult to implement from cost, risk, and change perspectives. Will these new polymers produce yet another material at end of life to separate out for special handling? There was good collaboration with the computational group and with several industry partners. The industry partners can help you to determine which properties to home in on for each application—most times, cost will be a major factor. Instead of a 25% cost increase, they won't be interested until there is at least a 15% cost savings because of all the transition and investment required. The additives, however, can be more readily changed, and the lower toxicity of bio-derived additives will help them sell their product to consumers and continue to do business with health and safety regulations. What about heat and light stabilizers from bioprivileged molecules?

- This project is well organized and shows excellent collaborative work with academic and lab partners. This range-finding work is important for extending the possible technical and market reach of bioproducts and further demonstrates the utility of bioproducts in a range of applications. There has been excellent use of TEA and LCA as a means of quantifying the relevance of these new products for comparison to incumbent products. For future work, I would suggest more industry involvement to help validate target identity and performance requirements.

- The team has screened an impressive number of different structures to identify moieties that can be procured at reduced cost and with lowered GHG emissions. The project seems to be at a point where the team can move from a wide screen to focusing on its "winners" and pushing them to higher TRLs. I am impressed that the team identified a beta-KA-containing polymer that degrades favorably and that the team is planning to scale it up. I appreciate that the team is working on several patents and is working with a startup company and textile major to scale up production of their performance-advantaged nylons from beta-KA. I would like to see the team continue the commercialization of this polymer. As this project is coming to an end, it would be worthwhile to explore whether next steps should target pushing commercialization of this project's lead candidates or continue on discovery work. I see merit in both and would suggest a two-pronged approach. First, proof out the platform by continuing scale-up and commercialization of the lead candidates. In parallel, use an analysis/experimental data science cycle platform to discover additional PABPs. I would be more specific in the desired metrics for this second set of PABPs by continuing to focus on the team's analysis that showed opportunities in heteroatom-containing chemicals, which exhibit the highest energy/GHG emissions of all chemical classes studied here. As the team is already working with industry partners to scale up the existing material, I would love to see validation of this need with industry partners before further synthetic efforts are taken.

PI RESPONSE TO REVIEWER COMMENTS

- We appreciate the positive and constructive feedback from the review panel. In terms of nylon properties, we will work with our industry partners to address the excellent feedback that was provided. The work on incorporating beta-KA into nylons was originally intended to focus on applications where higher glass transition temperatures would be useful (e.g., in automotive applications). In terms of DEI, we are drafting our formal DEI plan for FY 2024–FY 2026, but we have had impacts with workforce development, MSI outreach, and open-source tools. We agree that small molecules offer a new and exciting direction for formulated products, and we have shifted a substantial part of our portfolio in that direction. The heat and light stabilizer concept is also excellent—we will look into that. In terms of the comment regarding more industry involvement, we are actively working with industry. It is also notable that this is a fairly small project, and we are attempting to increase our industry engagement activities through supplementary programs like Energy I-Corps, the Technology Commercialization Fund, the West Gate Program, etc. We have active collaborations with companies as well as ongoing technology transfer. Last, we appreciate and agree with the concept of a two-pronged approach, wherein one project goal is to keep the discovery pipeline full and the other is focused on transitioning winners out of the discovery pipeline to larger scales, including with industry partners.

RESIN: RESPONSIBLE INNOVATION FOR HIGHLY RECYCLABLE PLASTICS

Northwestern University

PROJECT DESCRIPTION

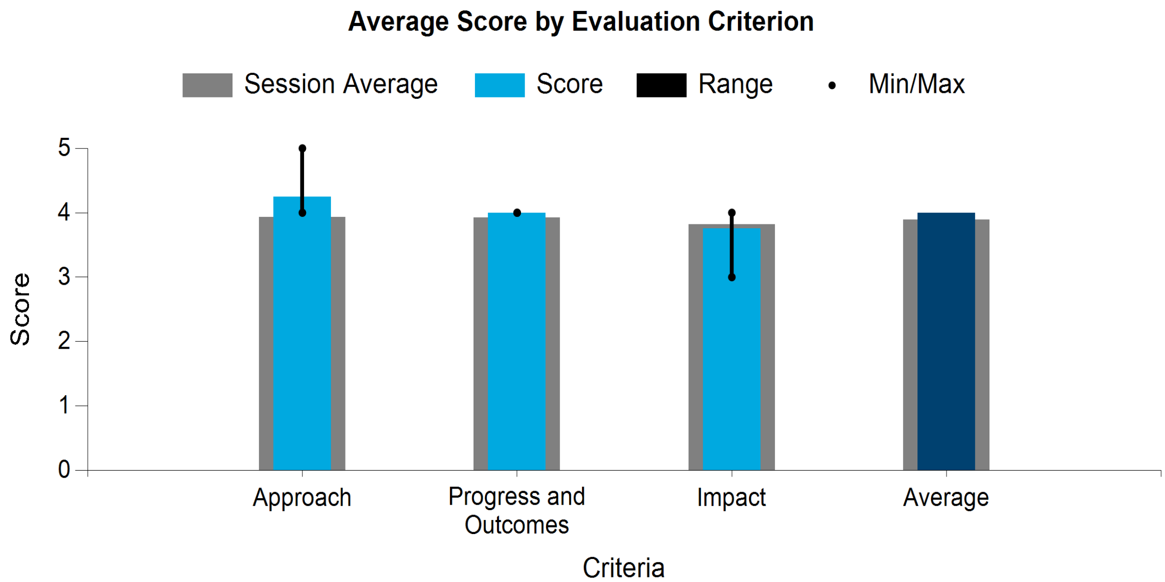
WBS:	2.3.4.607
Presenter(s):	Linda Broadbelt
Project Start Date:	10/01/2019
Planned Project End Date:	03/31/2023
Total Funding:	$3,156,484

As far as objectives, we are developing a responsible innovation approach that marries computational-based approaches with experiments to design materials that achieve high polymer recyclability and benign degradation products at end of life (EOL). We target the PU family of polymers, which ranks sixth in worldwide production, at 36 billion pounds produced in 2016, and has a 6% annual growth rate. PU is not recycled at any significant level when made of linear chains (thermoplastics) or cross-linked networks (thermosets). The state of the art (SOA) is 0% recovered monomers for PU.

The Northwestern University and Argonne National Laboratory team, in collaboration with its industry partners, focuses its efforts on the responsible recycling of bio-based PU-like materials, namely, bio-based polyhydroxyurethanes (PHUs) and polythiourethanes (PTUs), that offer the possibility of recovering value and improving sustainability in two ways: (a) recovery of monomer from spent materials, whether thermoplastics or thermosets, and (b) reprocessability of spent networks with full recovery of cross-link density and associated properties after reprocessing. Our approach is designed to meet the monomer recovery challenge as well as put thermosets on par with thermoplastics, where the melt-state reprocessing of spent polymer into recycled high-value products that meet original use guidelines is the most energy-efficient and responsible method of recycling.

In terms of approach, the overall design framework begins with biomass-derived intermediates as starting molecules. A computational framework for reaction pathway design is applied to this to generate potential monomers. These monomers are used to synthesize PHU and PTU that can be reversibly thermally reprocessed (chemically recycled) for their original use. Experimental studies and kinetic Monte Carlo simulations are used to explore conditions for optimal chemical recycling. Experimental design and kinetic Monte Carlo simulations of monomer recovery for PHU and PTU are also conducted, exploring conditions for high monomer yield. Environmental and economic analyses are being conducted in concert with these studies. Risk assessment for environmental performance is used to understand the potential impacts of the plastics' disposal pathways and the exposure analysis of the polymers and their monomers. Testing for EOL properties involves both engineered and natural environments. Finally, LCA and TEA are done to assess alternatives, explore economic feasibility, and evaluate sustainability.

In terms of impact, the project impacts multiple fronts. We have achieved chemical recyclability of 25% monomer recovery. We are developing modeling and analysis tools to guide the production and recycling of PHUs and PTUs that are at least 50% biomass-derived, benign at EOL, cost-effective, and less energy-, GHG-, and water-intensive than baseline PUs. An additional focus is to develop PHUs and PTUs that exhibit equivalent or improved properties compared to conventional PUs. Further outcomes of the project are publicly available tools for PHU and PTU design for recyclability, performance, and benignity that can be evolved to cover other types of polymers and publicly available cost and environmental assessment tools for recyclable bio-based polymers. A critical outcome is the demonstration of the successful design of a market-relevant polymer while incorporating key performance requirements and EOL considerations that can be replicated for other polymer types.



COMMENTS

- The prominence of the EOL property testing was much appreciated, and I would encourage that to be part of other projects as well.

- The proposed tasks and interplay between activities are well thought out, and the project is clearly aligned with FOA goals. It is highly ambitious, spanning new bio-based monomer discovery, novel polymer synthesis, and a novel prospective risk model for new polymers' life cycle. Having a clear framework for how to navigate this extremely broad space is critical for keeping the project focused and progressing. The fact that the project pivoted after guidance from the industry partner on the need to test foaming is encouraging and increases the likelihood of a commercial fit for these materials. Strengths of the project are the strategic risk assessment framework, the focus on understanding EOL degradation, and the use of TEA/LCA to identify monomers with the largest benefit for a bio versus fossil route. The focus on engaging industry collaborators and the full supply chain focus with the Greenhouse Gases, Regulated Emissions, and Energy Used in Technologies (GREET) model increases the chances of commercialization. The insight gained into critical properties for environmentally benign target products (Milestone 4.5) will be valuable in assessing other proposed biodegradable TPU systems. Likewise, the dissemination of the molecular discovery efforts will allow insights to be leveraged beyond this project for other development efforts in this space. The dynamic bonds leveraged for reprocessing (assessed by multiple pressing cycles) will also lead to substantial differences in flow behavior during processing. To understand the application space for these new materials, it would be useful to keep a keen eye on the rheological requirements for processes to flag potential roadblocks and to work with industry partners to focus on the most promising application space to evaluate material potential. The materials can foam and then be reprocessed into film, but due to the foaming mechanism, they cannot be re-foamed, meaning that a different application would need to be identified for recovered material unless chemically recycled.

- The PIs responded to and addressed the substantial concerns in the 2021 review. This is an aggressive and detailed analysis of these PU derivatives and promises to deliver a very comprehensive product. This project seeks to identify derivatives that bring higher functional value to suppliers and an additional sustainability benefit. The blueprint has the potential to have a greater industrial impact and pull. I am pleased that the team has an excellent understanding of risk areas and mitigation strategies in place. The work to date is reassuring. There is still a question as to whether, with full transparency and guidance from industry partners, these designed bio-phased PUs will gain entry and sufficient traction to enter the market, when there are always hurdles for business adoption.

- The close collaboration with industry is fantastic. The team is taking on the feedback that they need to produce a foam. The team is transferring material to Dow, producing co-publications, and receiving samples from Dow and Jenner. Close collaboration in this field of work is great preparation for bringing these technologies into the real world.

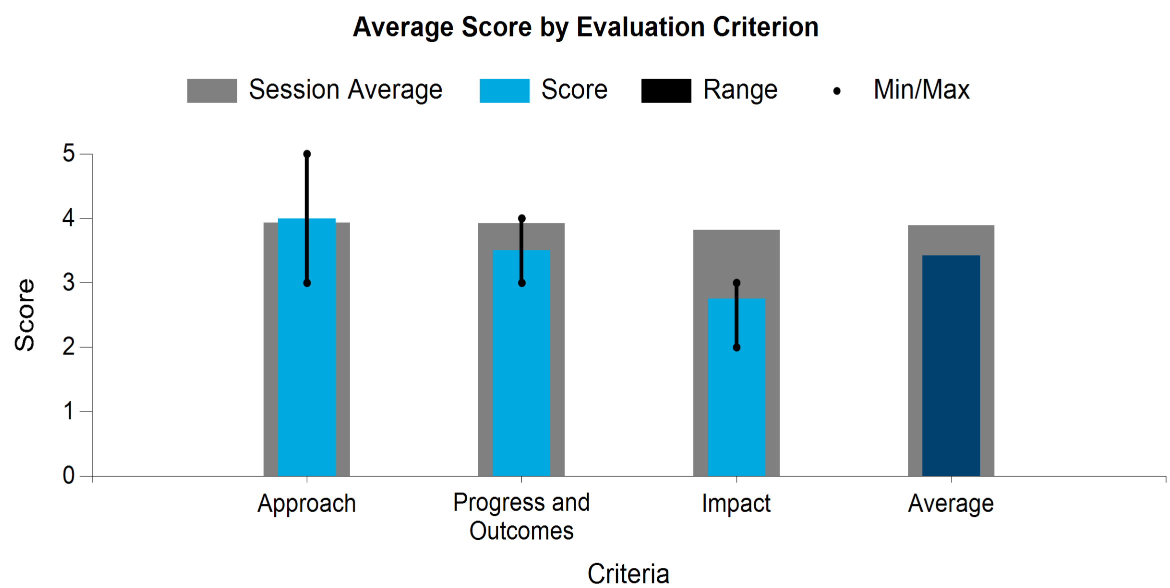
PI RESPONSE TO REVIEWER COMMENTS

- We appreciate the reviewers' positive and insightful comments regarding our project. We are particularly excited that they noted the strong partnership with industry and how the input of industry partners was critical in guiding, and indeed pivoting, the focus of our project. We are grateful that the reviewers appreciated how our team collaborated to bring a combined systems-level and focused science approach to a grand challenge, providing a model for the future. We would also like to note that DOE's support for a tightly integrated project among a university, a national laboratory, and industry that adopted a cradle-to-grave approach and was supported by detailed risk analysis, LCA, and TEA was greatly appreciated.

MULTI-UNIVERSITY CENTER ON CHEMICAL UPCYCLING OF WASTE PLASTICS (CUWP)

University of Wisconsin–Madison

WBS:	2.3.4.613
Presenter(s):	George Huber
Project Start Date:	10/01/2020
Planned Project End Date:	09/30/2025
Total Funding:	$12,500,000



COMMENTS

- The overall project structure and organization is clear. There is a clear logic to the interplay between the different focus areas as well as a clear management structure. My main concerns with the project are related to engagement with the wider ecosystem (MRFs, recyclers, and companies engaged in the pyrolysis space as well as other DOE consortia and institutes), such that the research activities are adding value and insight to the space and not duplicating information that already exists. Enhancing our understanding of pyrolysis is only impactful if it is connected with a path to commercialization. It seems like an excellent opportunity to build bridges between BOTTLE and Chemical Upcycling of Waste Plastics (CUWP), with their different research portfolios, to accelerate the entire polymer circularity space. Additionally, it may be fruitful to engage with the Reducing Embodied Energy and Decreasing Emissions (REMADE) Institute and the Biomass Feedstock National User Facility for opportunities and limitations on MRFs and sortation. In order to realize the ambition of being a global leader in plastic recycling technology R&D, wider and more intentional engagement is needed. A particularly nice output from the project is the view on what infrastructure is needed and how to optimally locate it. A stronger environmental justice lens for this analysis is recommended to avoid locating facilities in areas with already disproportionate burdens. For the educational aspect of the project, tracking the impact of materials would be valuable. Are the fact sheets or course materials being used by organizations outside CUWP? Which groups are being reached successfully, and which need different approaches? Understanding the reverse supply chains and the composition of recycling streams is critical, and it is good that this is part of the project focus; however, I am concerned that there was no mention of reaching out to other actors in this space. For example, the team could engage with surveys such as the Recycling Partnership's Residential MRF Survey, Stina's Annual Plastic Recycling Survey (https://stinainc.com/view/annualrecycling), Resource Recycling Systems' recently completed film recovery survey for California, and BOTTLE's analysis of plastic recycling systems ("Quantification and Evaluation of Plastic Waste in the United States"). Hand sorting a few bales is hugely time-intensive and yields low statistical power insights. Engaging with the recycling infrastructure to understand composition will be more fruitful. I fail to understand how the results of testing molded tensile bars from the bale is advancing understanding; this is something that is actively done at recyclers in this space, and it feels like engagement with them would be more useful. The solvent targeted recovery and precipitation (STRAP) work is among the strongest from the project, with partners interested in commercialization and scale-up work ongoing. As there are other solvent-based recovery technologies coming into the market, such as Pure Loop, understanding the TEA/LCA for STRAP against the wider landscape is critical. It is worth considering how insights into the variability of waste streams with location, season, and potentially long-term material shifts would impact the sizing, location, and design of STRAP facilities.

- The wide collaboration is impressive. The focus on pyrolysis is not innovative. The impact of the ML database is not clear. It was unclear whether the solvent analysis includes the costs to dispose of waste solvent (it should). The work sorting bales was done by hand by a graduate student and does not seem to be replicable in any scalable, financially viable way. I would like to know how they identify the number of the matrix plastic; that seems important and difficult.

- The absence of synergy between teams noted in the last review was addressed with regular meetings with constituencies within the consortium. I applaud the strengthening of the CUWP management structure and the existence of a board with representation from all stakeholders, including the IAB. Recovery from multilayers seems particularly solvent-intensive. The STRAP process is excellent, and I would like to see a validation step—a side-by-side comparison of STRAP-recovered polymers against virgin polymer in a multilayer film food application as a way to demonstrate equivalency in performance. One point of concern: The economics modeling that shows value in siting pyrolysis plants in high-population-density areas raises my concern for having a rigorous study of the impact through an environmental justice lens. I would encourage the investigators to engage with entities to thoroughly vet the potential environmental impact on the adjacent communities.

- For the research-focused aspect of the program, it would be great to collect data on the number of students that have attended the class, the number of undergraduate researchers that are sponsored, and the number of students that have participated in the exchange program. Bringing samples to share is fantastic! I believe that writing a review article at the beginning of the funding period is a great idea and could be included in several of the other funding programs. I think it is a very positive sign that the individual steps are building on each other, e.g., using the actual pyrolysis oils in the zeolite-guided conversion. This is important to demonstrate the full life cycle of the material.

PI RESPONSE TO REVIEWER COMMENTS

- We would first like to thank the reviewers for their kind comments about CUWP. We are very grateful to DOE for supporting this work. We are excited about everything that CUWP has accomplished to date, and we look forward to continuing to address the scientific and engineering challenges with plastic recycling. In the following, we offer our responses to the main comments by the reviewers.

- Comment (Reviewer 1): The overall project structure and organization is clear. There is a clear logic to the interplay between the different focus areas as well as a clear management structure. My main concerns with the project are related to engagement with the wider ecosystem (MRFs, recyclers, and companies engaged in the pyrolysis space as well as other DOE consortia and institutes), such that the research activities are adding value and insight to the space and not duplicating information that already exists.

- Response: Thank you for this comment. We received feedback from several companies engaged in plastic pyrolysis during the CUWP Annual Meeting. These companies are already part of the CUWP IAB. We will invite companies that own MRFs (like Waste Management) to participate in CUWP's IAB and are open to engaging with the wider ecosystem.

- Comment (Reviewer 1): Enhancing our understanding of pyrolysis is only impactful if it is connected with a path to commercialization.

- Response: We agree that the impact of any technology is measured by its eventual commercialization. Several of CUWP's IAB members are advancing pyrolysis to commercialization, including Anellotech, Braskem, Frontline, GSF, Ketjen, Saudi Basic Industries Corporation, and GAL Group. Independent of the efforts of CUWP, a number of companies are attempting to recycle or upcycle waste plastics via thermal depolymerization, including Agilyx, BASF, Brightmark Energy, ExxonMobil, Full Circle, Freepoint Eco-Systems, Nexus Fuels, Plastic2Oil, Plastic Energy, Renewlogy, and RES Polyflow. The CUWP IAB members are interested in advancing the current state of technology in pyrolysis to improve its technical and economic prospects for processing waste plastics into value-added products. Our experimental and computational studies on thermal depolymerization help overcome the challenges of plastics upcycling identified by TEA/LCA performed at CUWP. We also have a patent application on pyrolysis technology and are working with CUWP members to understand how it could be commercialized.

- Comment (Reviewer 1): It seems like an excellent opportunity to build bridges between BOTTLE and CUWP, with their different research portfolios, to accelerate the entire polymer circularity space. Additionally, it may be fruitful to engage with the REMADE Institute and the Biomass Feedstock National User Facility for opportunities and limitations on MRFs and sortation.

- Response: Further interaction with researchers within this space is indeed an important goal. In the near term, several members of CUWP will be attending the Plastics Recycling Gordon Research Conference in summer 2023, which will promote discussion with a variety of researchers (including those affiliated with BOTTLE and REMADE). We also plan to organize a mini symposium as part of the American Institute of Chemical Engineers annual meeting to establish further connections between groups.

- Comment (Reviewer 1): In order to realize the ambition of being a global leader in plastic recycling technology R&D, wider and more intentional engagement is needed.

- Response: DOE has limited us to partners that have operations in North America. We have attended international conferences and will continue to do so; however, DOE will have to give us permission to engage with the recycling community outside of North America.

- Comment (Reviewer 1): A particularly nice output from the project is the view on what infrastructure is needed and how to optimally locate it. A stronger environmental justice lens for this analysis is recommended to avoid locating facilities in areas with already disproportionate burdens.

- Response: Our computational models will be adapted to account for constraints on technology locations; here, it will be interesting to understand the interplay among environmental justice, carbon emissions, and economics (that might result from higher transportation demands). We are also currently developing a computational model to evaluate the impact of new recycling technologies on social justice in Mexico.

- Comment (Reviewer 1): For the educational aspect of the project, tracking the impact of materials would be valuable. Are the fact sheets or course materials being used by organizations outside CUWP? Which groups are being reached successfully, and which need different approaches? Understanding the reverse supply chains and the composition of recycling streams is critical, and it is good that this is part of the project focus.

- Response: We have provided the fact sheet to plastic converters and the media. We have provided the course material on plastic recycling to professors at universities outside of CUWP.

- Comment (Reviewer 1): I am concerned that there was no mention of reaching out to other actors in this space. For example, the team could engage with surveys such as the Recycling Partnership's Residential MRF Survey, Stina's Annual Plastic Recycling Survey (https://stinainc.com/view/annualrecycling), Resource Recycling Systems' recently completed film recovery survey for California, and BOTTLE's analysis of plastic recycling systems ("Quantification and Evaluation of Plastic Waste in the United States"). Hand sorting a few bales is hugely time-intensive and yields low statistical power insights. Engaging with the recycling infrastructure to understand composition will be more fruitful. I fail to understand how the results of testing molded tensile bars from the bale is advancing understanding; this is something that is actively done at recyclers in this space, and it feels like engagement with them would be more useful.

- Response: Thank you for the comments and suggestion. We are currently working with multiple MRFs and recyclers across the country. We do not have data on the composition and performance of sorted mixed waste landfill plastics. There is also no compositional data on mixed #3–#7 bales (in the United States), and as result, there is no performance data on the properties of sorted #3–#7 mixed bales. The process for sorting a whole bale is necessary to understand the entire composition, which, in turn, allows for implementation of ASTM International D5231 for sampling mixed waste for further processing. Per ASTM, washing, pelletizing, and molding is the globally accepted method for understanding the mechanical properties of these landfill-diverted mixed waste plastics for downstream operations. This characterization via injection molding helps MRFs understand which markets are suitable for these currently landfilled mixed waste bales. The injection process is a critical step in the optimization of new polymer delivery systems for the thermal oxo-degradation and pyrolysis teams. MRFs and recyclers do not mold test strips on #3–#7, and very little (if any) of this work is done by recyclers on recovered materials for characterization. Very limited characterization is done at converters on bales that have established markets. We have active relationships with six MRFs across the country. We have completed site visits at four MRFs and are planning to visit two more in late summer and early fall 2023.

- Comment (Reviewer 1): The STRAP work is among the strongest from the project, with partners interested in commercialization and scale-up work ongoing. As there are other solvent-based recovery technologies coming into the market, such as Pure Loop, understanding the TEA/LCA for STRAP against the wider landscape is critical. It is worth considering how insights into the variability of waste streams with location, season, and potentially long-term material shifts would impact the sizing, location, and design of STRAP facilities.

- Response: We appreciate the positive comment. We have compared STRAP to two competing solvent-based recycling processes (PureCycle and CreaSolv) based on information in the literature. We believe that STRAP has potential advantages, and we have applied for intellectual property (IP) in these areas. Moreover, we have already hosted a representative from CreaSolv at a CUWP seminar to exchange ideas and learn about their technology. We will host a representative from PureCycle as a seminar speaker in summer 2023. These conversations have already been helpful in understanding potential challenges, and possible solutions, that will be faced during scale-up of STRAP.

- Comment (Reviewer 2): The wide collaboration is impressive. The focus on pyrolysis is not innovative.
- Response: A more accurate characterization of plastics pyrolysis is that it is not a new technology; however, innovation can occur at any stage in the life cycle of a technology. Pyrolysis has been attractive to industry for many years because of its robustness in handling diverse plastic wastes and its ability to process those wastes at relatively high rates; however, pyrolysis faces distinct challenges that call for innovation in energy efficiency, contaminant neutralization, and products that have higher value than fuel oil. The CUWP pyrolysis program is targeting these challenges through studies on generating high-value olefins, aromatics, and even oleochemicals from waste plastics and eliminating external energy demand through autothermal operation. These innovations, along with emerging policies mandating better management of waste plastics, will make pyrolysis economically and environmentally attractive.
- Comment (Reviewer 2): The impact of the ML database is not clear.
- Response: Using characterization data collected by each MRF for landfill-diverted waste, we will establish an ML database that can be used to identify optimized pathways for each feedstock. The database will be used to determine the suitability of different waste streams for different processing pathways (particularly STRAP versus pyrolysis). The goal will also be to predict costs and emissions associated with such processing.
- Comment (Reviewer 2): It was unclear whether the solvent analysis includes costs to dispose of waste solvent (it should).
- Response: Currently, the TEA model does not account for waste solvent disposal, as we assume a high solvent recycling rate; however, we agree that this is an issue that needs to be considered, and it will be handled in our future models.
- Comment (Reviewer 2): The work sorting bales was done by hand by a graduate student and does not seem to be replicable in any scalable, financially viable way. I would like to know how they identify the number of the matrix plastic; that seems important and difficult.
- Response: Hand sorting is the accepted ASTM method for analyzing municipal solid waste (MSW) from landfill-diverted materials. The hand sorting is not intended to be scaled for MRFs or recovery facilities but instead used for analysis of a bale for research and data collection; however, most MRFs and recovery facilities are using manual sorting of recovered waste, especially MRFs in rural areas. As previously stated, we are currently working with multiple MRFs and recyclers across the country, and data on composition and performance of sorted mixed waste landfill plastics is not known. There is no compositional data on mixed #3–#7 bales (in the United States), and as result, there is no performance data on the properties of sorted #3–#7 mixed bales. The process for sorting a whole bale is necessary to understand the entire composition, which, in turn, allows for the implementation of ASTM D5231 for sampling mixed waste for further processing. Per ASTM, washing, pelletizing, and molding is the globally accepted method for understanding the mechanical properties of these landfill-diverted mixed waste plastics for downstream operations.
- Comment (Reviewer 3): The absence of synergy between teams noted in the last review was addressed with regular meetings with constituencies within the consortium. I applaud the strengthening of the CUWP management structure and the existence of a board with representation from all stakeholders, including the IAB. Recovery from multilayers seems particularly solvent-intensive. The STRAP process is excellent, and I would like to see a validation step—a side-by-side comparison of STRAP-recovered

polymers against virgin polymer in a multilayer film food application as a way to demonstrate equivalency in performance.

- Response: We agree with the reviewer, and we are already working on characterization of STRAP-recovered polymers to compare against virgin resins (as a collaboration between Topic Area 2 and Topic Area 5). Our data show that key properties of the recovered resins—including molecular weight distributions, thermal properties, and melt flow indices—are very similar to virgin resins. We have further scaled up the production of STRAP materials to recreate a cast polyethylene film for comparison to films from virgin resins. We will continue to incorporate such comparisons in our future studies.
- Comment (Reviewer 3): One point of concern: The economics modeling that shows value in siting pyrolysis plants in high-population-density areas raises my concern for having a rigorous study of the impact through an environmental justice lens. I would encourage the investigators to engage with entities to thoroughly vet the potential environmental impact on the adjacent communities.
- Response: We agree with the reviewer. Our computational models will be adapted to account for constraints on technology locations; here, it will be interesting to understand the interplay among environmental justice, carbon emissions, and economics (that might result from higher transportation demands). We are also currently developing a computational model to evaluate the impact of new recycling technologies on social justice in Mexico.
- Comment (Reviewer 4): For the research-focused aspect of the program, it would be great to collect data on the number of students that have attended the class, the number of undergraduate researchers that are sponsored, and the number of students that have participated in the exchange program.
- Response: Thank you for this comment. We will collect the data and provide this information in future reports.

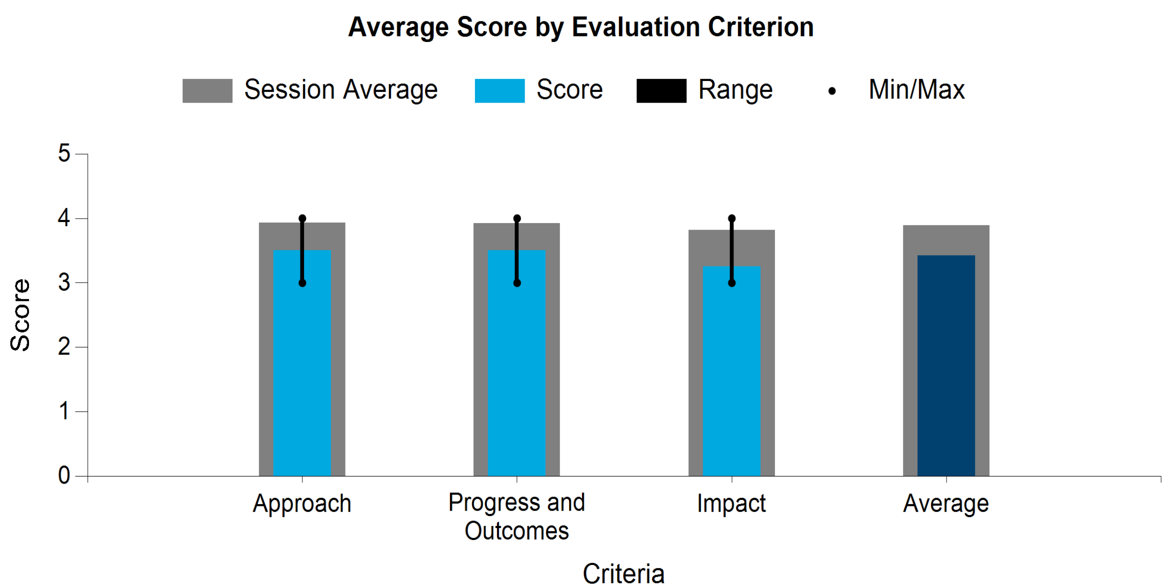
PRODUCTION OF HIGH-PERFORMANCE BIODEGRADABLE POLYURETHANE PRODUCTS MADE FROM ALGAE PRECURSORS

University of California San Diego

PROJECT DESCRIPTION

WBS:	2.3.4.615
Presenter(s):	Michael Burkart
Project Start Date:	01/01/2021
Planned Project End Date:	02/28/2024
Total Funding:	$2,500,000

For this program, we are adapting and scaling chemistry to convert algae oil into monomers for PU products, with a focus on TPUs. We previously developed routes to prepare both components of PU materials—polyols and diisocyanates—from renewable sources. We also showed that our PUs can biodegrade under compost conditions. We are expanding on these to increase the renewable content and the algae content, as well as ensuring that our TPUs retain biodegradability. We are preparing polyols with 80%–100% renewable content and ensuring that they retain properties as drop-in replacements for manufacturing. We have scaled a flow chemistry process for diisocyanate preparation and are optimizing it for yield and purity. To date, by combining these processes, we have prepared TPUs with a bio-content of 85% and an algae content of 61%. These materials show excellent physical characteristics, on par with commercial petroleum TPUs. We have initiated a route to a second algae-sourced diisocyanate via enzymatic epoxidation of polyunsaturated fatty acids. We have prepared a variety of product prototypes with renewable and biodegradable TPUs, including injection molded cell phone cases, coated fabrics, and 3D-printed filament. Finally, we have initiated TEA and LCA on the scaled processes.



COMMENTS

- Although key risks are identified, the proposed mitigations are very high level. A deeper explanation of how these technical challenges would be overcome would improve the chances of success. The general logic of the project is clear; however, the potential material design space is vast, and there does not seem to be a methodical approach to how it will be explored (e.g., what we *should* make from the range of what we *can* make). Interaction with other projects focused on developing biodegradable TPU-like materials could increase the probability of success. The novelty and strength of the project are in the synthetic routes from algae to diisocyanates and novel polyols. This aspect is well done and is progressing. The application and material performance aspects are significantly weaker. How does using heptamethylene diisocyanate (7HDI) for a TPU impact properties relative to the dominant isocyanates used in the market (such as methylene diphenyl diisocyanate (mdi) and others)? The slower reactivity could be a showstopper for commercialization or could limit the range of applications. Engaging an industry partner in the TPU space to give guidance and feedback here would increase the potential for commercial success. There is a huge variety of thermoplastic TPUs in the world that have been optimized for different processing or application spaces. The application space for these new proposed materials feels naïve (phone cases, watches)—these applications are highly cosmetic (color, feel, surface finish), which plays a key role in their potential success. Working with industry to gain more insight into how to match TPU physical and chemical properties to application spaces would increase the likelihood of identifying application spaces where the commercialization of these materials may be feasible. Finding a partner who is currently active as a TPU supplier or utilizer would strengthen the impact potential. Additionally, the logic of making these applications biodegradable rather than recyclable is not obvious. Identifying applications where the use is inherently dissipative (e.g., agriculture, paint) would be a stronger case. Similarly, the end-of-project milestone needs a sharper definition of performance and TEA/LCA to demonstrate industrial potential.

- The risks and mitigation slides at the beginning did not embody the risks and mitigation approaches described in the rest of the presentation. The approach slide needed to be reformatted to convey the intended information. The presentation became more informative at slide 8. I did not see information on communications, either within the project team or with entities outside the project team, including commercialization.

- Applying LCA/TEA upfront would be beneficial to point out critical steps that might be detrimental in scale-up and environmental impact. The team has shown progress in making the prepolymers, but process optimization is required to address slow polymerization. In terms of synthesis of diisocyanate, the team achieved very good yields of the diisocyanate product. The prototypes were impressive. How are the commercial project partners, BASF and PepsiCo, engaged to drive toward a commercially robust process with a targeted application that is realistic? On the initial biodegradation analyses: Are the tests performed with virgin polymer, or are they formulated with appropriate stabilizers for the intended applications (e.g., stabilizers typical for oxidation, thermal stability, and so on)?

- The team has made great progress toward developing their flow reactor and is close to reaching their productive target of grams per month. I applaud the team for producing application prototypes and believe that their plan to bring in industry partners to further this effort is essential. I would recommend that the team start their TEA/LCA as soon as feasible and integrate the learnings from the analysis back into the development of the flow reactor.

PI RESPONSE TO REVIEWER COMMENTS

- Comment: Although key risks are identified, the proposed mitigations are very high level. A deeper explanation of how these technical challenges would be overcome would improve the chances of success. The general logic of the project is clear; however, the potential material design space is vast, and there does not seem to be a methodical approach to how it will be explored (e.g., what we *should* make from the range of what we *can* make). Interaction with other projects focused on developing biodegradable TPU-like materials could increase the probability of success.

- Response: The full BOTTLE team, including both the University of California San Deigo and Algenesis teams, meets weekly to discuss goals, progress, and applications. Algenesis has clear goals for metrics for specific materials, specifically TPUs suitable for injection molding (for heel counters and other TPU-based footwear materials) and fabric coating. Unfortunately, the presentation format did not allow for a full discussion of these product developments.

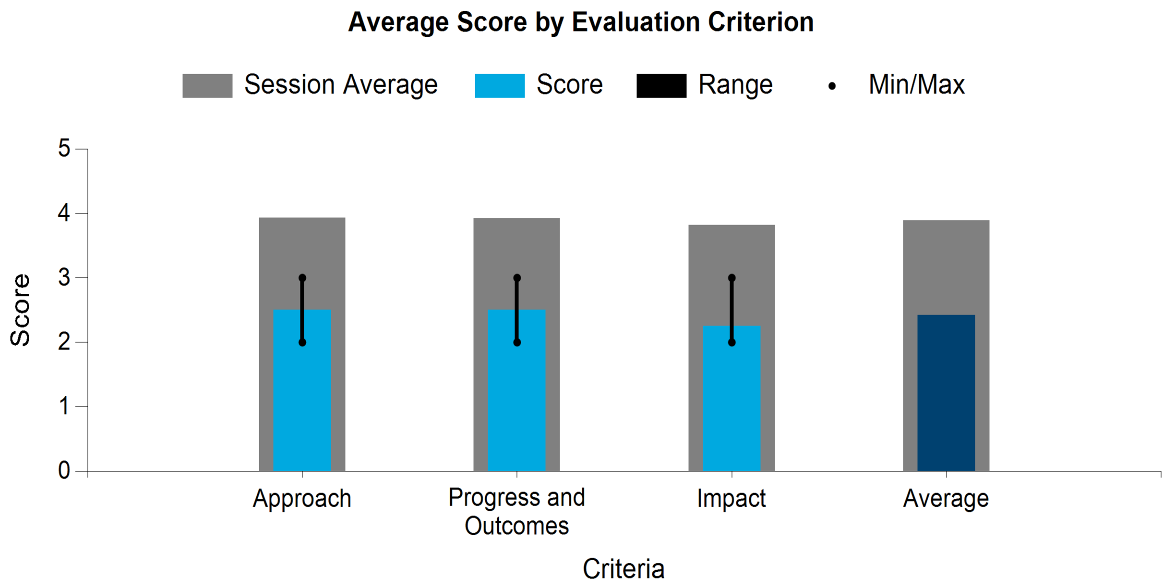
- Comment: The novelty and strength of the project are in the synthetic routes from algae to diisocyanates and novel polyols. This aspect is well done and is progressing. The application and material performance aspects are significantly weaker. How does using 7HDI for a TPU impact properties relative to the dominant isocyanates used in the market (such as mdi and others)? The slower reactivity could be a showstopper for commercialization or could limit the range of applications.
- Response: Both aromatic diisocyanates and aliphatic diisocyanates are used in commercial TPUs. Aliphatics have lower reactivity in general; therefore, TPUs must be cured prior to use. This is standard for the industry. 7HDI appears to be a drop-in replacement for other aliphatic isocyanates, such as hexamethylene diisocyanate (6HDI). According to our preliminary formulations and experiments, 7HDI reacts nearly identically to 6HDI. We believe that any differences in TPU material metrics can be addressed by formulation optimization, an area of significant expertise for Algenesis.
- Comment: Engaging an industry partner in the TPU space to give guidance and feedback here would increase the potential for commercial success. There is a huge variety of thermoplastic TPUs in the world that have been optimized for different processing or application spaces. The application space for these new proposed materials feels naïve (phone cases, watches)—these applications are highly cosmetic (color, feel, surface finish), which plays a key role in their potential success.
- Response: Algenesis has been working with large TPU manufacturers and users. For instance, the large engineered polymer company Trelleborg recently announced a collaboration with Algenesis for production of coated fabrics (https://www.trelleborg.com/en/engineered-coated-fabrics/media/trelleborg-partners-with-algenesis-to-create-sustainable-thermoplastic-polyurethanes). Therefore, we have an active engagement with product development and with end users.
- Comment: Working with industry to gain more insight into how to match TPU physical and chemical properties to application spaces would increase the likelihood of identifying application spaces where the commercialization of these materials may be feasible. Finding a partner who is currently active as a TPU supplier or utilizer would strengthen the impact potential.
- Response: We have industry partners who are actively interested and engaged in our research and downstream products. This includes cost-share partners PepsiCo and BASF. We have access to these companies to get feedback and advice, and we have had regular (monthly) interactions with members of their teams.
- Comment: Additionally, the logic of making these applications biodegradable rather than recyclable is not obvious. Identifying applications where the use is inherently dissipative (e.g., agriculture, paint) would be a stronger case.
- Response: It has become increasingly clear to us that recycling is not going to save our planet from the dangers of plastic waste. It has been reported that less than 5% of recyclable plastic is ever actually recycled, and that all plastics (even recycled ones) can end up as microplastics in our waterways and oceans. Microplastics can now be found in every animal on the planet, in all human organs, and all over the world (from the Arctic to the Himalayas.) Only biodegradable materials will solve this problem. We will make sure future presentations drive these facts home.
- Comment: The end-of-project milestone needs a sharper definition of performance and TEA/LCA to demonstrate industrial potential.
- Response: The TEA/LCA portion of this project has been slated to take place at the end of the program. We will ensure that performance and industrial potential are clarified in this work.

- Comment: The risks and mitigation slides at the beginning did not embody the risks and mitigation approaches described in the rest of the presentation. The approach slide needed to be reformatted to convey the intended information. The presentation became more informative at slide 8.
- Response: We apologize that these were not clarified in the presentation. Unfortunately, the slide template does not allow for great communication of these concepts, nor many of the most exciting aspects of the research.
- Comment: I did not see information on communications, either within the project team or with entities outside the project team, including commercialization.
- Response: We meet weekly to discuss the BOTTLE project with the entire team from both the University of California San Deigo and Algenesis. We have separate LCA/TEA meetings with the University of California San Deigo every two weeks.
- Comment: Applying LCA/TEA upfront would be beneficial to point out critical steps that might be detrimental in scale-up and environmental impact.
- Response: We are performing these TEAs now as part of the final task and final budget period.
- Comment: How are the commercial project partners, BASF and PepsiCo, engaged to drive toward a commercially robust process with a targeted application that is realistic?
- Response: BASF and PepsiCo are cost-share partners who have supported our research through their academic outreach arms—so they are interested, but their product development teams are not yet engaged. Regardless, we have regular interactions with their scientists and discuss applications for most interactions.
- Comment: On the initial biodegradation analyses: Are the tests performed with virgin polymer, or are they formulated with appropriate stabilizers for the intended applications (e.g., stabilizers typical for oxidation, thermal stability, and so on)?
- Response: The tests are done with virgin polymer. Certain additives are used to make the formulations, but they do not include stabilizers as the reviewer suggests, because many of those have been developed to hinder biodegradation and are not consistent with our desired applications.
- Comment: The team has made great progress toward developing their flow reactor and is close to reaching their productive target of grams per month. I applaud the team for producing application prototypes and believe that their plan to bring in industry partners to further this effort is essential.
- Response: Thank you!
- Comment: I would recommend that the team start their TEA/LCA as soon as feasible and integrate the learnings from the analysis back into the development of the flow reactor.
- Response: As stated above, we are beginning this now.

HYBRID APPROACH TO REPURPOSE PLASTICS USING NOVEL ENGINEERED PROCESSES (HARNESS)

Battelle Memorial Institute

WBS:	2.3.4.616
Presenter(s):	Kate Kucharzyk
Project Start Date:	05/01/2021
Planned Project End Date:	04/30/2024
Total Funding:	$2,499,778



COMMENTS

- I agree that there is a great need for foam alternatives. I did not get the information from this presentation that this work is making the connections necessary to achieve this. The answers in the Q&A did not provide the information missing from the presentation.

- This project has a high ambition to move from TRL 0 to TRL 6 during the course of a relatively small project in a 3-year time frame; however, the approach is inadequate for the ambition level. Success depends on multiple steps in the value chain, and it is not clear that there is sufficient engagement between the players. The project approach did not address any of the separation/process design steps needed after degradation of the foams. To date, the accomplishments are screening enzymes on a model compound for degradation and replication of a commercial recipe to form a TPU foam; however, no degradation results or feedstock preprocessing activities appear to have been started, and the LCA/TEA data shown on slide 9 appears to relate to PET, suggesting that the team is assuming the conclusions will hold for TPU. The TEA/LCA should be updated to reflect the materials and processes for this project. Focusing on high-volume polyether-urethane foams that currently do not have a viable recycling option at EOL has the potential for wide impact. If successful in recovering polyol and diamines at sufficient yield and purity, there is a clear route to reintroducing them into the market. Emphasis was put on industry partners, but their level of commitment to moving this project forward is not at all clear and would be critical for any eventual impact.

- The progress in this early-stage project has been satisfactory—it is nice that you have a quick visual (fluorescent) screen for the degradation of the PU. The work in the next four quarters on the tasks on enzyme optimization and upcycling to PU foams will be critical to driving a desirable outcome in the go/no-go decision. Achieving this seems daunting. I am puzzled by the projections to move the project from TRL 0 to TRL 6 at the project end—this seems pretty aggressive for a program developing a proof of concept and building a key technology (e.g., PU foam degrading enzymes). It is laudable that the team has engaged key PU foam formulators to provide an appropriate specification for upcycling to industry standards. It will also be valuable to have detailed economic analyses on the polyols and diamines captured from the PU foams.

- The researchers are looking to grow from TRL 0 to TRL 6 during the course of the project. The team should submit a more detailed plan for reaching these very high TRLs. TRL 6 includes engineering development of the technology as an operational system. The major difference between TRL 5 and TRL 6 is the step up from the laboratory scale to the engineering scale and the determination of scaling factors that will enable the design of the final system. A report by the National Aeronautics and Space Administration in 2015 analyzed 131 projects funded by its Small Business Innovation Research program and found that the average time to advance from TRL 1 to TRL 2 was 1.2 years, from TRL 2 to TRL 3 was 1.4 years, from TRL 3 to TRL 4 was 2.1 years, from TRL 4 to TRL 5 was 3.4 years, and from TRL 5 to TRL 6 was 4.6 years. Based on the presented plan, the scale-up work will be done by the industry partners. I would want to understand in more detail how the industry partners will be participating. Even if the scale-up is developed by industry partners, the group leader should know the challenges and processes required. The "Upscaling of Non-Recyclable Plastic Waste Into CarbonSmart Monomers" project demonstrated a great plan to achieve very high TRL from an early project and showed the types of equipment and systems that are required.

PI RESPONSE TO REVIEWER COMMENTS

- Thank you for the comments and for your time reviewing our project. We believe that we can advance swiftly from TRL 0 to TRL 3 once the enzymatic candidates for PE-PU degradation are found. Our team is already showing degradation of monomers and is starting to test solid feedstock material. The team has not started to work on the separation/process design steps, as these will come later in the program, once we show successful solids degradation. We do have an approach drafted for this work and are happy to share it with the reviewers. Thank you for the comment on the involvement of the industry partners. We created a slide that shows the material flow through our project team. We also have a pretty clear delineation of how the collaborators and industry partners contribute to the work. We are happy to share more information. The process that we are designing breaks down the PE-PU to the monomers. We are not clear yet about their purity and will have to determine what type of recycled foams we can make once the process continues through the next phase of the laboratory work. I apologize that we do not have information on this at the moment. The reviewers are correct that finding candidate enzymes for PE-PU degradation is not an easy task. During the first months of the project, we have spent time building our foundational capabilities, including detection and high-throughput detection assays, selection of proteins for degradation, and characterization of the industrially sourced foam material. We were also delayed by several months due to COVID, so our progress as planned is not on track. We will be requesting a no-cost extension to BP2. We will evaluate our progress (TRL 0–6) after the next milestone and will communicate with the project manager on any roadblocks. As I am reading your comments on the TRL progression, I wonder if we have used a different scale with which DOE is gauging the technology readiness. Based on our understanding, by the end of the project, our team would build a bench-scale prototype system that will be able to handle all the reactions at 10-L scale. We are not planning to bring the process to the industrial scale. We would like to seek more clarity and guidance on this issue. Thank you so much.

INFINITELY RECYCLABLE AND BIODEGRADABLE FILMS FOR IMPROVED FOOD PACKAGING

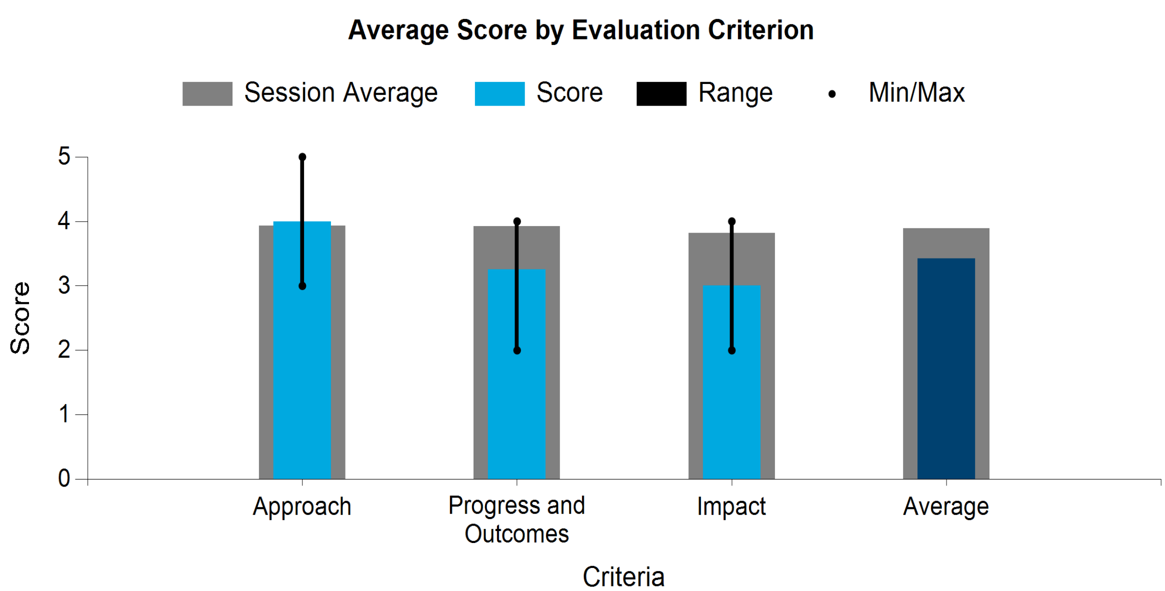
TDA

WBS:	2.3.4.618
Presenter(s):	Allison Robinson
Project Start Date:	10/01/2022
Planned Project End Date:	09/30/2025
Total Funding:	$2,011,426

PROJECT DESCRIPTION

Multilayer packaging is an extremely important and widely used technology; by combining many materials into a single film, the final product can have properties that are unattainable by any single material. Despite their benefits, these films generate an enormous amount of plastic waste. It is critical to transition to sustainable multilayer films, which maintain the benefits of traditional single-use packaging without producing plastic waste. This can be achieved by transitioning from today's fossil-fuel-based polyolefins to an ester-based paradigm that uses bio-derived materials to decarbonize the packaging industry.

In this effort, TDA Research Inc., NREL, and Sulzer will work together to design ester-based films for multilayer packaging. We will optimize a compostable nanocomposite (made of surface-modified cellulose nanofibers in a compostable polymer matrix) to act as a strong food contact layer with low water vapor permeability. We will separately optimize a recyclable-by-design polymer to act as an oxygen barrier layer and as a printable outer layer. The result of this effort will be a sustainable multilayer film that is safe for food contact, has oxygen and vapor barrier properties comparable to traditional films, and is entirely compostable and/or recyclable.



COMMENTS

- The specified risks do not seem comprehensive. The connections among the partners, their roles, and the systems that the project will need to connect to prove success (obtaining inputs, having recipients for outputs) were not shown sufficiently. Each may be succeeding in their individual role, but the role distinctions and relationships were not made clear. Overall, the project appears on track with where the team is in their timeline.

- This project is focused on the formulation of polylactic acid (PLA) nanocomposites and epoxy anhydrides as barrier films. The approach is logical. The risks and mitigations are focused on project management; I would like a more detailed approach toward the technical risks and their mitigation strategies. There is a clear and appropriate mitigation strategy around food contact compliance. I feel that a large risk for this project is that the proposed material will not have the needed melt processability to compete in this space. The proposed plan of hot pressing and extrusion coating will not give sufficient insight. I recommend involving a partner with more insight in the key processing properties needed for these applications early in the project to steer development. The project team is rather process focused (TDA/Sulzer), and I fear that it lacks sufficient insights into the full value chain necessary for the introduction and successful adoption of new polymer films for this market. The project is at an early stage and is encouraged to build this broader perspective now. As the proposed work is very much a product development activity without connection to the end use and processing, I am deeply skeptical that it can be successfully commercialized. The project plan includes specific DEI milestones, and the emphasis on exposing interns to multiple career paths is good. I would like the project team to discuss what they have selected as the SOA for benchmarking their metrics; linear low-density polyethylene is very cheap, suggesting that the cost parity target may be extremely challenging to achieve. Additionally, the cost metric will be influenced by the envisioned film production method (coextrusion, lamination, blown film), and it would be good to see this full route from synthesis to film production (not just pellet production) incorporated into the evaluation metrics.

- This is a well-thought-out plan, and at this early stage, the team is on track. The team provided great details and thoughtful consideration on (a) the nuances of barrier packaging needs for each application, (b) the design and execution of the DEI plan, and (c) navigating the regulatory considerations for food contact surfaces. Also, the end-of-project milestone detailed well-defined targets.

- I appreciate that the team has a plan to use the TEA/LCA results to guide their choice of materials. The team is collaborating with an industry partner, Sulzer, to develop and optimize the manufacturing and bring this technology to market. It is fantastic that the team is already thinking about the food safety of their product and is considering the FDA process for materials in contact with food. The team will consider both composting and chemical recycling for their multilayer films. All three—TDA, NREL, and Sulzer—will develop patents for this, and the goal will be to license to Sulzer. The IP out of this DOE-funded work should not be locked into an exclusive license—especially in the field of biodegradability and food packaging, widespread solutions should be the target.

PI RESPONSE TO REVIEWER COMMENTS

- We appreciate the reviewers' time and feedback on our project. We are happy to see that the reviewers think that the project has a "well-thought-out plan," that "at this early stage, the team is on track," and that "the team provided great details and thoughtful consideration," as these comments highlight what we have been trying to accomplish through this FOA and overall project. Our response to the reviewers' other comments, which focused on technical risks and material baselines, is as follows. The technical risks that we discussed were focused on ensuring that the material meets the barrier property targets we initially set. The barrier properties are the primary factor that determines whether a film will be viable for this application, so this is the most important technical requirement; however, this has been significantly mitigated through our initial demonstrations, which show that we are near the targets before optimization. In our next Project Peer Review, we will discuss the technical risks and mitigations in greater detail. One reviewer had concerns that the proposed material will not have the needed melt processability to compete. The project team believes that the optimized material will be able to compete; PLA films are already produced commercially, and our work to date has not shown evidence that incorporating the cellulosic additives impacts this. Previous work with the epoxy anhydrides has demonstrated that they are sealable and that their tunability could enable the required seal at moderate temperatures, as desired (e.g., T = 50°C). To ensure that this does not prevent commercialization, we will begin investigating these concerns early in the effort, as recommended. Further, we can involve additional partners if needed (as suggested); TDA, NREL, and Sulzer all frequently collaborate with companies across the value chain and have the ability to bring in additional expertise if needed. We selected our SOA materials based on films that have moderate barrier properties and high-volume use in the market. This will allow us to have a significant impact in displacing conventional films when we commercialize our film. If our material can attain better barrier properties (which we anticipate it can), we will be able to target even higher-value films as well. We will perform TEA throughout the project, and we will include the film production method in our calculations to ensure that we are accurately assessing cost parity. Importantly, we are baselining our production against ethylene vinyl alcohol and the total multilayer performance. Marking against any variation of PE would be insufficient. This is because PE alone does not provide the proper barrier properties for food applications, and the multilayer packages derive their ideal properties in conjunction with the critical ethylene vinyl alcohol. Additionally, because we are performing TEA at multiple points throughout the development process, we will be able to change our processing methods and/or material composition as needed to ensure we reach our cost parity goals. To clarify the roles of the team members, NREL is developing epoxy anhydrides, Sulzer is optimizing their PLA formulations, and TDA is developing composites out of TDA's cellulosic additives and Sulzer's PLA. This will make two-layer materials: the epoxy anhydride and the PLA composite. All three entities will work together to combine these materials into a multilayer assembly and to assess their performance. We see this as a collaborative effort among all three team members; although we have our own specializations within the project, we will work together closely throughout the entire effort.

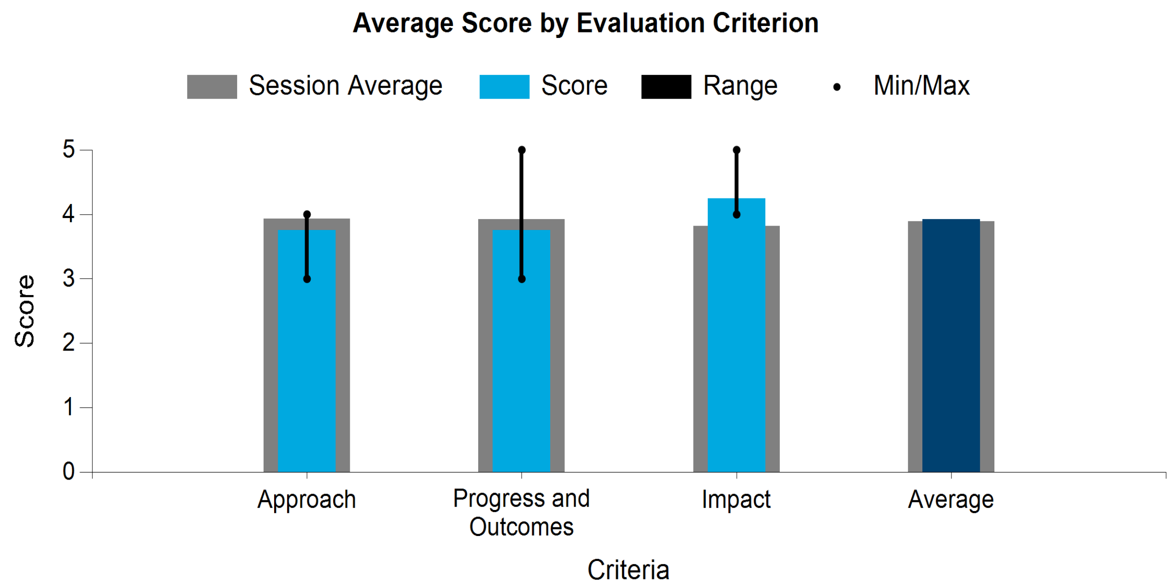
DEVELOPMENT OF INFINITELY RECYCLABLE SINGLE-POLYMER CHEMISTRY BIO-BASED MULTILAYER FILMS USING ETHYLENE/CARBON MONOXIDE COPOLYMERS

Braskem

PROJECT DESCRIPTION

WBS:	2.3.4.620
Presenter(s):	Hadi Mohammadi
Project Start Date:	10/01/2022
Planned Project End Date:	09/30/2025
Total Funding:	$2,500,000

Today, the majority of plastic littering the environment is packaging material. In this project, we will investigate the possibility of producing infinitely recyclable, single-polymer chemistry, multilayer films using bio-based non-alternating ethylene-carbon monoxide (CO) copolymers. We will design two multilayer films to fabricate stand-up pouches for use in packaging applications: (1) multilayer films made of one specific ethylene-CO copolymer and (2) multilayer films made of ethylene-CO copolymers with different CO content. The main recycling route for both of our proposed single-polymer chemistry, multilayer films is mechanical recycling. We will produce at least one prototype of our proposed multilayer films, which, when compared to conventional multilayer packaging, will display (1) 10% cost reduction, (2) comparable performance, (3) 60 wt % to 80 wt % recycled carbon utilization in the final design, (4) 60% energy savings during production, and (5) photodegradability. Cost-competitiveness, sustainability, and lower amounts of energy required for production will increase the value proposition for recycling multilayer films fabricated from bio-based ethylene-CO copolymer, encourage companies to invest in its recycling by decreasing the cost/benefit ratio, free landfills and oceans from its presence, and boost the economy by creating jobs in the recycling field.



COMMENTS

- Slide 5 is nice and clear. I would recommend more graphical slides. The roles of the non-Braskem partners were unclear. Is there no direct role for Braskem in DEI? The answers in the Q&A were clear and informative. I would recommend adding some of these to the presentation, as they will come up again (specifically the LCA-related questions and the existing relationship/current experience with the FDA). Braskem has recycling facility(ies) so can work out source materials internally, which is a benefit to this project in the long term. Similarly, their regular FDA food contact relationship is a useful resource.

- The project aims to produce a CO-bio-ethylene copolymer as an alternative to PE. If successful, it could have large emissions reductions given the volume of PE used. A strength of the project is the active engagement of industry partners, ranging from a material producer (Braskem) to an end user (Unilever), with converters involved via contracting. As Braskem also runs recycling facilities, there is a natural route to evaluating recyclability potential. This full supply chain vision allows clear performance metrics. Food contact compliance has been considered, but the ultraviolet stability of the proposed material is a concern. Biodegradation is not included in this project, as it has already been studied in the literature. As such, there is substantial potential for commercializing the proposed innovation. The project has a plan to address DEI both in terms of recruitment for the project as well as outreach/dissemination of the knowledge into underserved communities. The proposed tasks are logical, but as the project is just beginning, there were insufficient results to judge the quality of the approach and outcomes.

- The proposed plan for this early-stage project seems reasonable, and the engagement of two industry leaders, Braskem and Unilever, is promising. The DEI activities include hiring and educational outreach. Are these centered just at the academic institutions? Please describe the Braskem DEI commitment. How actively engaged is Unilever beyond providing specifications for the multilayer film barrier properties? It would be helpful to see more detail on the activities in BP2, e.g., the number and amount of copolymers to be produced. The technical plan seems lean, and there are a number of technical questions that are wanting. These include considerations for stabilizing these ethylene-CO-copolymers (which should be different than for polyolefin); how the discrete individual layers will be separated and reused given the mechanical recycling proposed; and an articulation of the vision for the process feed of the syngases, ethylene, and CO—from different microbial fermentations?

- This is a very early project. I appreciate that the team is working with its industry partners in developing their food contact products and has an internal group that works on this. They will work with this team to move forward with the regulatory approval. I appreciate that Braskem is open to licensing this technology to other manufacturers so that it can be deployed at scale. I would like to see a TEA/LCA of this project as well. In terms of presentation, the team could include some more graphics and data to illustrate the points they are making.

PI RESPONSE TO REVIEWER COMMENTS

- The presenter and project team would like to thank the reviewers for their thoughtful comments and suggestions. The DEI activities for this project will include hiring and educational outreach activities from academic and industrial institutions. In particular, Braskem will practice its standard hiring procedures where it prioritizes underrepresented comminutes in the interview process. Braskem will also engage in K–12 educational outreach efforts with local schools, such as PlastiVan, with a particular focus on disseminating our knowledge in MSIs and other appropriate institutions serving underserved communities. As a key partner in our project, Unilever will define the target end use for the stand-up pouches, help the team understand the packaging specifications in such applications, contribute to the design of the ethylene-CO-copolymer-based multilayer films, and test the final stand-up pouch made from virgin and recycled material in their facility. The results from the Unilever tests on our stand-up pouch prototypes will allow us to verify the performance of our solution in the target application. In terms of the number and amount of copolymers produced in BP2, we are targeting studying the copolymerization process in at least 20 different conditions with pressures from 40–200 bar, temperatures from 25°–200°C, and CO concentrations from 1%–50%. We will establish the effect of temperature, pressure, and CO concentration on activity of the designated catalyst system during ethylene/CO copolymerization while aiming to achieve a minimum catalyst activity of 500 mol(ethylene)/mol(catalyst)/h at 4 mol% CO incorporation. Regarding the recycling process, our vision is to not separate the individual layers during mechanical recycling. As our targeted solution, we will produce a multilayer film with different ethylene-CO copolymers in the layers. The balance between the mechanical and barrier properties in each layer can be adjusted by varying the CO content in each copolymer. In this design, the adhesion between the layers will be achieved through co-crystallization between the two adjacent layers. As the ethylene-CO copolymers are highly compatible, mechanical recycling of our single-polymer chemistry, ethylene-CO-based, multilayer films is expected to lead to a compatible mixture of the ethylene-CO copolymer with a CO content that is the average of all layers. After adjusting the CO content of the mechanically recycled mixture by blending it with pure PE (decreasing the CO content) or a high-CO ethylene-CO copolymer (increasing the CO content), it will be reused as a layer in the multilayer film structure. Finally, we envision producing bio-based ethylene and CO via two different routes: (1) converting sugars from corn to ethanol through fermentation, and then turning ethanol into ethylene by dehydration; and (2) producing CO from controlled combustion of biomass.

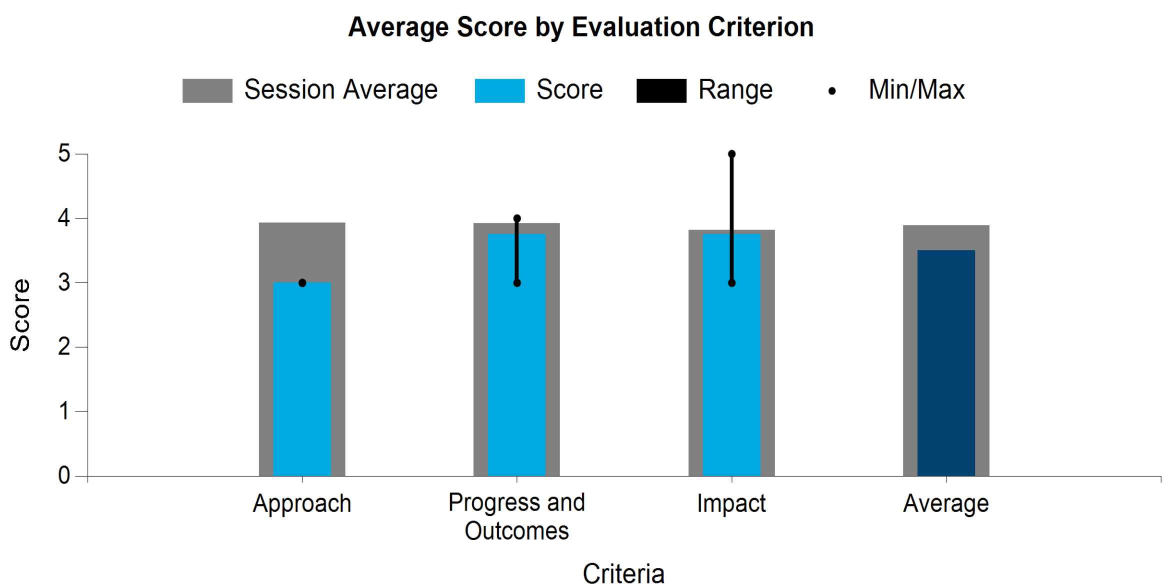
ENABLING LIGNIN VALORIZATION WITH LIQUID-LIQUID CHROMATOGRAPHY

Lignolix Inc.

PROJECT DESCRIPTION

WBS:	2.4.2.200
Presenter(s):	Eric Gottlieb
Project Start Date:	10/01/2021
Planned Project End Date:	03/31/2025
Total Funding:	$3,126,696

This project uses scale liquid-liquid chromatography for the separation and purification of lignin monomers and oligomers from Lignolix's proprietary process. The recovery of lignin products from lignin-rich reductive catalytic fractionation oils is notoriously difficult because they are chemically complex, viscous, contain fine particles, and have broad non-normal molecular weight distributions. Accordingly, these streams are not well suited for separation with traditional simulated moving bed chromatography because the high-molecular-weight compounds lead to rapid fouling of the resin stationary phase. Note also that the target products from these streams cannot be recovered with distillation due to the thermal instability of the solution. Countercurrent chromatography (CCC) is a form of liquid-liquid chromatography that is a scalable and uses the countercurrent motion of a two-phase liquid-liquid system to generate a chromatographic effect on the solvated compounds, separating them into pure components. Accordingly, CCC could represent a paradigm shift in lignin valorization through scaling and deployment in this project. It represents a single-step separation technology for these traditionally difficult-to-process streams due to its high throughput, ability to handle solids, viscous solutions, and low energy consumption. This project is developing the needed chromatography methods, solvent recycling, CCC validation, and TEA/LCA for the separation process.



COMMENTS

- The team is working on the development of continuous CCC for lignin deconstruction product streams. The team has made great progress in a short time. The project is on track. There are lots of similarities between their work and Gregg Beckham's project. I am not sure that this continuous CCC has the potential to create wider and more significant impact. The team should think seriously about feasibility and scalability. The PI needs to develop a DEI strategy and show the progress in the next review period or at the end of the project.

- This is a relatively new project. Internships and DEI efforts are not specifically outlined. It is difficult to separate the objectives of this project from the “Continuous Counter Current Chromatography” project led by NREL. It is good to see that the team is already working with industry to enable lignin-based hot-melt adhesives and 3D printing acrylates as potential commercial products. Good progress has been made in the initial verification and lignin deconstruction. The TEA and LCA show significantly reduced energy consumption and higher productivity.

- This is an early-stage project that, if successful, could provide access to a broad range of new molecules useful for either fuel or chemical product applications. The project approach could use more clarification. If the target is an SAF, the need for individual molecular separation is less clear than if the objective is lignin specialty product valorization. Although there is a good strategy to identify and mitigate risks, the risks associated with scale-up may not be adequately captured. For early-stage projects such as this one, I think that articulating the vision for success through commercial deployment has value, even if it can only be conceptual at this stage of technology development. One comment of concern was that the largest CCC unit in existence had a 5-kilogram/day (?) capacity, which does not support that the risks are low. It appears that there are good collaborative efforts with the labs.

- The presented work is very similar to the “Continuous Counter Current Chromatography” work presented by Gregg Beckham. It would be very helpful in judging progress and milestones if it was clear which work was funded by which grant. I would want to see more details on the team’s DEI efforts and how they will be incorporated into the rest of the work. It is great that the team is currently talking to adhesive companies who can be offtakers for their lignin products to turn them into adhesives.

PI RESPONSE TO REVIEWER COMMENTS

- We thank the reviewers for their valuable feedback, and we appreciate the positive comments on the progress made to date. In response to specific points raised by the reviewers: There are active DEI efforts undertaken under this project, and they will be highlighted in more detail at the next review. Regarding this work in comparison to the “Continuous Counter Current Chromatography” work presented by Gregg Beckham, the works are highly complementary, and we work diligently to ensure that there is no overlap between our efforts. Specifically, this project applies CCC toward the particular mixtures generated from Lignolix’s process for a particular set of commercial targets, which are different from the inputs and outputs of the Separations Consortium’s work. These differences in product streams and separation targets lead to distinct sets of technical targets and optimization constraints around which the continuous CCC technology is being developed. Regarding feasibility and scalability, we agree that this is one of the key risks to this separation approach. We would like to note that the existing upper limit demonstrated for CCC throughput is in a batch mode, and that a continuous CCC is expected to have higher productivity. Also, this project is targeting binary separations, which will yield further improvements to CCC productivity compared to the existing limits.

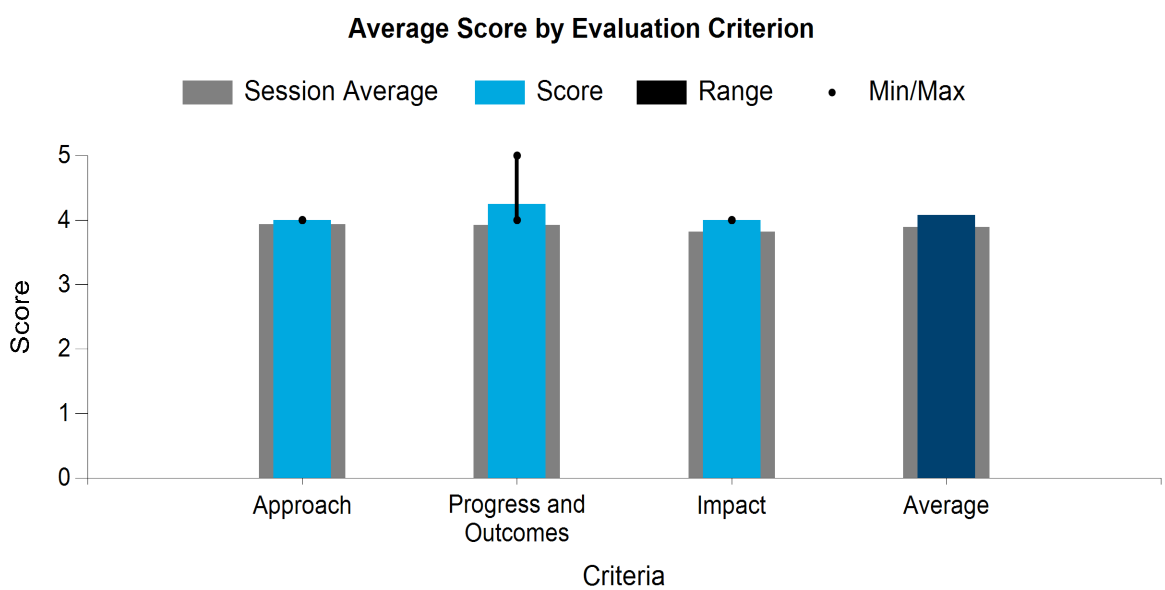
PHYSICAL PROPERTY DATA AND MODELS IN SUPPORT OF BIOPROCESSING SEPARATION TECHNOLOGIES FOR ORGANIC ACIDS SEPARATION

RAPID Manufacturing Institute

WBS:	2.5.1.200
Presenter(s):	Ignasi Palou-Rivera
Project Start Date:	10/01/2021
Planned Project End Date:	04/30/2025
Total Funding:	$5,472,690

PROJECT DESCRIPTION

Cost-effective separations are a key barrier in the scale-up of bioproduct technologies to industrially relevant volumes. Process simulation reduces the cost and risk of scale-up, but models available in current commercial simulators are limited in their ability to accurately predict bioproduct separations. This project will establish a framework for developing highly predictive process models applicable to multicomponent product streams to accelerate the development and commercialization of bioprocessing separations. The work will target the separation of organic acids from multicomponent mixtures, which is typically encountered in bioprocess operations. It will focus on two separation technologies: (1) adsorption and (2) membrane techniques. In both cases, lab-scale thermodynamic and physical property data will be collected, followed by the development of new thermodynamic models; these models will then be incorporated into commercial simulators and used in existing, complete process models. These tasks will be completed by experimental validation with pilot-scale data. The thermodynamic and process models developed in this project will consistently agree with experimental data (lab and pilot) with errors below ±20%. The developed methodologies will be applicable to the separation and recovery of organic acids from aqueous solutions, such as fermentation broth or lignin-rich streams.



COMMENTS

- The project is on track. There is a clear plan on DEI initiatives. This represents a great collaboration among a national lab, a university, and an industry partner. The team successfully collected adsorption lab data (isotherms, kinetics, and column breakthrough) and completed Milestone 2.1.1 on time. It would be great if the team could improve their capability to predict adsorption equilibria for three organic acid systems with prediction errors below ±10%.

- The team has a solid and multifaceted DEI plan. The team collected an impressive amount of adsorption data on three organic acids (and their binary and ternary mixtures) encountered in bioprocess separations. Adsorption thermodynamic modeling was started for single and binary mixtures. How many components will you need to include (and how many will you be able to include in the models) in order to get a useful thermodynamic model of a typical bio-separation? There is communication with simulation software partners, and good progress overall.

- This is an excellent approach to building a methodology that has broad applicability to a range of bioproduct separations and the potential to reduce development costs and cycle times across a range of process technologies. Significant progress has been made in both validating the approach for the work and generating useful data for a number of separations. The program is well managed, organized, and collaborative across the labs, industry, and academia. The risks are low, and the mitigation strategies are well thought out. I would consider placing additional priority on generating useful basic thermodynamic data for bioproducts in general to further support the industrial development of products like SAFs.

- I appreciate the team's work on building a prediction model to cut down the experimental space that a researcher here has to investigate. It is fantastic that the team is looking to make this work publicly available and is engaging with industry players to implement their models. I think that this work should be integrated with the wider BETO portfolio to see if there are synergies between this project and other efforts in the portfolio. I appreciate that the team can expand their model in the future if they have access to computational or experimental data on other targets.

PI RESPONSE TO REVIEWER COMMENTS

- We thank the review panel for their positive comments and constructive input. We are very excited about our initial success and the potential for this team to develop process models for bioprocess separations backed by multicomponent organic acid physical property data as well as thermodynamic data and models. DOE's support for a tightly integrated project between a university, a national laboratory, and an industry partner is greatly appreciated. Reviewer 2 asked a question about how many components need to be included in order to get a useful thermodynamic model of a typical bio-separation. To validate the thermodynamic model as a useful model for a typical bio-separation, up to three components need to be included. The model is being developed for multicomponent systems, and there is no upper limit on the number of components to be included in the models.

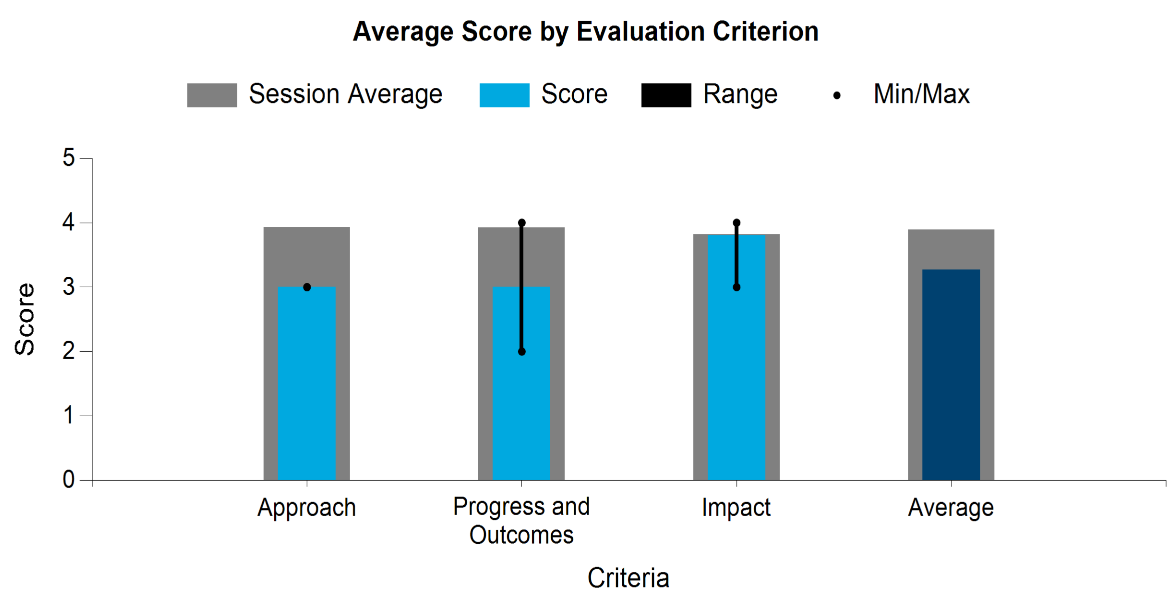
INVERSE BIOPOLYMER DESIGN THROUGH MACHINE LEARNING AND MOLECULAR SIMULATION

National Renewable Energy Laboratory

WBS:	2.5.1.500
Presenter(s):	Brandon Knott
Project Start Date:	10/01/2017
Planned Project End Date:	09/30/2023
Total Funding:	$400,000

PROJECT DESCRIPTION

This work aims to identify PABPs through computational property prediction. Given the exceptionally large biologically available design space, a major goal of this project is to guide the targeted synthesis of polymers and small-molecule materials toward those that have the highest likelihood of performance advantages. This is pursued in collaboration with the "Synthesis and Analysis of PABP" project. High-throughput property prediction enabled by ML and the elucidation of structure-function relationships enabled by molecular simulation facilitate a hypothesis-driven approach for the down-selection of candidate biomolecules to pursue experimentally. We have established bioproduct-relevant data sets, developed high-throughput polymer structure generation, and built end-to-end neural networks to predict various physical properties on the order of a million biopolymers. The ML tool developed here (PolyID) produces accurate property prediction (e.g., predicting glass transition temperature within 20°C) and >20 polyesters and polyamides synthesized by experimental partners further validate the model predictions. Molecular simulation tools (e.g., density functional theory) are also facilitating the discovery of the mechanisms of polymer formation and chemical recyclability. The computational tools actively developed within this project are increasingly being deployed via industry collaborations to industry-relevant problems.



COMMENTS

- There has been some progress in this review period. The project is on track. The milestones have been completed on time, and most milestones have resulted in peer-reviewed publications. The team used neural networks for polymer property prediction (glass transition (Tg), melting temperature (Tm), barrier, and moduli). Discovering suitable polymer materials with PABP for use in certain applications lies in accurately predicting the required properties. The team needs to talk to materials experts in industry to understand the real-life problems. Tg, Tm, barrier, and moduli are important, but these are basic properties and do not provide great information for the targeted applications. The team should predict environmental stress cracking (polycarbonate and polycarbonate/acrylonitrile butadiene styrene [ABS]), thermal conductivity (without fillers), flammability (without fillers), etc. The PI did not present their DEI strategy and progress. They should also extend their studies to the polymer blends property prediction.

- The integration of PickAxe and PolyID has the potential to create and down-select new polymers and small-molecule PABPs from bio sources. The approach will advance the SOA and help guide and narrow synthesis of PABPs. Predicting Tg alone will not predict structure-function relationships of the polymers synthesized, so it remains to be determined whether new polyhydroxyalkanoate (PHA) has other desired properties. Are there other properties you can select as outputs apart from Tg? It looks like there are very good collaborative partners/partnerships with a wide variety of industries. I really like the lower-toxicity plasticizer work, as there is great need for these types of materials. Replacing PET with a novel PHA is going to be difficult to accomplish industrially. Looking at bio-derived sources for PFAS replacements might be interesting, as many states are starting to regulate its use. Is there a DEI plan attached to this project?

- This review is based solely on viewing the slide deck. It appears that this project is much more narrowly conceived than (most) others. The information presented is dense and detailed, but it does not include a number of the evaluation bullet points. Slide 20—the overview quad chart—states that the project has two end-of-project milestones. The first is the computational method that is described. The second is to "predict solubility of PET in ≥25 novel lignin-derived performance-advantaged solvents and benchmark against experimental values." The project appears to be ending in 2023, and I did not see information on this second milestone. Slide 23, which is after the ending slides, *might* be related to this milestone, but it does not appear to have data on solvents. The design of slide 23 makes the information it contains very difficult to understand.

- This project takes an interesting approach to a complex computational problem by trying to use ML to predict the properties of new polymer systems, with the expectation that the development cycle will be sped up by reducing the amount of experimentation required to define polymer properties. There are potential flaws in this approach, as the available data from which to run the ML are very infrequently collected using the same parameters and are often not designed to be fed into large data sets. The risks of poor initial data quality and the potential negative impact on the calculations may not be appropriately mitigated. The team should also consider expanding the range of calculated properties beyond something like the glass transition temperature to more properties that would be useful for technical application, such as mechanical properties. The team should also consider expanding into mixtures; polymer blends are very common, and there could be significant utility for plastics recycling efforts, which frequently deal with mixed component streams.

- The team has mainly been predicting thermal properties for polymers like Tg and Tm. The goal of the project is predicting the solubility of PET in 25 novel performance-advantaged solvents. It is not clear to me how the presented work will reach that milestone before the end of the funding period. I appreciate that the team increased its efforts to partner with industry and presented on its ongoing work with Exxon, Patagonia, Algix, Tempur Sealy, and Kraft.

PI RESPONSE TO REVIEWER COMMENTS

- The project team thanks the reviewers for their time, attention, and thoughtful feedback. The suggestions to expand the number and type of properties predicted by PolyID align with our future plans. Although thermal properties are important for various manufacturing and performance applications, we acknowledge that many other properties are also critical for the industrial deployment of a new polymer. We plan to expand the number and type of properties by seeking out more data as well as developing correlations that may allow for the prediction of more specific, industrially important properties from those with more data available. Toward this goal, we plan to engage materials experts from industry to identify particular properties for application spaces (e.g., thermal conductivity) and polymer production (e.g., melt viscosity). We also appreciate the questions related to our DEI plan. We did not discuss our DEI efforts per BETO's guidance, as our project was established before the current BETO framework around DEI; however, this project is currently undergoing merit review for another 3-year cycle. Therefore, we are actively building these efforts into our plan for the next 3 years. These plans to address DEI include actionable goals for outreach to MSIs, which include recruitment and educational activities aimed at increasing the participation of underrepresented groups in STEM. We are also planning trainings for our current project staff and have ongoing efforts around internship programs that target MSIs and underrepresented groups. In our presentation, we did not delve into our progress on our end-of-project milestone around performance-advantaged solvents, as this is a relatively new effort (starting and stretching throughout the current fiscal year). Our target for performance-advantaged solvents was developed in response to the increasing demand from several experimental BETO and industry-funded projects for the computational screening of solvents for applications in polymer upcycling, separations, and more. In the current fiscal year, we have developed a workflow utilizing quantum mechanical and thermodynamic-based property prediction tools and are on track to convincingly meet this end-of-project milestone around bio-derived solvents for PET. The framework developed for these solubility calculations will be leveraged in future work for the development of performance-advantaged solvents for various applications with partner BETO projects. At the outset of this project, a significant risk was that insufficient data would be available to make sufficiently accurate property predictions to guide the synthesis of PABPs. As we discussed in our presentation, we have compiled a database of ~1,800 polymer chemistries with experimentally determined properties. This has been the basis for training our novel ML framework. We have quantified the error in model predictions (e.g., ~20°C mean absolute error for glass transition temperature), and our experimental PABP partner project synthesized 22 polyesters and polyamides that were experimentally determined to have thermal properties that fell within the prediction range. These mean absolute errors are nontrivial and are likely due to factors such as those noted by the reviewer (and others); however, the predictive capability of the PolyID tool has proven sufficient to be useful toward its end goal: targeting synthesis to more promising PABP candidates. As we move forward into the prediction of more specific, industry-driven properties, as described above, the property prediction errors will be continually quantified, and the sources of error will be determined within the framework of our database and novel domain of validity method. If specific functionalities or property predictions are found to be important to industry, and if they are lacking in our models, we will make concerted efforts to add them to the database to increase the domain of validity and decrease prediction errors.

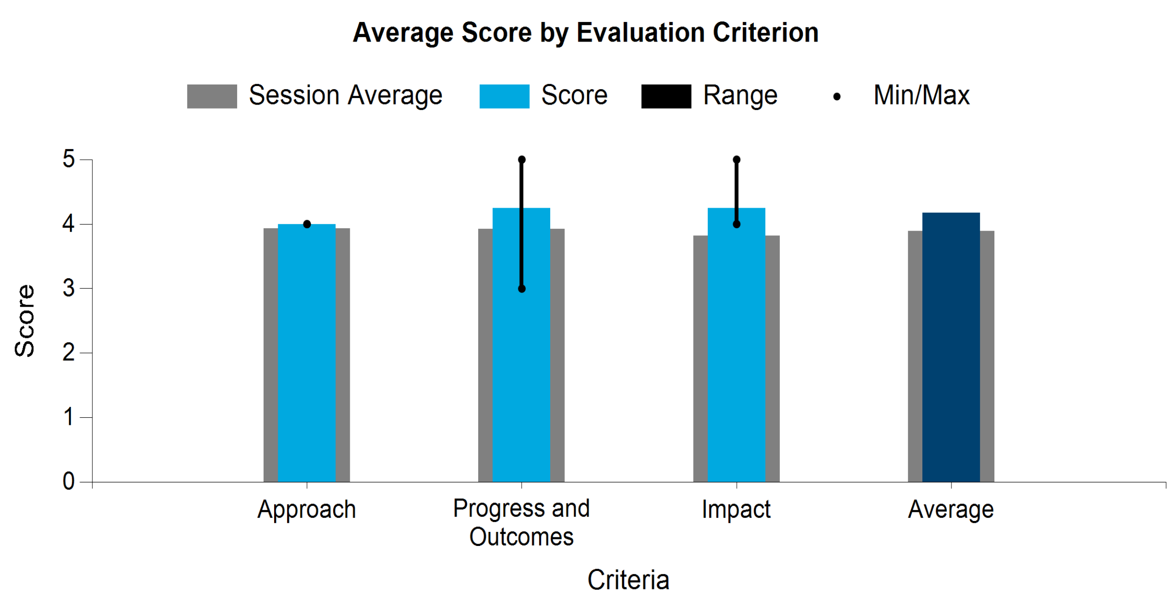
IDENTIFYING PERFORMANCE-ADVANTAGED BIOBASED CHEMICALS UTILIZING BIOPRIVILEGED MOLECULES

Iowa State University

PROJECT DESCRIPTION

WBS:	2.5.1.600
Presenter(s):	Brent Shanks
Project Start Date:	07/01/2020
Planned Project End Date:	09/30/2022
Total Funding:	$3,125,000

Given the lack of structure/function relationships between chemical structure and end-use performance, there is a need to develop a systematic strategy to identify performance-advantaged molecules. We explored the use of bioprivileged molecules as the basis for such a strategy. Two known bioprivileged molecules, triacetic acid lactone and muconic acid, were used as the basis of molecular libraries for the synthesis and screening of novel organic corrosion inhibitors and flame-retardant nylon polymers, respectively. Simultaneously, we worked on developing a computational framework to identify new bioprivileged molecules. We used literature data mining in conjunction with AI to identify promising chemical structures that could be used in conjunction with reaction network generation to suggest potential bioprivileged molecules. A goal of the project was to then use those new molecules to synthesize and screen additional molecules for use as corrosion inhibitors and flame-retardant moieties.



COMMENTS

- There has been great progress in this review period. The project is on track. The presenter gave an excellent presentation. Developing organic corrosion inhibitors and PFAS-free flame retardants for plastics using biomass-derived molecules with improved performance is really important and timely for the end-use applications. It is important to be able to address the flammability issue on the development stage and yet obtain information that pertains, in a well-defined way, to full-scale applications of interest, especially electric vehicles and consumer electronic applications. Although assessing the flammability of plastic materials through ASTM D2863-2013 is out of scope for this project due to the scalability of materials at this point, in future work, it would be great if the PI and team could compare the flammability of the biomass-derived molecules introduced to polyamide 66 (PA66) with limiting oxygen index and UL 94 VO testing to investigate the ignition time, burning time, and time taken to retard the fire. The team should also think about a patent application for their chemistry to reduce the melting temperature of PA66. A reduction in temperature will open the new space to compound natural fibers with PA66, which is extremely challenging due to natural fiber degradation, color change, and odor. The team should also use an artificial neural network-based system to predict ignition time, peak and total heat release, and mass residue after a burning reference and flame-retarded PA66 in a mass-loss-type cone calorimeter using small-scale thermal and flammability test results and structural properties. Flame-retardant PA66 applications are not common for the automotive and consumer electronic industry. Polycarbonate, polycarbonate/ABS, PP, and PET are the most common materials used in thermally challenging areas. The PI did not present their DEI strategy and progress.

- The researchers have identified very good targets for this project, especially in the realm of flame-retardant plastics. In automotive, there is a very urgent need for non-brominated flame retardants for new battery vehicles, so if a safer alternative can be developed from bio sources, it could have a large impact. The target polymers would be in nylon, but even more important is PP for battery covers (new designs) and ABS for electrical components. Brominated coatings are also applied to PET fabrics as a flame retardant, and safer alternatives are desired. The char results look promising, but adding V0 vertical burn tests would provide further confidence that the flame retardants could pass automotive tests. Because there are two parallel objectives—(1) synthesizing novel molecules with improved performance and (2) using data mining and AI to develop a systematic strategy for the identification of new bio-advantaged molecules—there has been significant progress and innovation. There should be engagement with industrial material suppliers from the transportation sector (nylon (BASF), PP (LyondellBasell), and ABS) to gauge their level of interest and to begin to anticipate the cost of synthesis of the molecules proposed.

- This is an innovative approach to identifying PABPs that could solve industrially important technical problems for corrosion inhibition and flame retardancy, both of which require environmentally difficult molecules to function. The computational approach is valuable and has the potential to reduce both the time to market and the cost of development for new bioproducts. This general approach is used broadly for pharmaceutical target molecules to good effect, and this model has been demonstrated here to have potential for industrial bioproduct applications. The future challenge for this approach is that the mechanisms for targeting need to be better elucidated to make this approach fully practical. A pharmaceutical model usually has a target site or metabolic pathway identified, and the retrosynthesis and ML can be tailored to more effectively meet the target. In this project, because the mechanisms for flame retardancy and corrosion inhibition are not well understood, the targeting efficiency is relatively low.

- I appreciate that the team is investigating two properties that are very highly sought after (corrosion resistance and fire resistance) and not very well understood on a structure-action level. The team is closely integrating with Linda Broadbelt's PickAxe effort. Although the team is struggling with cutting down the number of potential molecules to feed into the reaction network to synthesize, and with closing the loop of their discovery platform, the team's data mining efforts have already identified molecules with advantaged properties. I would suggest that the team collaborate with a synthesis-focused team to close the loop of the system. I would suggest that the team further explore industry connections to allow more people to identify bio-derived molecules with superior properties to SOA materials.

PI RESPONSE TO REVIEWER COMMENTS

- We appreciate the comments of the reviewers as well as their suggestions on how to further the work after the completion of the project. We have been pursuing outreach to industry, but the reviewers have provided some interesting additions to our efforts.

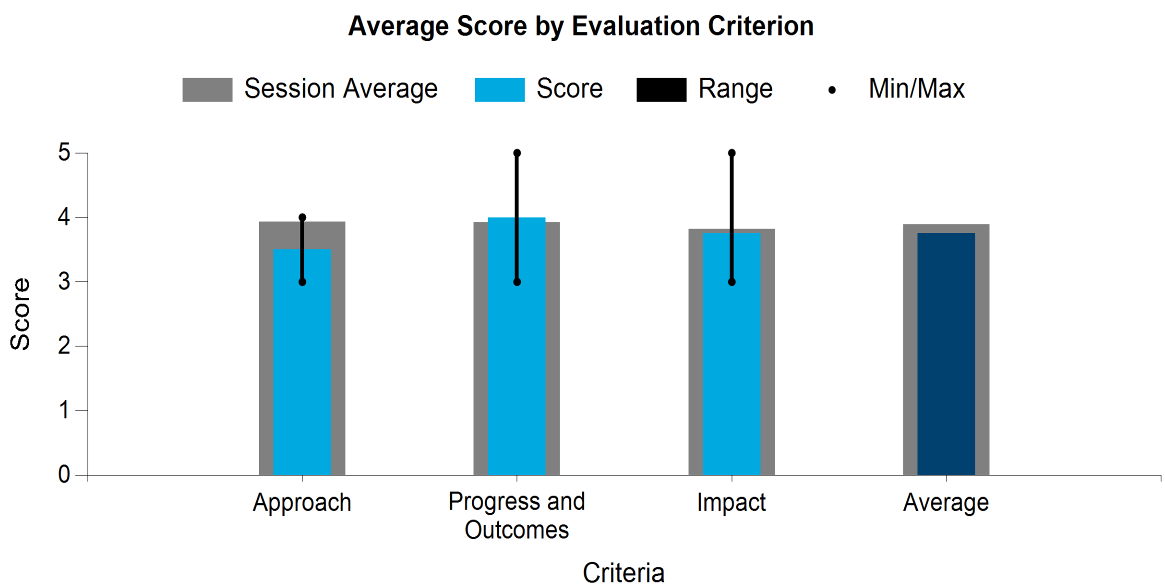
DEGRADABLE BIOCOMPOSITE THERMOPLASTIC POLYURETHANES

University of California San Diego

WBS:	AMO.01
Presenter(s):	Jon Pokorski
Project Start Date:	04/01/2021
Planned Project End Date:	03/31/2024
Total Funding:	$2,088,114

PROJECT DESCRIPTION

The goal of this project is to transform TPUs into a biodegradable composite material that incorporates viable bacterial spores. This would eliminate a significant plastic waste stream that is currently unrecyclable and undegradable and that generates ~1 million tons of waste annually in the United States. Additionally, we aim to improve the functional properties of the TPU, increasing tensile toughness by at least 20%. To do this, we are incorporating evolved bacterial spores into TPU biocomposite materials. The spores act as soft particles and, when well dispersed, will improve toughness. The evolution of the spores will serve two goals: (1) utilizing TPU as a sole carbon source to promote biodegradation upon spore germination and (2) improving tolerance to processing conditions (e.g., extrusion). By the end of the project, we aim to achieve the degradation goals set forth in the DOE BOTTLE FOA. The project aims to develop a simple method to impart biodegradation that will have minimal disruption in product formulation; simply, bacterial spores would be added during extrusion with little to no modification of the production method.



COMMENTS

- Because this presentation was not given, it is unclear what work has or has not been completed based on the information provided. Were “proper nutrients,” as described in the notes for slide 3, provided during the biodegradation shown on slide 20? What is the nutrient mix, and how likely is it that a piece of TPU would encounter that mix in a landfill or other environment?

- The concept is both elegant and novel. Taking a current commercial material and rendering it biodegradable via the addition of spores while also enhancing mechanical toughness is particularly attractive. The interaction between different project aspects is strong. The results show excellent progress in terms of improving the heat tolerance of spores, and that addition can improve the mechanical properties of TPU. From the degradation experiments, adding spores does not appear to increase the rate of degradation, but it does appear to change the microbial consortium. Focusing on melt processing is appropriate, as this is representative of what will need to be demonstrated for commercialization. Overall, the team is making excellent progress toward achieving the goals and is focusing on demonstration of the enhanced degradation with the addition of spores. To demonstrate substantial commercial potential, demonstrating that adding spores does indeed enhance degradation is necessary. Having BASF as an engaged partner on the project will help answer these questions and determine the viability of commercialization. If enhanced degradation can be demonstrated, further analysis to understand the degradation products is warranted. Interaction with other projects in the BETO portfolio focused on TPU degradation could have added value here. Are there any regulatory handling risks with a spore-based approach that would need to be assessed if commercialized?

- This early-stage project has made good progress in its evolutionary engineering work to improve the heat tolerance of a particular spore strain that could also use TPU as a sole carbon source. The touted attributes of a spore-embedded polyether urethane (submicron/soft/living/green) represent an interesting concept and could be compatible with applications where spores would not be foreign—e.g., in agriculture (seed coatings or soil nutrient supplements). I encourage the team to work with their industry partners to validate the market concepts proposed for this degradable TPU, to initiate LCA/TEA, and to determine whether there are regulatory hurdles in introducing a product with spores.

- The team did not present in person. Built-in degradation through spores is a creative approach to accelerating the degradation of plastics. Is the discoloration of the plastics due to the spores an issue for applications? How are you going to further test the viability of spores in the plastic over time? What are the targeted applications for these spore-containing plastics? The team should explore industry partnerships to test whether their composites remain viable under the conditions required for these applications.

PI RESPONSE TO REVIEWER COMMENTS

- We thank the Project Peer Review committee for the comments, and we apologize that we were unable to present due to illness.

- As far as we know, there should not be any regulatory hazards with the chosen spores. These are components of over-the-counter pharmaceutical products (probiotics) and are generally regarded as safe.

- The biodegradation studies were carried out in healthy, microbially rich compost. We have performed germination studies in compost extract to verify that the nutrients laden in compost were sufficient to germinate spores and yielded similar results as Luria Bertani media. More recent results indicate that the spores are able to germinate and facilitate degradation in otherwise sterile compost, indicating that composting conditions and the relative health of the compost play a minimal role in degradation.

- Thank you for the comment regarding additional applications. This type of composite material could also find use in agricultural films, and in that domain in general, and it is something we are actively exploring.

- Yes, the spores are responsible for the brown color of the plastics, which could be mitigated with dyes or additional components. Some intended applications are footwear, and the construction of such products could mask the physical appearance of the biocomposites. We are working with BASF to further validate the target applications. Last, we have stored spore-containing materials for more than a year, and they retain the ability for full germination.

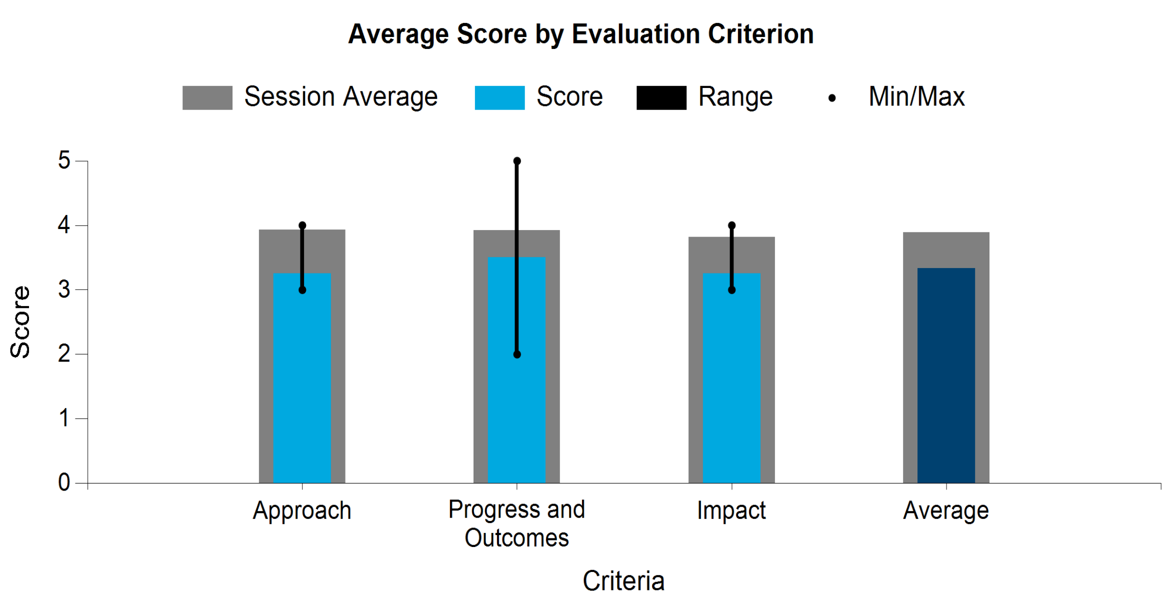
HIGHLY RECYCLABLE THERMOSETS FOR LIGHTWEIGHT COMPOSITES

University of Akron

PROJECT DESCRIPTION

WBS:	AM0.02
Presenter(s):	Junpeng Wang
Project Start Date:	04/01/2021
Planned Project End Date:	05/31/2024
Total Funding:	$2,049,242

The project focuses on the development of recyclable thermoset composites that can be produced from >50 wt % bio-based carbon sources (CO_2 and biomass). We aim to develop novel, highly recyclable thermoset materials, termed vitrimers, that combine the mechanical advantages of thermosets with the recyclability of thermoplastics. At service temperature, vitrimers are rigid like conventional thermoset polymers; at temperatures above the vitrimer transition temperature (Tv), vitrimers flow and behave as viscous liquid thermoplastic polymers. We will (1) prepare thermosets from CO_2-based materials, (2) demonstrate the mechanical recycling of thermosets, (3) demonstrate the chemical depolymerization of thermosets and carbon fiber recovery, and (4) model the cost and carbon footprints of circular composites. It is estimated that the manufacture of the next generation of recyclable CFRP will have huge market growth opportunities in aviation (commercial and military), transport, wind turbines, and compressed gas storage tanks for natural gas vehicles and fuel cell vehicles, among others. This highly collaborative project brings together expertise from the University of Akron, PNNL, and Raytheon Technologies Corporation. The project outcome will have both economic and environmental benefits in alignment with the interests of DOE's BETO and Advanced Manufacturing Office.



COMMENTS

- If I understand the goal(s) here, the timeline is very ambitious. Alas, I did not gain sufficient understanding from the presentation or the subsequent discussion to have a clear grasp on either the beginning (how the materials would be sourced) or the end (how the materials would come apart). I have questions about the economic viability, particularly on the end-of-(first)-life collection. These seem like key aspects if this is intended to be commercially viable.

- The approach here is innovative, targeting a new CO_2-based matrix material that allows recycling to recover the energy-intensive carbon fiber. If successful, it would address the carbon intensity and recycling challenges faced by current carbon fiber PP composites. The management and communication plan is clear. As lightweight, high-strength materials will play a role in decarbonizing transportation, it is important that the high-energy-intensity and recyclability challenges for these materials are addressed. EERE has substantial investment in this space via other offices, such as the Wind Energy Technologies Office and AMMTO—has this project leveraged that wider ecosystem for feedback? My largest concern with the approach is how the focus on unfilled vitrimer recycling and particle size sensitivity will be used to inform the final target, which is composite recycling. The integration between the LCA/TEA and the experimental work is very nice, as is the engagement of Raytheon as an industry partner. If the target will be an alternative to thermoplastic PP/carbon fiber (CF) composites, does the use of the vitrimer substantially change the EOL options, given that PP/CF composites could also be downcycled? The mixture of benchmarking between epoxy/CF and PP/CF should be resolved so the benchmarking of all properties is against a single reference. This project's main benefit resides in the use of CO_2 in the vitrimer synthesis to lower the matrix carbon footprint, as well as the potential for vitrimer systems to be chemically degraded, although that has yet to be demonstrated. Vitrimers have substantially different flow and creep characteristics relative to thermoplastic or thermoset matrix materials, making their introduction technically challenging. If you are not going to exploit the thermal reprocessability of vitrimers for recycling, I wonder if the challenges of introducing them as matrix materials are sufficiently offset if you are going to recover monomers via a depolymerization route. Strong industry engagement is necessary when developing a vision for a path to market at the end of the project. The substantial technical hurdles for introducing a new matrix into the composite space as well as new recycling routes raises doubts about the eventual commercialization of this technology.

- This is a very early-stage project with many works in progress. The team management and meeting structure describe regular task and teamwide meetings—do these include Raytheon and PNNL? It would be very valuable to see details on the TEA/LCA to better understand the expected improvements in carbon footprint, material reuse, and CF recyclability for composites in the automotive/aerospace industries. At this stage, it seems that technical success may be likely; however, at this stage, the team does not appear to have the deep industry engagement in place to have the desired impact and adoption.

- It is great that the team has already completed the LCA for the resin precursor, polycarbonate synthesis, and can use the learnings from the analysis to further develop their technology. In terms of reprocessing the vitrimer to retain mechanical properties, the team will have to further optimize their strategy. The proposed process of ball milling will not work on the carbon fiber-vitrimer composites. The team will have to develop an entirely different reprocessing technology for their composite products. Do you have additional strategies for reprocessing optimization?

PI RESPONSE TO REVIEWER COMMENTS

- Overall, the reviewers have identified several common threads that we plan to prioritize moving forward, and we thank them for their feedback. Now armed with BP2's experimental support, we feel confident in meeting an epoxy baseline. Regarding commercial engagement, we will also more outwardly engage with industry on the application of these materials as automotive composites and wind blades using our composite's performance. This includes engaging with DOE consortia and the National Science Foundation's Innovation Corps, as well as conducting more extensive TEA of the potential routes forward. The process and viability of material recovery was also raised several times. We hope to further develop the mechanical and chemical recycling in BP3 through solvent assistant and injection molded processes. Regarding Reviewer 1's question on opportunities for feedback, we aim to transition the project's goals to be more outward facing, now that material synthesis, characterization, and assessment have been established. To date, we have leveraged feedback from conferences, including the Society for the Advancement of Material and Process Engineering (SAMPE), the American Chemical Society, and the AMMTO REMADE Institute. We aim to solicit feedback from the Institute for Advanced Composites Manufacturing Innovation (IACMI) (DOE) and the American Composites Manufacturers Association in BP3. If there are additional mechanisms or organizations the reviewer would suggest we engage with, we would be grateful for any specific recommendations. We thank Reviewer 1 for their helpful comments regarding (1) recyclability, (2) benchmarking, and (3) commercialization. (1) In terms of recyclability, these materials do exhibit unique EOL options relative to PP, epoxy, and other vitrimers. Specifically, they have all EOL options available: they are mechanically, thermally, and chemically recyclable. Mechanical reprocessability of the vitrimer brings energy advantages relative to epoxy in prepreg storage (no refrigeration is needed, and shelf lives are longer) and in repair of defective parts. As the reviewer indicates, shredding of the composites and compression molding of the downcycled composite is not unique to the vitrimers and is comparable to PP composites. The additional comment regarding creep is an issue with vitrimers we are aware of, and are actively investigating in these materials. We are optimistic that the variation of malic acid/epoxide-CO_2 formulations will result in control over the ratio of dynamic:permanent cross-links and thus the degree of creep; however, this has not yet been demonstrated. Chemical depolymerization of the unfilled materials has since been demonstrated, and we are now investigating the CF filled materials. The TEA indicates that recovering the CF will be the primary economic driver, so our chemical depolymerization priority is the energy-efficient recovery of fibers and their surface integrity. (2) In terms of benchmarking, we aim to meet/exceed an epoxy benchmark. In the original proposal, we targeted an improvement in both the baseline mechanical performance and recyclability; however, exceeding epoxy mechanical properties (an extensively optimized industry standard) was presumptive for a new material, whereas comparison to its mechanical recyclability was moot. Now that our mechanical studies indicate we are within 10% of epoxy tensile strengths, we have analytical data supporting a uniform baseline: epoxy. (3) In terms of commercialization, addressing the technical challenges of new material adoption is an important part of our TEA. We have identified technical challenges with viscosity and cure time, which we continue to develop solutions to, and we anticipate that this will require specific tasks in BP3 to be revised. We also aim to centralize processing changes in the supply chain (i.e., prepreg manufacturing will be considerably altered as a new business, but composite production will not). Regarding recycling commercialization, our TEA and LCA indicate that the highest impact is in the monomer and precursor synthesis. As such, we propose that the precursor manufacturer will be the party that benefits the most from material recovery and recycling. We thank Reviewer 2 for their comment, and we hope our written response provides more clarity. The TEA pricing is based on quotes from suppliers who would serve as material sources, specifically of epoxide/CO_2 polycarbonates (Aramco and Covestro), succinic anhydride (Tate & Lyle), epoxide (Envisions LLC), and CF (Profabrics). All capital equipment and solvents are accounted for. We also agree that the material recovery supply chain and the economic viability of recovery is important and a challenge. We will evaluate whether a precursor synthesis business, prepreg manufacturer, or composite company will best serve as the recipient of secondary feedstocks. This depends on the impacts and costs of resin, CF, and melt-recycling, respectively. We agree with Reviewer 3 and feel that significant progress has been made since the last Project Peer Review. First, yes, Raytheon and PNNL do join our regular meetings, and their participants bring both researcher-level detail and commercial perspectives to the results generated. The TEA has been thoroughly conducted on the current composite manufacturing process and shows that a positive return on investment can be achieved with a reasonable capital investment. The initial cost of the resin is certainly higher than our envisioned target, but we believe this can be significantly improved through our other formulations. Understanding the trade-offs between performance and these TEAs/LCAs for our target composite applications is necessary. As a result, we also agree that deeper industry engagement is needed to meet the performance metrics of the composites (within 10% of epoxy strengths) while maximizing our TEA/LCA viability. Reviewer 4's comment is very helpful, and we have also reached the same conclusion regarding the reprocessing methods. We plan to focus BP3 on additional processing methods, both of the virgin materials and the reprocessing. Specifically, the TEA of prepreg manufacturing would benefit considerably from injection molding of the vitrimer, which would also translate into chopped secondary composites. Solvent-based methods have also been investigated for reducing the viscosity of the composites during virgin synthesis, and they also improve the depolymerization dispersion. We have found that acetone and propylene carbonate both work well, which we find particularly intriguing as cyclic carbonate is the product of depolymerization.

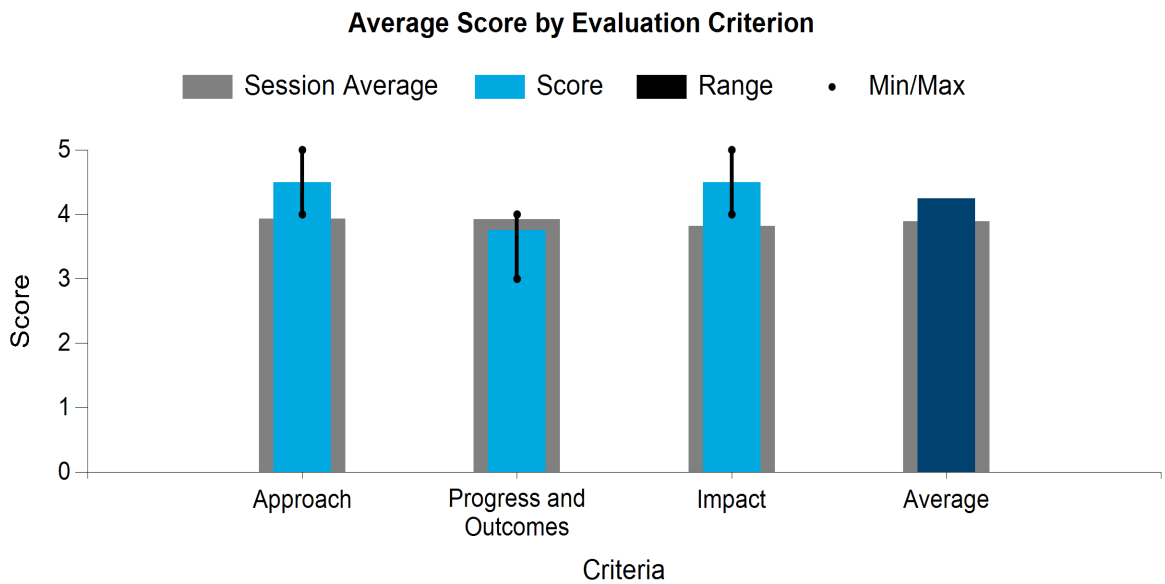
MODULAR CATALYTIC REACTORS FOR SINGLE-USE POLYOLEFIN CONVERSION TO LUBRICATING OILS FROM UPCYCLED PLASTICS (LOUPS)

Iowa State University

PROJECT DESCRIPTION

WBS:	AM0.03
Presenter(s):	Aaron Sadow
Project Start Date:	05/01/2021
Planned Project End Date:	04/30/2024
Total Funding:	$2,500,000

More than 150 million tons of the polyolefin plastics (POs) manufactured each year become waste in landfills and the environment. This PO waste contains the equivalent energy content of ~17% of domestic annual oil consumption. Our team is developing catalytic hydrogenolysis of waste POs into oils for application as lubricants, investigating their properties, and analyzing the economic and environmental implications of this manufacturing route. The project is developing efficient, economic, scalable catalyst preparation and catalytic conversions of POs into LOUPs; studying the tribological properties of the LOUPs; and identifying economic and environmental impacts of this route compared to conventional plastic waste processing and lubricating oil production. Challenges include synthesizing a catalyst that produces comparable or improved reactivity and selectivity compared to that obtained from the small-scale preparation, producing and separating LOUPs from catalyst and residue, and creating molecular structures that lead to high-performance lubricating properties that exceed the optimized conventional manufacturing processes. We have succeeded in finding scalable routes for both catalyst synthesis and LOUP preparation, and TEA and LCA have identified current pressure points that can reduce costs and GHG emissions. LOUP samples show a synergistic improvement in wear scar upon blending and with additives.



COMMENTS

- The approach targets polyolefin waste to lubricant oils, which has the potential to both address the plastic waste issue and reduce the demand for virgin oil for lubricant production. This represents a substantial innovation in that market. This project is a continuation of a Basic Energy Sciences-funded project. The approach is sound, with a clear communication plan between participating entities. The team has been making good progress toward scaling up to larger reactors and expanding to different olefin feedstocks. The parallel LCA/TEA work, along with the experimental activities, is very good, as is the focus on identifying scalable production routes for the catalyst. If the improved lubricant performance remains with real waste feedstocks and commercial additive packages, this would bring an additional technical advantage over just the reduced footprint. A startup company has been spun out from the research effort, increasing the commercialization potential. The clear focus on end application needs, early evaluation to ensure compatibility with additive packages, and engagement with players along the value chain are real strengths of the project, as is the integrated, multidisciplinary project team. Evaluating real PCR waste olefins should be a focus as the project moves toward larger scales to ensure sufficient robustness to potential contaminants and how catalyst lifetimes impact the TEA. There are other efforts targeting polyolefin waste to lubricants—has the team mapped out how their approach compares to other efforts in this space?

- The visual nature of the slides was very helpful in conveying the information. The presentation was fairly thorough in defining terms, which helped me focus on the narrative of the work rather than double-checking units or abbreviations to make sure I understood. If I understand correctly, the process identified so far will work on pre-consumer waste streams where the contents of the materials are predictable, but the catalyst economics will not work for post-consumer waste unless collection is very careful/controlled to produce a consistent stream. I think that doesn't quite match the remit.

- This is a very compelling proposal to convert and upgrade the annual polyolefin waste cost ($60 billion) into a profit of $160 billion for lubricants. The team is leveraging outcomes from a companion Energy Frontier Research Center project built to identify the catalyst $Pt/SrTiO_3$, and it has performed early TEA/LCA with defined boundaries—understanding major GHG contributors (solvents) and developing a risk mitigation strategy. They employ a moderate reaction condition (low pressure, modest temperatures).

- I am very impressed with the progress that the team has made during the funding period. The team is taking the whole system into account when optimizing the TEA/LCA; for example, they are using the reactive heat for process separation. The team has a focus on industry needs and has tested the compatibility of their lubricants with the most commonly used additives. This work has already resulted in a spinoff company called Aeternal that is pursuing the commercialization of polyolefin upcycling to lubricants. Great work!

PI RESPONSE TO REVIEWER COMMENTS

- Reviewer 1 excerpt: Evaluating real PCR waste olefins should be a focus as the project moves toward larger scales to ensure sufficient robustness to potential contaminants and how catalyst lifetimes impact the TEA. There are other efforts targeting polyolefin waste to lubricants—has the team mapped out how their approach compares to other efforts in this space?

- Response: Thanks for the comments. During our last budget period, we are focusing on polyolefin plastic waste from our commercial partners as providers. The primary approach for the chemical upcycling of polyolefin plastic waste involves pyrolysis, which is currently performed at the large pilot scale by a few commercial entities. The most important technical distinction between our process and pyrolysis is the much higher selectivity afforded by low-temperature chemical catalysis.

- Reviewer 2 excerpt: If I understand correctly, the process identified so far will work on pre-consumer waste streams where the contents of the materials are predictable, but the catalyst economics will not work for post-consumer waste unless collection is very careful/controlled to produce a consistent stream. I think that doesn't quite match the remit.

- Response: We appreciate the comment on the clarity of our presentation. Most of our process has focused on polyolefin wastes, currently from post-industrial providers. These are mixed (PEs and PP as films or laminates with adhesives, polyvinyl alcohols, colors, stabilizers, etc.). This waste is high volume, and even though it is not yet consumer-contaminated, it should not be discounted as an easier conversion. Nonetheless, our process has been shown, in some cases, to tolerate additional contaminants. We also think that any sustainable plastic waste management system will require some control along the way—that is, we should not manage all waste as single stream and then separate it, as point-of-source separations can be effective and add efficiency to the life cycle of carbon-based materials.
- Response to additional reviewer comment: We appreciate all the feedback and your time and enthusiasm for this review. Many thanks!

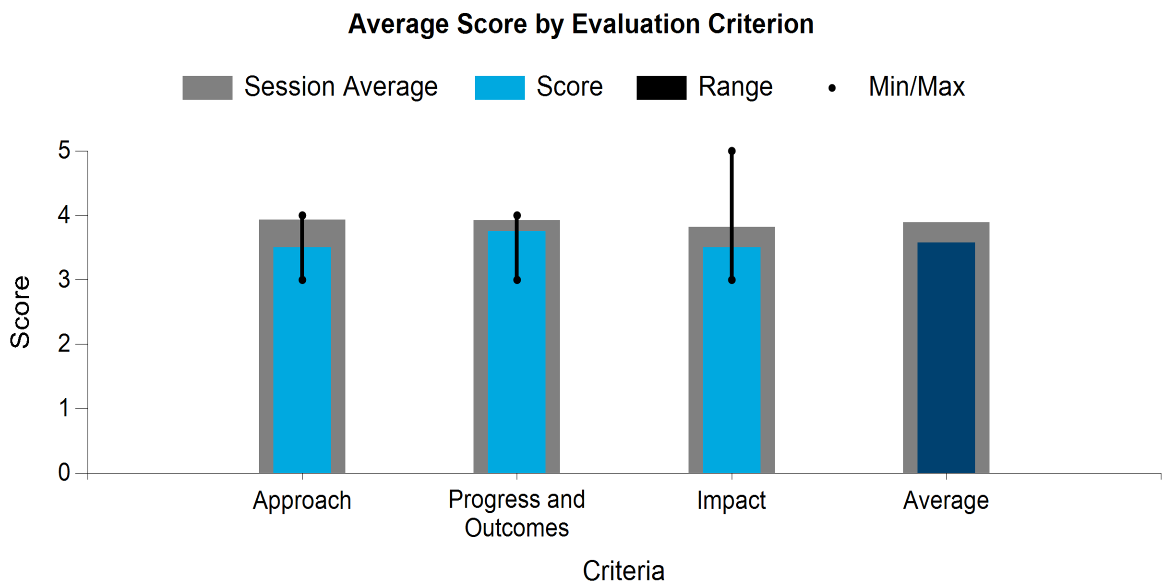
HYBRID CHEMICAL-MECHANICAL SEPARATION AND UPCYCLING OF MIXED PLASTIC WASTE

Case Western University

PROJECT DESCRIPTION

WBS:	AM0.04
Presenter(s):	Joao Maia; Mike Hore
Project Start Date:	06/01/2021
Planned Project End Date:	11/30/2024
Total Funding:	$2,498,539

The main objective of this project is to develop a hybrid mechanical-chemical recycling technology for multilayered and laminated plastics. In particular, we aim to separate and upcycle more than 80% of the two main constituents of such structures, polyolefins and polyesters, for a significantly lower cost and at higher energetic efficiency and much larger throughputs than chemical recycling while maintaining the low-cost and high-volume advantage of mechanical recycling.



COMMENTS

- Please double-check that titles are used equitably and appropriately for all researchers. It was unclear whether that was an artifact of the presenter or the project itself. Accounting for fillers or additives in the materials is one of the core difficulties of working in this space, and it would be reassuring if the presenter/project discussed the risks and mitigations of that aspect directly.

- Leveraging continuous twin screw extrusion combined with the chemical recycling of mixed plastic waste is a very high-risk, high-reward project. If successful, the continuous process and reduced capital costs would increase commercial potential. The technical hurdles are substantial, but the project plan is structured in a way to handle them methodically. The multiscale modeling approach is elegant. The accuracy of predictions must be validated against the experimental results. The project is making good progress along the plan. A key risk that is insufficiently addressed is that real feedstocks will contain contaminants in addition to PET/polyolefins, and these may substantially alter the phase separation behavior or poison the catalysts. The lack of focus on this aspect early in the project is a weakness, and the team should work to include a plan to evaluate these real waste streams earlier in the project, perhaps exploring small-scale mixing experiments while the twin screw extrusion process is being stood up. The team needs to engage with their industry partners to better understand the potential composition of incoming material. Given that gaseous products are proposed to be produced during extrusion, the project team must ensure that the safety aspects of the process are proactively addressed.

- This project has promise because it is agnostic to feedstock, and the capital expenditure projects are 25% of that of SOA pyrolysis. I am intrigued by the hybrid nature of the approach but also by the fact that the substrate is mixed (polyester and polyolefins). I also like the great versatility of this project if it is successful: It may be possible to do drop-in operations and colocate at recycling facilities. The repeated graphic with the split out of the tasks relative to the two feed streams (PET and polyolefins) is very useful—the charts have an adequate description of the tasks and risks; however, the inclusion of a single chart detailing the milestones, tasks, and critical decision points against the projected timeline (in quarters over the budget periods) would be helpful.
- The team is working with model feedstock; if their process is agnostic to feedstock, how can they prove that? The team should focus on proving that their system can work with varied real-world waste streams and sourcing real-life feedstock from local partners. It is great that all three of their industry partners have expressed interest in taking this technology to market.

UPSCALING OF NON-RECYCLABLE PLASTIC WASTE INTO CARBONSMART MONOMERS

LanzaTech

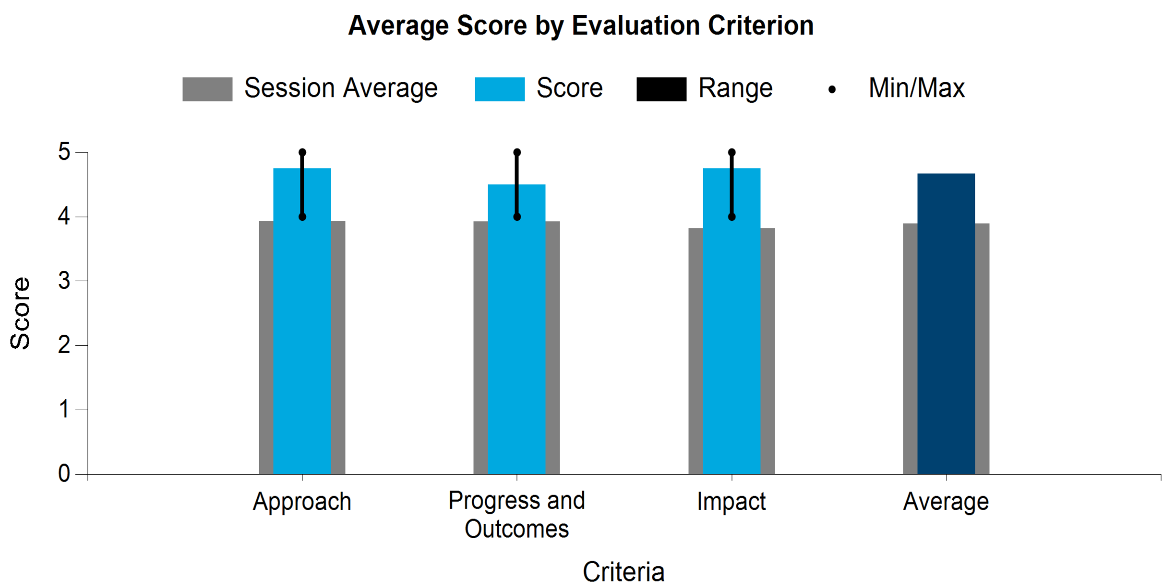
WBS:	AM0.05
Presenter(s):	Sean Simpson; Ching Leang
Project Start Date:	11/01/2021
Planned Project End Date:	03/31/2023
Total Funding:	$1,890,001

PROJECT DESCRIPTION

The project is developing a novel microbial process to produce monoethylene glycol (MEG) directly from nonrecyclable, mixed waste plastic at high yields and efficiencies. MEG is important in the production of PET, a plastic polymer used in common, single-use consumer goods, such as drink bottles, food packaging, and polyester fiber in clothing. Mixed and contaminated waste plastics represent a significant percentage of potentially recyclable MSW residues that are currently landfilled due to a lack of techno-economically viable processes for material recycling/upcycling. LanzaTech is developing technologies to convert these waste plastic residues to MEG, an important chemical in the production of PET. MEG produced from nonrecyclable plastic wastes offers a sustainable, cost-effective alternative to fossil-derived MEG to meet the needs of end users in the chemical and consumer products industries who are seeking to de-fossilize.

Our technical accomplishments are as follows:

- Multipronged engineering approaches have successfully identified pathway bottlenecks and improved MEG production 400 times.
- Enzyme variants were identified via *in vivo* assays.
- Intermediate feeding experiments identified bottlenecks for MEG production.
- InEnTec demonstrated fermentable syngas from multiple MSW feedstocks.



COMMENTS

- The slides were both comprehensive and well laid out. The researchers are skilled, and the microbial work is impressive. If the location of the system boundary for the TEA is the same as the location of the system boundary for the LCA, then neither is sufficient for understanding the way that this would work (or not) in practice. I would recommend clarity on whether metal ingots and vitrified glass slag have market value. The presentation indicated they do, but as those are not as common in plastics analysis, more specifics on that would be useful. The term “recovery” is undefined in this context, at least regulatorily, and can cover a wide range of actions—sort of like “natural” foods.

- This is a well-conceptualized and well-executed project with partnerships along the supply chain (waste collection, gasification, and conversion to MEG). The screening approach appears to be robust, leveraging modeling and high-throughput experimentation. Utilizing both cell-free and *in vivo* screening allowed the project to progress despite the cell-free route proving inconclusive. The project is progressing on time and making appropriate progress to meet the milestones established. The team has successfully increased MEG production 400 times using a combination of approaches that identified different bottlenecks. This is particularly impressive because there is not natural MEG production in microbes. Because the work has proved successful, widening the view to MEG uses is warranted to evaluate the suitability of MEG produced for different end uses (purity, separations needed, etc.). For the LCA, comparing not only cradle to gate for MEG production but also other PET recycling options (e.g., PCR waste -> PET production) would be valuable for understanding the potential of this technology in the PET recycling ecosystem. The potential impact of this technology is high, as bio-MEG can be introduced into the existing supply chain relatively easily.

- This is a very focused project that addressed each key area: management, approach, and outcomes. The team chose a very experienced gasification partner, InEnTec, to provide the substrate or feedstock for their microbial fermentation process to produce MEG. The milestone chart was very clean and clear. The team took a multipronged approach to overcoming the challenges in identifying stable enzyme variants. The increase in MEG titer (500 times) by pathway engineering and enzyme screening is a tremendous achievement in a short time. It would be helpful to know where the current production is relative to MEG rate/titer/yield metrics (not specified in the presentation) that would make the bioprocess economically viable.

- The team is working with industry partners in both waste management and gasification as well as consumer brands. I greatly appreciate the team’s effort to engage players on both ends of the supply chain. I appreciate that the team is dedicating a whole team to TEA/LCA to inform their decision making in developing their process. This project is a good example of demonstrating a pathway to TRL 7 while starting with a very early technology by leveraging the know-how of industry partners.

PI RESPONSE TO REVIEWER COMMENTS

- Note: Subject to disclaimers on the associated presentation.

- Comment: This is a well-conceptualized and well-executed project with partnerships along the supply chain (waste collection, gasification, and conversion to MEG). The screening approach appears to be robust, leveraging modeling and high-throughput experimentation. Utilizing both cell-free and *in vivo* screening allowed the project to progress despite the cell-free route proving inconclusive. The project is progressing on time and making appropriate progress to meet the milestones established. The team has successfully increased MEG production 400 times using a combination of approaches that identified different bottlenecks. This is particularly impressive because there is not natural MEG production in microbes. Because the work has proved successful, widening the view to MEG uses is warranted to evaluate the suitability of MEG produced for different end uses (purity, separations needed, etc.). For the LCA, comparing not only cradle to gate for MEG production but also other PET recycling options (e.g., PCR waste -> PET production) would be valuable for understanding the potential of this technology in the PET recycling ecosystem. The potential impact of this technology is high, as bio-MEG can be introduced into the existing supply chain relatively easily.

- Response: We are pleased by the enthusiasm the reviewer expressed about the potential impact of MEG produced using our biocatalyst approach. We are targeting the production of MEG that meets the specifications for PET production, which is one of the highest grades of MEG used in manufacturing and has the largest market. LanzaTech has significant expertise in this area, predicated on the company's experience producing MEG "indirectly" through an ethanol intermediate. In those instances, the MEG produced has been used as a chemical feedstock for polyester textiles that have been used to make consumer apparel. MEG produced directly from gases will be suitable for numerous downstream applications. The LanzaTech vision goes beyond PET recycling/depolymerization. The LanzaTech process represents the opportunity to valorize municipal waste streams containing unsorted, nonrecyclable plastics and other materials. It is estimated that only ~5% of plastic waste is recycled in the United States, primarily due to costs associated with material separation. The LanzaTech process is unencumbered by heterogeneity and offers a means to recover and recycle carbon from plastic waste at much higher rates than offered by pathways that require highly pure (i.e., PET) plastics.
- Comment: The slides were both comprehensive and well laid out. The researchers are skilled, and the microbial work is impressive. If the location of the system boundary for the TEA is the same as the location of the system boundary for the LCA, then neither is sufficient for understanding the way that this would work (or not) in practice. I would recommend clarity on whether metal ingots and vitrified glass slag have market value. The presentation indicated they do, but as those are not as common in plastics analysis, more specifics on that would be useful. The term "recovery" is undefined in this context, at least regulatorily, and can cover a wide range of actions—sort of like "natural" foods.
- Response: LanzaTech appreciates the reviewer's comment. LanzaTech uses TEA and LCA as a tool to quantify the impact of various performance metrics to ensure that the project outcomes will be commercially relevant. In this instance, TEA and LCA are not used for assessing a business case for the technology and process; however, the outcomes of the LanzaTech project are anticipated to inform the development of multiple business case scenarios, including those that may have additional value from multiple products (i.e., vitrified glass slag, ethanol, etc.). This effort will be executed downstream of the DOE project. "Recovery" in this instance specifically refers to carbon recovery. High rates of carbon recovery will reduce the amount of virgin fossil resources required to address the global demand for plastics, which at this time continues to increase.
- Comment: This is a very focused project that addressed each key area: management, approach, and outcomes. The team chose a very experienced gasification partner, InEnTec, to provide the substrate or feedstock for their microbial fermentation process to produce MEG. The milestone chart was very clean and clear. The team took a multipronged approach to overcoming the challenges in identifying stable enzyme variants. The increase in MEG titer (500 times) by pathway engineering and enzyme screening is a tremendous achievement. It would be helpful to know where the current production is relative to MEG rate/titer/yield metrics (not specified in the presentation) that would make the bioprocess economically viable.
- Response: We are grateful for the reviewer's satisfaction with the gasification partner and with the project approach and management overall. We are also grateful to receive the reviewer's acknowledgement of the significant titer achievement. The titer and selectivity-based targets set out in our proposal were based on our preliminary TEA supporting their relevance to commercialization. Because this project is still in the early stages, and we are focusing on identifying and implementing optimal pathways to produce MEG in continuous stirred-tank reactors (CSTRs), we have not begun reporting rate and yield metrics; however, in Year 3 of the project, we will begin pilot-scale

fermentations using our best MEG-producing strains. Pilot-scale performance is a much more accurate representation of strain performance compared to CSTRs. We intend to use rate and yield metrics more when we advance to optimization in pilot-scale fermentation architecture, and we agree with the reviewer's assessment that these will be critical to evaluating the economic viability of the process.

- Comment: The team is working with industry partners in both waste management and gasification as well as consumer brands. I greatly appreciate the team's effort to engage players on both ends of the supply chain. I appreciate that the team is dedicating a whole team to TEA/LCA to inform their decision making in developing their process. This project is a good example of demonstrating a pathway to TRL 7 while starting with a very early technology by leveraging the know-how of industry partners.
- Response: We thank the reviewer for these comments, and we appreciate that we agree about the importance of engaging partners that are involved in various aspects of the commercial process. LanzaTech continues to work directly with customers to ensure that the products made from plastic waste will meet all necessary specifications for varied applications in packaging and fibers.

CIRCULAR ECONOMY OF COMPOSITES ENABLED BY TUFF TECHNOLOGY

University of Delaware

WBS:	AM0.06
Presenter(s):	Joseph Deitzel
Project Start Date:	07/01/2021
Planned Project End Date:	12/31/2024
Total Funding:	$2,499,983

PROJECT DESCRIPTION

Carbon fiber composites (CFCs) exhibit superior properties and, combined with part consolidation, significantly reduce system weight compared to metal approaches. CFCs are utilized in lightweight structures in automotive, aerospace, wind, infrastructure, and many other applications. Growth rates are significant (7% in the United States alone) and are driven by automated processing approaches, lower-cost CF materials, and (in the case of automotive applications) the need to meet government regulation. Raw material costs of continuous CF can range from $20 per kilogram for low-modulus and high-filament-count carbon tow materials (Zoltek, DowAksa) to more than $200 per kilogram for ultrahigh-modulus CFs. In addition, CFCs have high embodied energy (~230 megajoules/kilogram), resulting in a significant energy burden during virgin fiber production. Recycling CFCs has the potential to recapture the material value at a much lower cost, reduce the embodied energy of the CFCs, and provide a pathway to reducing waste. Nevertheless, CFC recycling is in its infancy as an industry in the United States, with the key challenges being (1) the ability to recover both the fiber and polymer content and (2) conversion of the recycled material into high-value CFC without significant property loss, reducing the original embodied energy and cost.

Existing recycling strategies include thermolysis, pyrolysis, and solvolysis, and these have yet to demonstrate a viable process capable of simultaneously producing high-quality fibers while recovering useful chemical functionalities from extracted epoxy resin. The diversity of epoxy resins used in CFCs has led to a variety of new catalytic approaches to depolymerize these binding resins using milder conditions, which would allow for the economic degradation of epoxy resin and the recovery of intact CF. Existing catalytic methods in the academic literature for chemical depolymerization systems generally employ either oxidation or Lewis-acid-catalyzed hydrolysis; however, in all cases, no publicly available sources have attempted to build a TEA-driven process approach to target key limiting factors in transitioning this technology to a continuous unit operation.

Another challenge of the existing recycling technology is the breakdown of the continuous fiber material into recycled, discontinuous chopped or short fiber random and semi-aligned forms, which results in reduced mechanical properties of fabricated composite structures. For example, the tensile strength of Massachusetts Institute of Technology's DEP recycled preforms (156 million pascals with fiber length distribution between 6 and 25 millimeters) molded with polyester is ~20% compared to an equivalent IM7 fiber quasi-isotropic prepreg composite. This is mainly due to the random fiber orientation and low fiber loading of the preform and reduced fiber and adhesion properties of the recycled fibers.

The University of Delaware Center for Composite Materials (UD-CCM) will team up with members of the BOTTLE Consortium, including NREL and Colorado State University (CSU), to address these challenges and develop and demonstrate a novel CFC recycling process. NREL and CSU will develop the fiber/polymer separation and depolymerization process, and UD-CCM will demonstrate CFC processing of the recycled discontinuous fiber content using the Tailorable Universal Feedstock for Forming (TuFF) process, allowing full property translation. The patented (U.S. 10,669,659) fiber alignment process has been developed under a recent 4-year Defense Advanced Research Projects Agency (DARPA) program led by UD-CCM and scaled in a pilot facility at UD-CCM (10,000 pounds/year, 18 inches wide) that converts short fibers to high-performance parts in one facility. The innovation has been selected by the American Composites

Manufacturers Association as the 2019 winner for the "Infinite Possibility for Market Growth" Award for Composites Excellence for its potential for greatest impact to new and emerging markets.

The objectives of this program are to combine (1) an innovative short fiber alignment process developed by UD-CCM with (2) a new recycling approach to separate fiber and polymer content of CFCs innovated by NREL and (3) a novel recyclable polymer material (CSU) to (4) demonstrate the closed-loop recycling of high-performance CFC to implement a sustainable CFC manufacturing approach for the first time. The program will demonstrate the feasibility of manufacturing highly aligned short fiber composites at our industry partner sites with high fiber volume fraction (>50%) and mechanical properties that are two- to threefold higher than current recycling methods. If successful, the proposed approach will allow true composite recycling with associated energy reduction (75%) and carbon utilization (>85%) over multiple component life cycles without significant loss of performance.

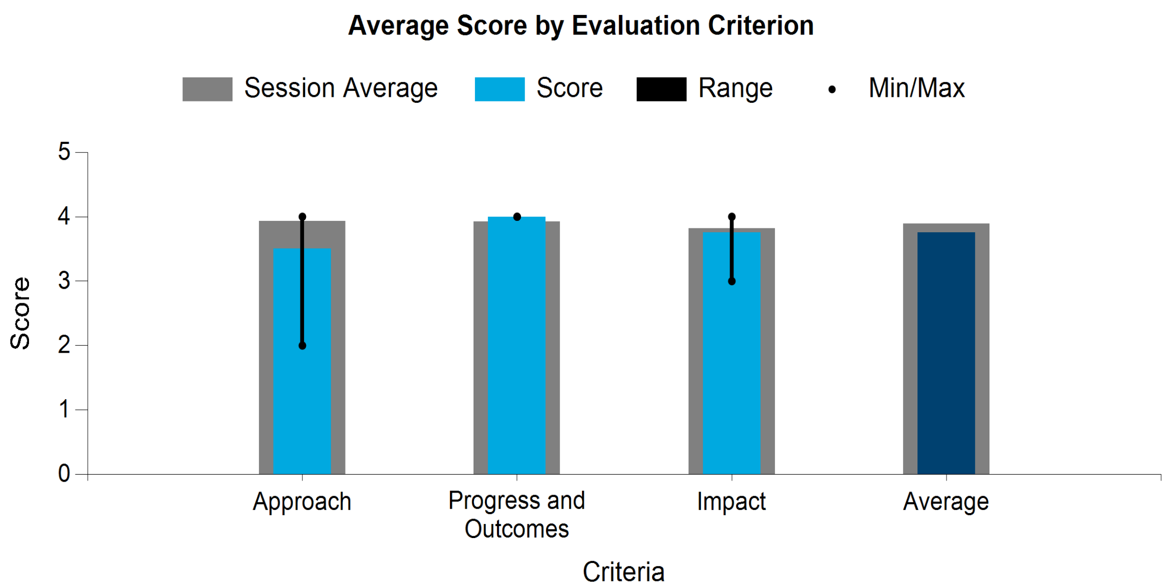
By combining the unique talents of NREL in catalysis, process engineering, solids handling, and TEA and LCA with the University of Delaware's expertise in short fiber aligning technology, the polymer engineering breadth of CSU, and the expertise of our industry partners, this proposal will provide a unique opportunity to tackle the grand challenge of CFC recycling and will meet/exceed the stretch goals of this FOA (70% energy savings and 85% carbon utilization). We will engage our industry members (Arkema, Axiom, Composites Automation) to provide waste CFC, support material production, evaluate our recycling process, and transition opportunities to commercial material forms and applications.

Accomplishments in the program to date include:

- Established baseline epoxy/T700 composite properties
- Conducted initial demonstration of single-iteration recycling for vacuum-assisted resin transfer molding process processed Elium/T700 part achieving ~100% translation of tensile modulus and 64% translation of tensile strength in the recycled part compared to the virgin T700/Elium TuFF composite
- Measured baseline interfacial shear strength for Elium thermoplastic and T700 with Elium compatible FOE sizing
- Successfully fabricated high fiber volume fraction (~50%) Elium T700 TuFF panels
- Successfully fabricated, for the first time, Epoxy/TuFF prepreg materials using commercial equipment (Axiom)
- Translated conventional heating data to sand bath reactors for thermoplastic recycling reactions. Ordered new reactors to further this preliminary work with cross-linked thermosets. These reactors arrived in January 2023, so they will be used to test higher-temperature reactions for thermosets in Quarter 2 of FY 2023.
- Finished optimizing thermoset swelling conditions in both acetic acid and N-Cyclohexyl-2-pyrrolidone as solvent
- Characterized two cross-linked epoxies received from Axiom and generated two partially cross-linked thermosets to mimic the thermal properties of these commercial materials
- Based on the results of the model epoxy compound studies, we have down-selected to a base-mediated epoxy recycling scheme as the focus for future work.
- Evaluated monoethanolamine and methylene valerolactone (MVL) monomers as candidates for recyclable resins. Monoethanolamine was a key candidate at first but suffered from low polymerizability at room temperature and exhibited low Tg. MVL was selected to address the polymerizability. Upon

further study, poly-MVL showed enhanced thermal properties and recyclability (~99% monomer recovery for 1-gram samples).

- Adjusted the initially reported small-scale synthetic pathway for MVL based on the initial TEA to bring sustainability and economic feasibility to the forefront of the large-scale synthesis (>100-gram quantities)
- Shipped 10 grams of poly-MVL to the University of Delaware for initial studies and will soon send 30 grams of MVL monomer for further studies
- Began TEA of the large-scale synthesis, polymerization, and recycling (currently in progress via collaborators at NREL).



COMMENTS

- This is a highly ambitious project that addresses multiple sustainability challenges for CF composites: (1) EOL recovery of fibers from current epoxy/CF composites, (2) demonstration that the TuFF process can use recycled fibers, (3) development of recyclable matrix materials with lower carbon footprints, and (4) demonstration of monomer recovery from new matrix materials. This vision would allow the recovery and reuse of high-embedded-energy CF from current materials and transform them into more circular composite systems for extended material lifetime of the CF components. Given the highly ambitious nature of the project, the approach is logical, with risks mitigated by a layered approach. The communication within the project and to the wider community was not explained. Is there an advantage to connecting with ongoing activities via IACMI, for example? Despite the delay with starting due to subcontracting, the project appears to be making good progress toward the defined milestones and go/no-go decision points. The project has strong links across the value chain of partners that will be needed to bring the innovation to market, which is a strength, with a strong emphasis on evaluating whether the TuFF material can be integrated into existing prepreg processes. These are nice to see given the early TRL of the activities. If successful in recovering CF for multiple life cycles, this project has the potential for large impact due to the high embedded energy in the fiber components. The project has potential for impact even if all activities are not fully successful (e.g., the value of CF recovery and reuse, even if novel matrix development is not achieved). If successful, demonstrating the compatibility of TuFF with CF recycling would strengthen the case for the commercialization of a potentially revolutionary composite processing technology. Addressing the high embedded energy of CF composites—which have a role to play in lightweighting and clean energy technology by enabling the use of recycled short fibers—has wider decarbonization impacts.

- I was unable to determine where the team is in the timeline (slide 9 is opaque to an outsider) and whether the unknowns are resolvable in the proposed timeline. The presenter did not shape the presentation to match the time allocated. This made it difficult to assess. Streamlining the presentation would help strengthen the clarity of the narrative to make it more effective. Combining the ~7 slides with pinch points and a timeline into one clear slide would have been great. Although some visuals are helpful and text-heavy slides can get in the way of communication, this slide deck has many visuals that are not doing the work to justify their inclusion. The red and green text in the slides was helpful to me to illustrate benchmarks and comparison; I recommend using a tool such as https://www.color-blindness.com/coblis-color-blindness-simulator/ (there are others, this is not an endorsement of any particular tool) to make sure that this color distinction is viewable by all potential viewers.

- This project leverages a DARPA-funded TuFF manufacturing facility and merges several key technologies to design readily deconstructable and recyclable CFC. The demonstrated TuFF process is central to the proposition in this project. The recycling by design landed on MVL as the better monomer candidate to produce a resin matrix that is both recyclable and has the desired physical properties. I did not understand the rationale for using model small molecules and polymers for the catalyzed degradation instead of one of the new recyclable resins. Is there insufficient quantity? Please explain the task priority: It is stated that working to overcome surface residuals and appropriate length sorting of fibers is critical, as these factors negatively impair performance, yet the tasks for BP3 do not reflect this. Will this impair/impact the go/no-go for BP2, where 95% retention of property(ies) for recovered composites must be achieved (to date at 60% for tensile strength)?

- I cannot understand what the TuFF process is or how it works from the presentation and additional questions after the session. I appreciate that the team has three industry partners already. I would encourage the team to share samples with these industry partners as well as with their academic partners.

PI RESPONSE TO REVIEWER COMMENTS

- I would like to thank the reviewers for their attention and comments on our presentation. I will try to respond concisely to the questions raised by each reviewer, and I invite those interested in further discussion to reach out to me directly at jdeitzel@udel.edu.

- Response to Reviewer 1: We thank Reviewer 1 for their kind comments. With respect to the question about communication within the group: We have regular monthly meetings for the materials development group (UD-CCM, CSU, NREL) and the processing group (UD-CCM, Composites Automation, Arkema, and Axiom) to discuss progress on various aspects of the project. We chose to divide this into two separate meetings to better manage discussions. Additional meetings between individual members are scheduled as needed to follow up on specific questions about chemistry, processing, etc. With respect to outreach to the larger community, members of the group engage in the usual academic avenues of participating in technical conferences and peer-reviewed publications. Examples of technical conferences include the Composites and Advanced Materials Expo, JEC, SAMPE, and the Frontiers in Biorefining Conference. Members of UD-CCM and Composites Automation also participate in the monthly meetings for the SAMPE recycling working group. We are, of course, open to and actively seeking further opportunities to engage with other parts of the wider recycling community.

- Response to Reviewer 2: We thank the reviewer for their comments and suggestions for streamlining future presentations. As pointed out by the first reviewer, this program is ambitious and has multiple research thrusts aimed at addressing different aspects of the composite recycling problem, which makes summarizing the program in a 20-minute time slot challenging. Moving forward, we will endeavor to present follow-on progress in a more concise manner. We also very much appreciate the suggestion of tools that can be used for making future presentations more accessible to viewers who are colorblind.

- Response to Reviewer 3: We thank the reviewer for their questions. As we discussed in the introduction slides of the presentation, we are pursuing several different research thrusts to address the key challenges associated with recycling composite systems. The first challenge is how we convert recycled fibers, which are almost always found in a discontinuous (chopped) form, into composites with properties comparable to those made with virgin, continuous CF. We address this with the TuFF process, which takes an aqueous bath slurry of discontinuous fibers and creates a preform of highly aligned short fibers that can be infused with a resin matrix and consolidated into high-performance composites. The second challenge is recycling the resin component of the composite. Here, we are pursuing parallel paths. Path 1 looks at the catalytic breakdown of the matrix for epoxy-based composites in order to recover the fiber and monomer. This is of great interest because epoxy composites comprise the majority of composites used today. In this research, NREL first developed model linear epoxy-amine molecules to be used to evaluate various base and acid catalyst candidates. This enabled exact analysis of both starting materials and depolymerization byproducts using standard chromatography and spectroscopy techniques that would not be possible with cross-linked thermosets. Using this approach, we identified an optimal catalytic depolymerization route, using base catalysts in this instance, which demonstrated the recovery of ~60% BPA monomer. Current work has begun applying this approach to cross-linked epoxy systems. Path 2 looks at the novel class of recyclable-by-design polymers, which have the advantage of a more efficient path to monomer depolymerization. For this class of materials, our baseline is the commercial Elium resin made by Arkema. For this class of material, Elium will be the baseline used to demonstrate multiple iterations of recycling because of its availability in terms of quantity. In parallel, we are also evaluating bio-derived versions of the recyclable-by-design resins being developed by Colorado State, which have the advantage of greater thermal stability (higher Tg) and more efficient depolymerization (>95% monomer recovery, compared to ~70%–80% for Elium). These materials, however, are available in relatively small quantities; thus, they are considered exploratory in nature, with a focus on determining their baseline mechanical properties and their ability to be processed into composites on a small scale. The third challenge is the need to clean the fiber surfaces of residue left over from the depolymerization process. At the outset of the program, it was understood that there could be a need to revitalize the surface chemistry of the recycled fibers to promote good resin adhesion, and strategies to address that issue are in place (ozone treatment of the surface, followed by vapor deposition of adhesion promoters); however, what we did not anticipate at the program's start was the sensitivity of the TuFF process to the presence of residual byproducts of the depolymerization process on the fiber surface. As a result, a major focus of the research at the end of BP2 and into BP3 will be to look at how to (1) optimize the depolymerization process for all matrix candidates (epoxy, Elium, RgB resins) to minimize residue and (2) develop low-cost, non-energy-intensive strategies to clean the recycled fiber surfaces in order to get optimal dispersion in the TuFF slurry. This will be the key to maximizing the full translation of strength in the recycled fiber TuFF composites (which has already been demonstrated for both chopped virgin CFs and waste CFs from weaving and prepregging processes that have not been exposed to a resin matrix). Finally, it is important to note that the demonstrated 100% translation of modulus and 60% translation of tensile strength for the recycled TuFF composite exceeds the published values for composites made from recycled short fibers by a large margin. We believe that we understand the mechanisms (adhesion of filaments into small fiber bundles due to the residue from depolymerization byproducts), and, as previously stated, a major focus of BP3 will be looking at ways to limit the formation of the residue as well as potential methods for removing it from the fiber surface.
- Response to Reviewer 4: The TuFF process takes an aqueous slurry of short fibers and turns them into a sheet of highly aligned fibers that can be stacked into thicker preforms and infused with resin to make composite parts with mechanical properties equal to that of traditional continuous fiber composites. A

short video describing the process in greater detail can be viewed here: https://www.youtube.com/watch?v=xKhPywwhjao. Regarding the question of sharing the TuFF material with our industry partners, that is a key aspect of the program. Composites Automation holds the sole license for the TuFF process and is the one making the material to be used in the project. They are working together with UD-CCM, Axiom Materials, and Arkema to demonstrate the feasibility of making TuFF-based prepreg materials with both epoxy and Elium resins. TuFF material has already been provided to Axiom, and we have demonstrated that TuFF/epoxy prepreg can be made using a standard commercial film impregnation process. Currently, Axiom is working with Arkema to explore potential approaches to achieving the same success with the Elium resin.

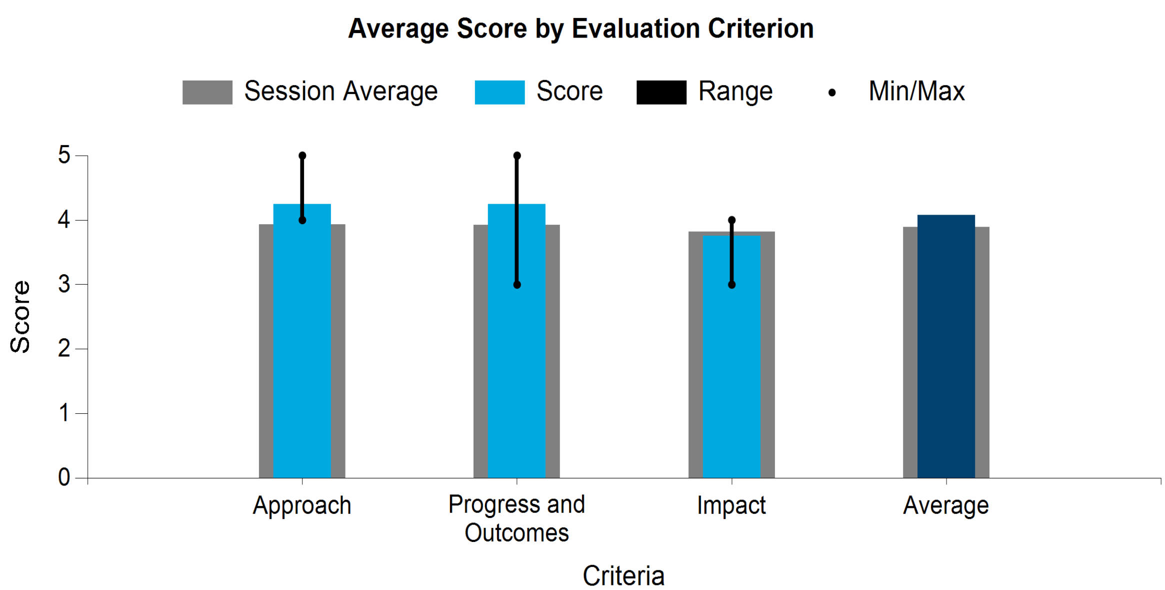
RECYCLABLE AND BIODEGRADABLE MANUFACTURING AND PROCESSING OF PLASTICS AND POLYMERS BASED ON RENEWABLE BRANCHED CAPROLACTONES

University of Minnesota

PROJECT DESCRIPTION

WBS:	AMO.07
Presenter(s):	Paul Dauenhauer
Project Start Date:	06/01/2021
Planned Project End Date:	05/31/2024
Total Funding:	$2,499,997

A chemical process is proposed for parallel closed loops of renewable polymers based on alkyl-caprolactone monomers, allowing for a broad range of materials and applications while also providing flexibility in material EOL options without long-term waste. A "lignin first" approach converts lignin (derived from trees and grasses) to aromatic monomers using the existing Massachusetts Institute of Technology and NREL reductive catalytic fractionation process. These alkylated aromatic monomers then undergo tandem reduction to cyclic ketones and Baeyer–Villiger oxidation to alkyl-caprolactones for use in multiple classes of polymeric materials. These polymers can biodegrade to carbon dioxide and water and are eventually converted back to lignin or other biomass via photosynthesis (loop #1). Alternatively, chemical processes convert the polymers back to their base monomers (loop #2). The entire project aims to design a complete process from biomass to monomers to PUs and polyesters as well as the recycling technology to convert the polymers back to the monomers.



COMMENTS

- Please double-check that the correct spelling is used for all researchers' names; this sort of detail would reflect a collaboration that is open to correction, which is important in making use of the talents of all participants. The forthrightness of the presenter in admitting the many unknowns, both personal (on parts of the project led by others) and broad (aspects of the work that are not complete) was good. The first set of unknowns does seem to indicate a weakness in internal communication. I would recommend making sure that the visuals in the presentation are interpretable to viewers and thus justify their inclusion. The process flow diagram on slide 10, for example, needs to be redesigned for this format if it is critical to understanding the narrative of the presentation. At present, it takes up space without informing.

- This project is an early TRL proof of concept for transforming a bio-based monomer to polymer to recycling via monomer recovery; however, some risks are mitigated by basing the concept on an existing lignin reductive catalytic fractionation process previously funded by DOE at NREL, as well as early TEA/LCA with a focus on meeting the target price for the monomer of interest. The project is well structured with a clear connection between the different technical tasks and work streams. The continual sharing of reaction conditions to ensure integration between processes is particularly strong. It is useful that BASF is participating so that feedback on potential applications can be identified for the resulting polymers as well as for process engineering. While the biomass monomer development is very strong and the potential for degradable polymers is good, the chemical recycling route is more uncertain, as it would require substantial changes in the recycling infrastructure as well as the collection of the material at EOL. For an early TRL activity, this project has a very good focus on ensuring a route to commercial viability with potential to commercialize the monomer even if the polymer development does not become successful.

- The management structure is good—there is clear alignment of task responsibilities among institutions and individual PIs. The team has demonstrated good oxidation outcomes (yields and selectivity) to lactone (with peroxide) synthesis from biomass precursor, and with tin octanoate catalyst, they have demonstrated excellent polymer conversions (> 95%) and depolymerization.

- The team included a reaction chart that shows which university/industry partner is responsible for which steps (slide 8). It would be helpful if other projects that divide different reaction steps between institutes could include an equivalent chart. I appreciate that the team started their TEA/LCA before going into the experimental parts. In terms of carbon recyclability, the team has been able to reach 90% conversion in a glass vial. In translating to a continuous process, the team has not been able to reproduce these yields yet. I appreciate that the team is figuring out mitigation strategies to reproduce these yields in a scalable process. The team did not include a quad chart in their presentation; these are very useful to judge their progress.

A CLOSED LOOP UPCYCLING OF SINGLE-USE PLASTIC FILMS TO BIODEGRADABLE POLYMERS

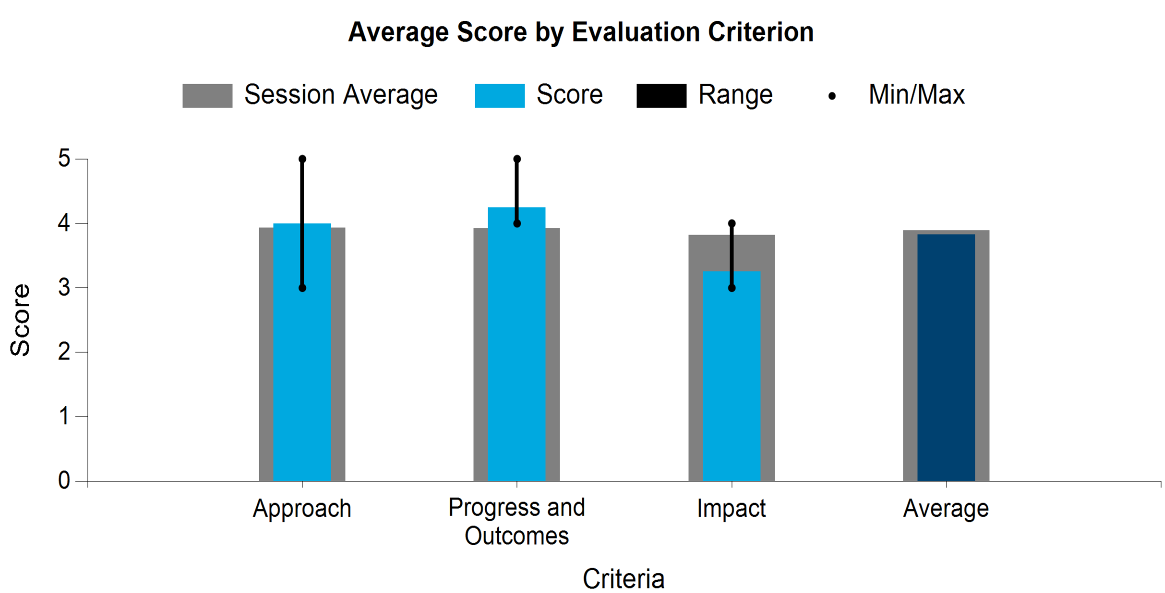
Iowa State University

WBS:	AMO.08
Presenter(s):	Xianglan Bai
Project Start Date:	06/01/2022
Planned Project End Date:	05/31/2025
Total Funding:	$2,500,000

PROJECT DESCRIPTION

The goal of this proposed project is to develop a novel plasma-biological hybrid technology to upcycle mixed single-use flexible plastic film (SUPF) wastes into biodegradable PHAs using a circular carbon approach. The detailed objectives to achieve this goal include: (1) characterize SUPFs containing mixed waste streams from MRFs to understand plastic feedstock variability; (2) innovate the industry protocol for film recycling to obtain decontaminated SUPFs primarily comprising polyolefins; (3) use low-temperature plasma and CO_2 to produce a fermentable intermediate liquid from the decontaminated SUPF wastes; (4) biosynthesize PHAs from the SUPF-derived intermediates to increase conversion efficiency and improve PHA recovery; and (5) employ TEA and LCA modeling to determine process economics and environmental impacts of the proposed technology.

Successful completion of the project will enable an innovative plasma and biological hybrid process to convert waste plastic film to biodegradable polymers with reduced energy inputs, carbon emissions, and production costs. An economically viable plastic-wastes-to-PHAs process leveraging waste CO_2 as a carbon source will advance and innovate the incumbent SOA technologies for plastic recycling/upcycling and PHA production. The project will interest stakeholders in various sectors, such as waste management industries, polymer manufacturers, polymer users, and renewable energy industries. The research outcome of the project will also advance scientific understanding and deliver new knowledge in multidisciplinary fields. Overall, upcycling plastic wastes meant for landfills into low-cost biodegradable plastics via a circulated carbon approach provides a straightforward solution to the challenging problems caused by the increased production of petroleum-based plastics and their disposal, utilizing waste CO_2 at the point source.



COMMENTS

- The slide deck is clear and comprehensive. The benchmark comparison with petroleum is helpful, but it needs clearer labeling to indicate which is which on the slide. LCA boundaries matter for this plasma pyrolysis technology; resolution of the presorting of input materials is needed to accurately assess/understand how practical it might be. The problem statement includes this aspect in the risks identified, and the description of the project team's collaboration was impressive. There is no arrow indicating contaminants on slide 5, and it does not appear to be included in the TEA/LCA on slide 14.
- The project management structure and communication are appropriate and clear. The integration of TEA to identify risks is a strength. The DEI plan is described and adequate, although moving forward, ensuring that all the diverse team experiences equal inclusion should be a focus. The goal is to convert PCR polyolefin waste into oils that can be metabolized by microbes to produce PHA. The novelty of this project is in the catalysis of the waste plastic to oils and demonstrating that oils from real waste streams can be successfully converted to PHA. As such, a continued focus on utilizing real waste streams is essential. It is great that the LCA/TEA began early in the project, but the waste handling and pretreatment steps must also be included in these. Industry partners are identified, but it is not clear how engaged they are in the project. The vision of utilizing the CO_2 from the biosynthesis in the plasma process may have implications for siting commercial systems, e.g., may require colocation. The team should consider whether it makes sense to site the waste-to-oil installation closer to waste collection sites and transport the oil to the biosynthesis facility or colocate both. A patent is pending by Iowa State, but what is the plan to pursue commercialization beyond that if the results are successful? More actively engaging potential partners for commercialization would increase the potential for impact.
- There was transparency about DEI and assumptions for the LCA and TEA. The key value is using single-use plastic as feedstock. In this early-stage project, the first step—deconstruction to make oxidized intermediate liquids—is promising, and the team has promising results. I am skeptical about whether fermentation can produce PHAs at commercial efficiency and have an economic impact. The team should engage with a partner(s) in a detailed market analysis to determine the economic value of the types of polyalkanoates that are proposed.
- I appreciate that the team is working with three MRFs to source their feedstock waste. I appreciate that the team is including TEA/LCA at this early stage of the project. The team is working with their industry partner, QEG, for bio-based PHAs and expert advice on commercially ready products. I would encourage the team to strengthen this partnership and try to exchange materials with their partners during this project.

PI RESPONSE TO REVIEWER COMMENTS

- Response to Reviewer 1: The TEA assumes a SUPF price ($30/tonne) that includes waste handling and pretreatment expenses. Waste plastics are usually available for a tipping fee of 10–$50/tonne (https://www.epa.gov/sites/default/files/2015-12/documents/historic_tipping_fees_and_commodity_values_02062015_508.pdf). This SUPF price represents a handling and pretreatment cost of $40–$80/tonne. The LCA and future TEA will include a more detailed representation of the supply chain steps from waste collection to the conversion facility. One of our future tasks is conducting a supply chain analysis to identify potential siting locations colocated with CO_2 sources. Some material resource facilities have both a CO_2 source from organic waste combustion and a plastic stream. QEG is a commercial company working on the anaerobic fermentation of organic waste to produce biofuels. QEG will work with the team on the PHA fermentation and purification and evaluate its commercialization potential for scale-up. Quasar's facility has a CO_2 byproduct (>97% purity; the residue is mainly methane) after methane is recovered for renewable natural gas. The QEG facility is generally built close to waste handling facilities. There is potential to build new refinery facilities next to QEG digesters.

- Response to Reviewer 2: The system boundary for the initial LCA begins with waste collection and ends with the PHAs and hydrocarbon production. Presorting is included in the waste collection step. Information on the presorting data and contaminants will be collected from this project. The contaminants are typically handled through sedimentation ponds and are often reused within the facility. The water is sent to the municipal water treatment plant when it becomes too dirty.
- Response to Reviewer 3: QEG is working with the research team to evaluate the commercialization potential of the PHA fermentation process. We are expecting to increase the PHA yields in the PHA fermentation process. Our commercial strategy is to co-ferment plastic wastes derived from oxidized intermediate liquids with VFAs produced from AAD to produce PHAs. We will evaluate the economic feasibility of this option. At the same time, we are working on applying PHAs for higher-value products in another project.
- Response to Reviewer 4: The project team has been working closely with QEG by holding biweekly subtask group meetings and monthly team meetings to discuss project progress and develop industry-relevant strategies for PHA synthesis/extraction. Physical material exchanges are also planned.

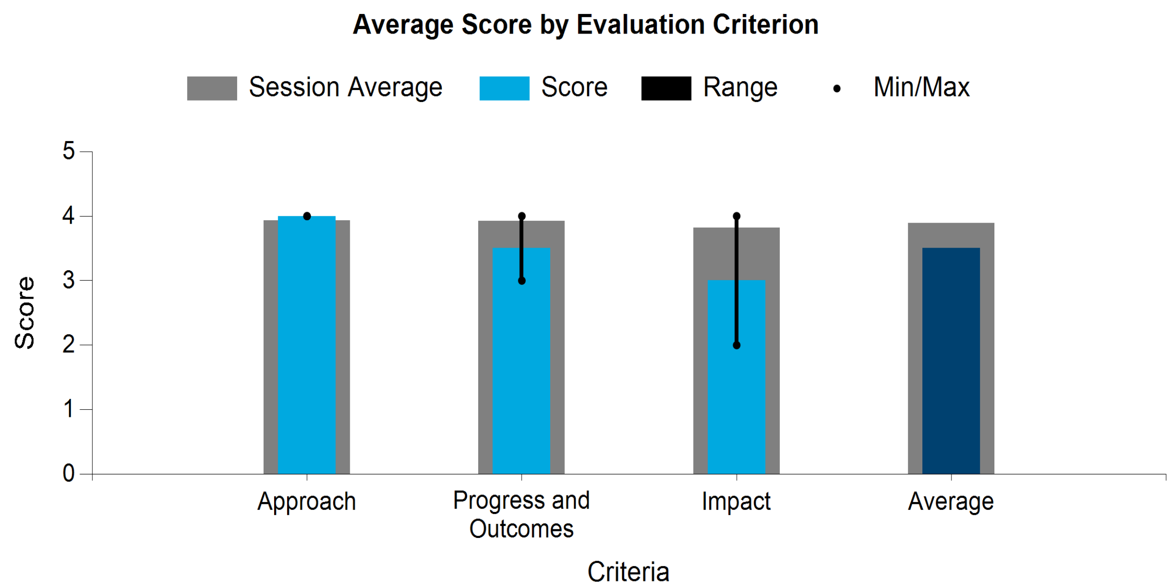
INTEGRATED CHEMOLYTIC DELAMINATION AND PLASMA CARBONIZATION FOR THE UPCYCLING OF SINGLE-USE MULTI-LAYER PLASTIC FILMS

University of Massachusetts Lowell

WBS:	AMO.09
Presenter(s):	Hsi-Wu Wong
Project Start Date:	07/01/2022
Planned Project End Date:	06/30/2025
Total Funding:	$1,600,276

PROJECT DESCRIPTION

We are developing an integrated chemolytic delamination–plasma carbonization process to upcycle single-use, multilayer waste plastic films with heterogeneous compositions into high-value chemicals, carbon materials, and hydrogen. The main objective is to develop a process that is capable of (1) treating a wide range of different compositions, (2) producing high-value products, (3) using environmentally benign solvents, and (4) scaling modularly for small-scale, distributed manufacturing. In addition, the project aims to develop a more diverse, equitable, and inclusive workforce for the sustainability sector by supporting and engaging underrepresented students in STEM and underserved communities in Massachusetts. Major project accomplishments to date include (1) identification and testing of two safer solvents for film delamination, (2) design and assembly of a plasma carbonization reactor, and (3) an apparent kinetic model for noncatalytic delamination.



COMMENTS

- More explanation of how the chemical variety in the layered input will be handled, or, given the early stage of the work, an explanation of the plan to develop a strategy for this would have provided more confidence in the team's ability to achieve the intended results. The attention to worker safety is positive. More explanation of the plan for identifying input material was needed. Does the team have a sense of the type or range of films currently in the pre-consumer or post-consumer waste streams that is their target and whether that segment or range can be obtained in a form that aligns with the intended process(es)? The project team has defined their work more narrowly than I understand the remit to be.

To convey the intended information, slide 17 would need to be redesigned, both for scale/visibility and for content. The DEI plan was concrete, specific, and well described, which was distinctly superior to other presentations.

- This is an ambitious project targeting novel delamination, PET decomposition, and plasma carbonization to convert multilayer films to PET monomers, hydrogen, and carbon products. As such, it carries multiple technical challenges to be overcome. The team has identified risks and has a mitigation plan in place. The viability of the project will depend on the potential market for the variety of different products envisioned (PET monomers, H_2, carbon), and starting to think about potential commercialization routes as technological progress is made would increase the potential for impact. Additionally, considering how to incorporate real waste streams to understand the potential challenges that may arise beyond the system studied currently (contamination, wider range of compositions)—leveraging the connection with Dow and other projects in the space—would be useful. The bar for commercialization appears to be high. It appears that entirely new installations are envisioned; thus, the siting and integration into supply chains deserves consideration. As the two stages of the process (delamination/PET decomposition) and plasma can be decoupled, the team is strongly encouraged to engage with others in this space to evaluate whether the delamination step is necessary, given the high activity around different PET deconstruction chemistries and options. There was a strong DEI plan as part of the project, with outreach to a variety of different communities, including special lectures for learning-disabled students. The team is congratulated on their approach here, going beyond recruiting a diverse range of early-stage researchers. Although Dow has been active in providing advice about the selection of model test materials, identifying partners who could move commercialization forward if successful should remain on the team's radar.

- The team has just begun project work, but their goals are very clear—there are quantitative metrics, and they are very aligned with BETO goals. The team has provided one of the more detailed and active DEI plans. It is too premature to assess the impact, but the team has engaged industry partners, and this input shapes the probability that the technology products are at least commercially relevant.

- I appreciate that the team is incorporating their TEA/LCA to guide the development of their solvent and chemolysis and treats the development of TEA/LCA and their reclamation system as an interactive process. I appreciate the team's pragmatic approach to tackling waste of different compositions. The plan is to remove the PET and carbonize the material that cannot be separated out. I would want to see the team treat films of different compositions and move on to treating mixtures of films of unknown composition.

PI RESPONSE TO REVIEWER COMMENTS

- The project team is grateful for the reviewers' thoughtful comments. We have summarized the primary topics that reviewers have raised, and our responses are below.

- Treatment of real waste streams of variable or unknown composition: The two-layer commercial-grade film under study in the project thus far consists of a layer of aluminum-containing PE and a layer of PET, bonded by an adhesive layer of PU. The film was selected on the recommendation of our Dow collaborators due to its simplicity. The baseline performance of the chemolytic delamination and plasma carbonization processes can easily be evaluated and established for comparison, yet it is still a widely available commercial-grade film. We do plan to treat other real-world waste films with more complex compositions in the near future, as documented in our statement of project objectives, starting in Quarter 8 for Task 2 and Quarter 6 for Task 3. The effects of solid contamination and the ability to use melt filtration to remove such contamination will also be investigated in collaboration with our Dow collaborators. Because up to 40% of the manufactured multilayer films are discarded during the packaging fabrication processes, our initial focus will be on the pre-consumer waste streams; however, post-consumer waste films with more heterogeneous compositions (e.g., food waste) will also be studied if time permits.

- The necessity of delamination: The project team has started investigating and developing a one-step chemolytic delamination process using glycolysis. Our preliminary results showed that the model two-layer Uline film described above was partially delaminated under mild conditions (150°C, 1 hour), with the possibility of the PET layer being simultaneously decomposed into small products. The identification and quantification of the glycolysis products are underway and will be shared with the DOE team soon. The results will inform whether a one-step chemolytic delamination process or a two-step chemolytic delamination process is more feasible and economically viable.

- Commercialization pathways and connecting with potential partners: In addition to considering the trade-offs between a one-step and a two-step chemolytic delamination process, the project team is also looking into the pros and cons of a plasma carbonization process (with target products of hydrogen and carbon materials) versus a plasma-gasification process (with target products of hydrogen and carbon monoxide, i.e., synthesis gas). The process flowcharts of all these scenarios have been sketched (and shared with the DOE team during monthly progress meetings, as opposed to slide 17 presented during the Project Peer Review). The establishment of the mass and energy balance bounds of critical operations is underway. Once these bounds are established, the process flowcharts of different scenarios will be sent for TEA and LCA. We would also like to clarify that our industry partner, Dow, is not only actively engaged in providing suggestions of feedstock selection but also playing a critical role in guiding the selection of the most attractive commercialization pathways and potential products for our proposed technology.

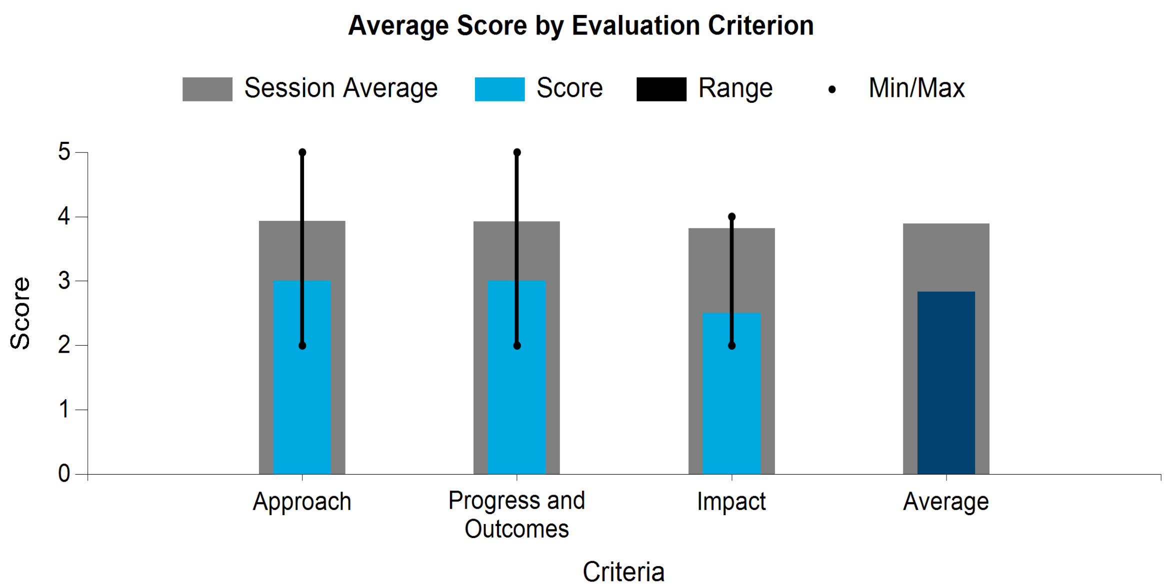
CATALYTIC DECONSTRUCTION OF PLASMA TREATED SINGLE-USE PLASTICS TO VALUE-ADDED CHEMICALS AND NOVEL MATERIALS

North Carolina A&T

WBS:	AM0.10
Presenter(s):	Debasish Kuila
Project Start Date:	08/01/2022
Planned Project End Date:	07/31/2025
Total Funding:	$2,499,994

PROJECT DESCRIPTION

The objective of this project is to advance a combination of nonthermal plasma (NTP) treatment of waste polyolefins and subsequent catalytic deconstruction to selectively make C2–C4 olefins; benzene, toluene, xylene (BTX); and oxygenated intermediates (target products) at lower energy consumption than virgin plastics. The objectives include: (1) evaluating NTP pretreatment of PE and PP and characterizing polymer modification and functionalization via scanning electron microscope, Raman, Fourier-transform infrared, and X-ray photoelectron spectroscopy techniques; (2) establishing a mechanism for depolymerization and oxidation pathways using reactive dynamics simulations; (3) developing novel catalyst compositions and structures to achieve >40% selectivity to C2–C4 olefins via the thermal catalytic deconstruction of plasma-treated plastics; (4) achieving >70% carbon utilization of target products via the catalytic deconstruction of plasma-treated polyolefins; (5) demonstrating up to 50% energy reduction in target products (C2–C4 olefins, BTX aromatics, and oxygenated intermediates); and (6) demonstrating feasibility via TEA/LCA. The expected outcomes and deliverables are as follows: (1) a lab-scale prototype of a combined NTP and upcycling process using waste polyolefins demonstrating >70% carbon utilization of target products and >40% selectivity to C2–C4 olefins with 50% energy reduction; (2) a technical report detailing the plasma conditions, catalyst compositions and operating conditions, performance of the lab-scale system, and spectra of the high-value products and byproducts; and (3) TEA and LCA with a development plan for scale-up and a commercial process. Finally, these objectives and deliverables will be advanced under a framework that motivates the improved inclusion of underrepresented groups in STEM through mentorship and investigator engagement in equity and inclusion training.



COMMENTS

- I would suggest updating the slide deck for future presentations to clarify what activities are being evaluated and included in the team's analysis and what is meant by "renewable energy." The work of preparing waste materials that are ready to use is in the flow drawing but not in the to-do list. Excluding the LCA of the input materials significantly affects the ability to evaluate the proposed and accomplished outcomes. The benchmark table in slide 4 is helpful, but the scale of the information on this slide renders much of it invisible. Consider redesigning this, possibly into multiple slides where each has one point. A plan for tuning the project for target olefins would be reassuring. Slide 13 probably contains really useful information. "Key performance parameters to be tracked through the lifetime of the project" and benchmarks' "yield slate" are both potentially informative for a reviewer (like me); however, the slide is confusing, and I was unable to identify those items.

- This project was the most difficult for me to understand, both in terms of the approach and progress. As such, I am concerned that even if the project is technically successful, communicating about it to those who could move the technology forward to commercialization will become a barrier. This should be a point of attention for the project team. Given the size of the DOE investment ($2.5 million), the desired outcome of moving from TRL 2 to only TRL 3 is substantially less than other, smaller projects in the portfolio. Because the project was delayed in starting, there has been limited progress, and as such, it is appropriate for this beginning state. Because one of the benefits envisioned is the ability to use green electricity rather than thermal cracking, it is worth noting that the electrification of crackers is planned at the commercial scale in Europe, so there are potential other routes to providing a similar benefit. Given the range of partners involved, ensuring clear communication between different work streams will be important. Whereas the carbon utilization is compared to pyrolysis, the energy metric is baselined against fossil olefins. To judge the commercial potential of this process, carbon utilization, energy, and cost should be benchmarked against a pyrolysis recycling route, as that is what it would be competing with in the market.

- This project is in a very early stage—just beyond the conceptualization stage. Perhaps this accounts for the fairly diffuse project milestone statement in the quad chart. As such, I can recommend that the team develop a more detailed valorization plan. The transition in technology readiness is not very ambitious for the project's lifetime and leads to concern about how transformative and impactful the technology will be.

- The researchers were not able to effectively communicate their plans for the future development of their work. The preliminary data does not show production of their desired targets. I would implore the team to put their future development plan down in writing, demonstrating how to get to these targets in higher selectivity using their plasma treatment. I appreciate that the team will use actual plastic waste in BP2.

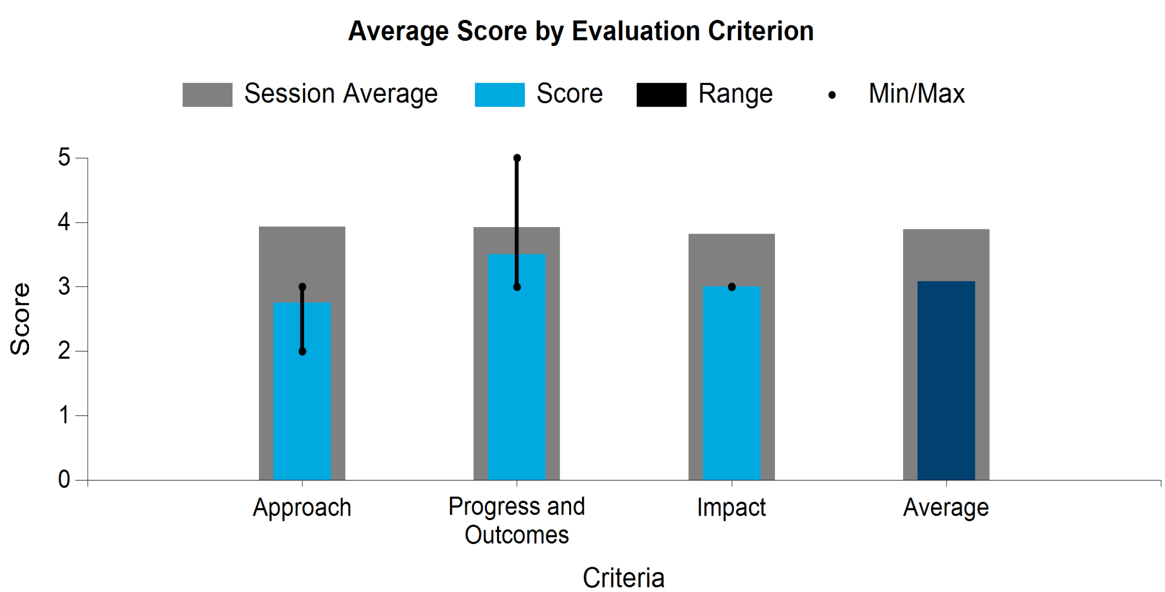
PROCESS INTENSIFIED MODULAR UPCYCLING OF PLASTIC FILMS TO MONOMERS BY MICROWAVE CATALYSIS

West Virginia University

WBS:	AMO.11
Presenter(s):	Yuxin Wang
Project Start Date:	07/01/2022
Planned Project End Date:	06/30/2025
Total Funding:	$1,500,001

PROJECT DESCRIPTION

The overall objective of the project is to develop a plastic film upcycling technology that is economically feasible, has lower GHG emissions, and achieves efficient conversion of the embodied energy of plastics to value-added monomers. Compared to a conversional ethane or naphtha cracking process for ethylene production, the proposed technology can achieve 55%–66% energy savings, 50%–63% GHG reduction, 57%–76% post-use carbon management, and 69%–75% cost savings. The technology is based on microwave-specific effects on the heterogeneous catalytic conversion of plastic films to monomers. The use of microwaves enables the depolymerization of plastic films both kinetically and energetically favorable at lower temperatures with high selectivity. The project will be accomplished by exploiting microwave-initiated interfacial chemistry in the selective cracking of C-C bonds in plastic films. The project will focus on the development of microwave-susceptible catalysts, microwave reactor design, and process optimization. Specifically, the scope of the project consists of designing and building a continuous feeding microwave reactor for process scale-up as well as conducting TEA and LCA to evaluate economic, energy, and environmental benefits. Kinetic parameters of the optimized catalysts will be determined based on which reactor and process designs for the pilot- and commercial-scale microwave catalytic upcycling system are developed.



COMMENTS

- Slide 2 has a cycle diagram that nicely outlines the ins and outs of the process, although the subsequent parts of the presentation do not account for the non-carbon fraction of the waste stream that would be coming in. The benchmark slide is also well done. There was insufficient information on the risks and mitigation factors. The DEI plan seems underdeveloped compared to the timeline and the other projects at this stage.

- This project targets the catalytic decomposition of waste polyolefins to BTX and olefins via microwave catalysis. This route would have a lower energy requirement and a higher yield than current alternative recycling routes (pyrolysis, gasification, hydrogenolysis); however, there are two key technical hurdles. One relates to the design of a continuous microwave reactor handling solid/high viscous feedstocks. The second is the need to recover and recycle the very high catalyst loadings needed because the catalyst also controls the heat transfer in the system. As real waste streams will contain both contaminants that may poison the catalysis and nonorganic content (pigments, fillers), continual recycling of the catalyst will either lead to accumulation of ash in the system or require separation from the catalyst. To understand if these challenges can be overcome, real waste materials should be incorporated early in the project. A DEI plan exists but is rather limited in ambition, as it just focuses on recruiting underrepresented researchers. The team is encouraged to monitor the inclusion that the team members experience in the project.

- The team is leveraging an existing Advanced Research Projects Agency–Energy project to inform the design of the microwave reactor. Because this is a fairly basic research project, I would like to see more details in the plan to examine catalyst and reaction conditions (lower temperature). It would also be helpful to have the economic analyses define a desirable product mixture target (e.g., % BTX) that achieves the most desirable economics. Currently, the demonstration or proof-of-concept system deploys a high catalyst load. The key for chart 12 for the gas composition is confusing; both the pink and blue are designated as C2–C4, so it is not easy to interpret what the compositional change is between the thermal and microwave processes.

- I appreciate that the team is building a preliminary TEA/LCA in BP1 and is then expanding on it in BP2. I would like to see if the team can increase selectivity by its choice of catalyst as well as using a feedstock that is closer to real-world waste. In terms of collaboration with industry, I would love to see the team reach out to industry partners to use their products as feedstocks for resin production as well as sourcing waste to use in their process.

PI RESPONSE TO REVIEWER COMMENTS

- Many thanks to the review panel for their time and comments. Through this project, we strive to develop a SOA microwave catalysis technology to decompose plastic polyolefin films to valuable monomers. The catalysts and continuous microwave reactor are the two main challenges in this project. To mitigate these risks, a microwave-susceptible catalyst will be developed and fully evaluated to optimize the activity and selectivity to the target monomers. Meanwhile, the kinetic parameters (reaction temperature, ramping rate, catalyst/plastic ratio, etc.) will also be fully studied. Moreover, as part of the real-world waste feedstock and continuous feeding process, catalyst poisoning and recovery will be studied. To mitigate the risk of catalyst deactivation by contaminants in waste plastics, we plan to develop a catalyst regeneration protocol. Meanwhile, we will develop low-cost catalysts that can be purged with ash at EOL. In addition to collecting real-world plastic for laboratory continuous reactor processing, we have reached out to a single-use plastic recycling company for commercial-scale processing information, which will facilitate the design of a larger continuous flow reactor. Further, the TEA/LCA models will be developed to analyze the economic, energy, and environmental benefits of the developed technology with real-world plastic as feedstock based on laboratory results and industry information. As in the beginning stage (Quarter 1), DEI is still in development; however, all team members are working on it, and we expect the whole team to foster more DEI at all levels. In addition to recruiting underrepresented researchers, we will also monitor how inclusive the team members' experience is. Outreach activities are also planned; these include volunteering in initiatives that educate K–12 youth on STEM principles and/or the importance of plastic recycling/circularity, and disseminating information about plastic waste and recycling in local communities.

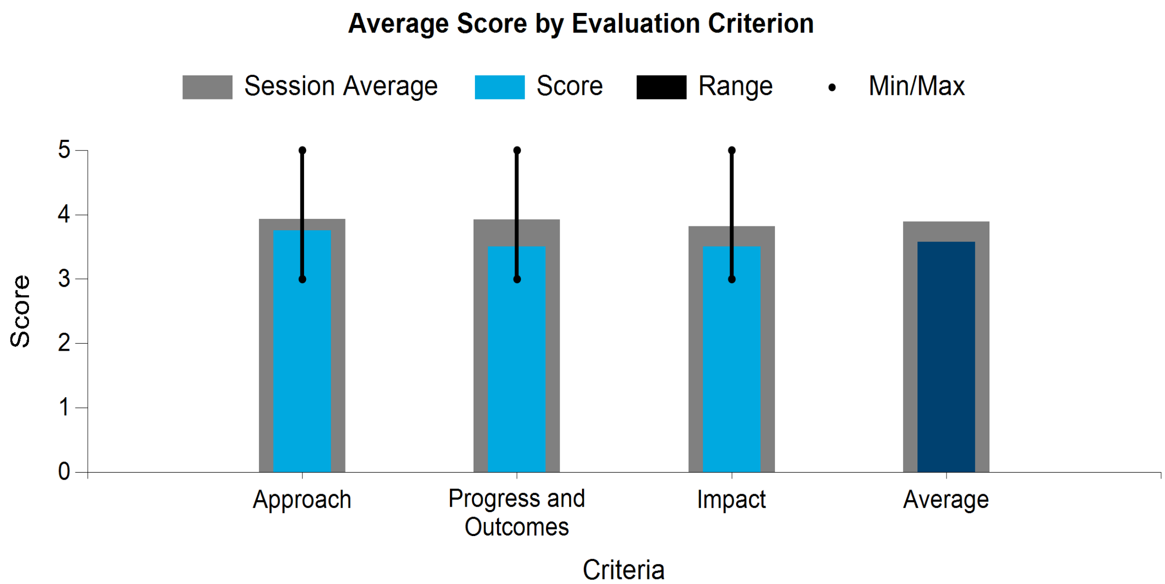
ALL-POLYESTER MULTILAYER PLASTICS (ALL-POLYESTER MLPS): A REDESIGN FOR INHERENTLY RECYCLABLE PLASTICS

Michigan State University

WBS:	AMO.12
Presenter(s):	Muhammad Rabnawaz
Project Start Date:	06/01/2022
Planned Project End Date:	04/30/2025
Total Funding:	$1,705,811

PROJECT DESCRIPTION

Due to their adaptability and affordability, more than 100 million tons/year of flexible and rigid multilayer plastics (MLPs) are produced worldwide, accounting for >30% of all plastics produced; however, MLPs possess complex structures comprising up to 12 layers of various materials with different physical, mechanical, and chemical properties. This complexity limits the recyclability of MLPs and impedes the development of EOL solutions. The overarching goals of this project include: (1) producing "all-polyester MLPs" that offer packaging performance (sealing, barrier, mechanical, etc.) that matches or exceeds those of commercial 5–12-layer MLPs; and (2) providing multiple EOL solutions (e.g., chemical and mechanical recycling) for all-polyester MLPs.



COMMENTS

- Please check the slides against a colorblindness tool. Despite having only the slides to review (no oral presentation), I am able to follow the narrative of the presentation and understand where the project is and where it is going. This is excellent communication.

- This project focuses on the development of an all-polyester multilayer film for food packaging applications. As such, it is envisioned that the films can either be mechanically or chemically recycled with high carbon recovery. A strength of this project is the active involvement of Amcor to provide input on processing and property requirements. As such, this project has a wider view on performance metrics than many of the other projects presented. This is a strength of this work. There is a DEI plan included, and it is good that the team is not just focusing on completing an activity but on following the impact of the outreach. The project is at an early stage (month 8) but is on track for meeting the milestones identified. Incorporating TEA/LCA as early as possible to help steer development choices is recommended. Because chemical recycling is an envisioned EOL option, engaging with other projects focused on the chemical recycling of polyesters early on is strongly encouraged. As the LCA has not been completed and the composition of the materials is not shown, it is very difficult to judge the potential environmental impacts of the candidate materials at this stage of the project.

- This is a very intriguing proposal to create an all-polyester multilayer film; I would have liked to have heard the presentation. The team has engaged a good industry partner; Amcor is an industry leader in providing film packaging for a broad variety of consumer and specialized applications. The DEI plan has specifics, and the team has good engagement activities with underrepresented students. The technical approach description could be significantly strengthened (or more helpful to the reviewer) if, instead of providing sweeping performance metrics for the all-polyester multilayer film, it showed discrete tables that aligned the performance of each candidate polyester functional replacement (polyester 1, polyester 2, and polyester 3) against the specifications for each of the three layers (structural, barrier, and sealant) that each will replace. Other elements that would be helpful in the project description include (1) describing what the technical risks are and how you plan to mitigate them; (2) tailoring the desired barrier properties to a particular application (the needs are different across various applications/industries)—what is the first target for the prototype you are developing?; (3) developing a testing strategy for multilayer stability; and (4) addressing other issues—e.g., sterilization and regulatory considerations.
- I would encourage the team to perform a TEA and LCA early in the development cycle of this multilayered product. If possible, I would encourage the team to have industry partners test their multilayer films. As this will be in contact with food, the team will need to ensure that their multilayered product is food safe and passes all regulations. As this is a product that is supposed to be a widely available technology (recycling), I would encourage the team to *not* support exclusive licenses to the technology.

PI RESPONSE TO REVIEWER COMMENTS

- We would like to express our appreciation to the reviewers for taking the time to participate in BETO's 2023 Project Peer Review of this project and for their thoughtful and excellent analysis and constructive feedback on this project. In response to the valuable feedback from our reviewers, we are keen to incorporate their insights into our future work. One recommendation was to include TEA and LCA in the early stages of our project to guide our development choices. We anticipate that our initial TEA and LCA will be ready by or before month 12 of this project (currently, we are in the 10th month of this project). We will also start early on the chemical recycling portion of our project, focusing on polyesters at Michigan State University. We will be supported in this endeavor by the advice and expertise of PNNL. We will also create discrete tables where we will align the performance of each candidate polyester functional replacement against the specification (structural, barrier, and sealant). Managing potential risks is a crucial part of our process. Therefore, our project team consistently discusses potential technical risks and brainstorms mitigation strategies during our regular project meetings. As recommended, the project team will finalize the initial target application(s) for the first multilayer polyester prototype early on. Further, we will work with our industry partner on the stability of the multilayer, sterilization, and regulatory considerations as well as on the testing of all necessary properties of the multilayer films by industry. Finally, following the recommendation, we will work closely with our Michigan State University technology team to develop a nonexclusive licensing strategy for this technology.

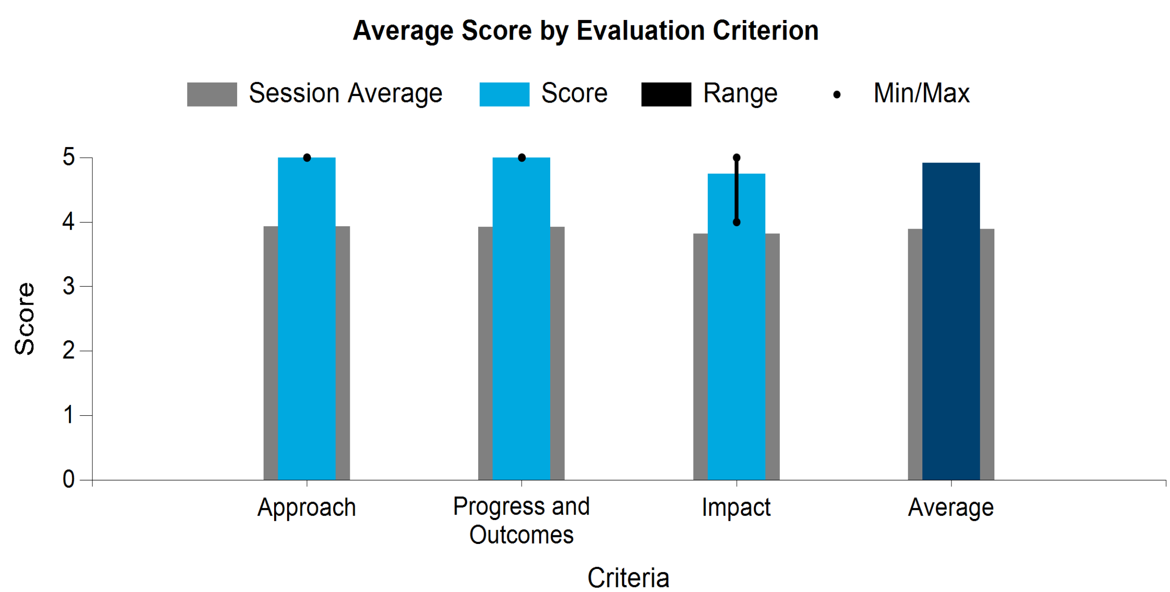
INTRODUCTION AND BOTTLE OVERVIEW

BOTTLE Consortium

PROJECT DESCRIPTION

WBS:	BOTTLE1
Presenter(s):	Gregg Beckham; Megan Krysiak; Michelle Reed
Project Start Date:	10/01/2020
Planned Project End Date:	09/30/2023
Total Funding:	$250,000

The BOTTLE Consortium aims to develop robust processes to upcycle existing waste plastics and develop new plastics that are recyclable by design—both of which are in direct alignment with DOE's Strategy for Plastics Innovation. We accomplish our work in the BOTTLE Consortium through an organizational framework that includes three primary research tasks (Deconstruction, Upcycling, and Redesign), which are supported by three crosscutting tasks (Analysis, Characterization, and Modeling). BOTTLE also has tasks focused on industry engagement and DEI. This presentation will review the approach and management structure of BOTTLE, the importance of analysis-guided research, and the key metrics for carbon, economic, energy, and GHG emissions. In the FY 2021–FY 2023 period, BOTTLE has drafted and enacted a comprehensive DEI plan, assembled a world-class technical advisory board to provide constructive feedback on our performance, had our first in-person all-hands meeting in summer 2022, and onboarded and off-boarded research activities based on active project management and analysis. From an impact perspective, BOTTLE researchers have published more than 40 peer-reviewed manuscripts (many in leading journals), submitted >30 patent applications, and initiated six funds-in industry partnerships.



COMMENTS

- This was a polished presentation that covered the bullet points in each review question.
- Overall, the BOTTLE Consortium approach and management structure are extremely well thought out, well executed, and well aligned with DOE and BETO priorities and strategic goals. Particularly impactful is the integration of analysis into the projects early on, as well as the yearly portfolio review, which has resulted in decisions to stop activities and focus on more promising routes. The active engagement of the technical advisory board should be continued and fostered, with composition

reevaluated as the BOTTLE portfolio shifts and as projects move up in TRL. The communication structure is clear, and there are multiple routes being leveraged for internal and external collaboration. As BOTTLE expands, active evaluation of the communication structure is encouraged to ensure it is still effective. The DEI plan is under development, and several supporting activities have already been accomplished. With continued attention, this area is on a good trajectory. One aspect of the value chain that is not currently strongly represented is the recovery of plastic waste that would flow into the proposed processes. Whether this should be encompassed within BOTTLE or a different DOE activity, it is important to include so that the technologies developed have a viable supply chain and are developed to handle realistic feedstock forms, compositions, and contaminants. As their activities are moving toward TRL >2, BOTTLE should develop a strategy here to ensure that the commercialization of the technologies has the desired impact. BOTTLE has the opportunity to be a thought leader in driving sustainable plastic innovation forward. BOTTLE serves as a convenor of actors along the value chain to not only develop new technical routes but also to nucleate actors behind the most promising pathways to help build the critical mass needed for deployment and drive system change.

- Projects nested in the BOTTLE Consortium are advantaged by the technical resources in this integrated structure of five national labs, five academic institutions, and industry partners linked by CRADAs. BOTTLE meets and excels in all review criteria. It is very hard to find a better-structured program with a clear, cohesive structure and exceedingly capable leadership. The meeting structure seems to be sharing data and resources efficiently and regularly with internal members and also with external audiences, as the team has created a functional, live website to push out current articles. The risk/mitigation strategy is solid, and BOTTLE is swift to sunset projects that are not on a trajectory to meet targets. The DEI plan is thoughtful and proactive. BOTTLE has demonstrated a strong commitment to moving projects toward development and commercialization with a much-strengthened engagement with industry. Several actions reveal that the consortium leadership team seriously responded to suggestions from the 2021 Project Peer Review. These inputs will inform their asks in the 2023 renewal application, which will expand their reach to bring on institutions/experts with large-scale polymer processing capabilities. The consortium is aware of at least one gap—polymer processing—that would enhance its agility in moving its targets, and this will likely be addressed in the 2023 renewal proposal.

- The team leader seems to have very open communication internally, which is also evident in their very cohesive presentation. The lead is aware of risks and mitigations in individual sub-efforts. It is great that the team conducts portfolio analysis to decide what to accelerate, discontinue, or continue. These decisions are usually made within a group; the group members take on a new project, and this does not impact funding for each individual group. I wonder how this project will develop in the future. Is the team going to return to early-stage research and get more hard problems from industry to solve, or is the team going to push the research further down the TRL pipeline? There are advantages and disadvantages to both approaches. If the team wants to continue into higher TRLs, I believe they will have to bring additional teams into the consortium that are specialized in processing and scale-up.

PI RESPONSE TO REVIEWER COMMENTS

- We thank the review panel for their positive feedback. We also sincerely appreciate the constructive input, and we will act upon these suggestions. In terms of the "plastics waste recovery" value chain, this is excellent feedback, and we agree that this is a critical element of the supply chain to ultimately enable a circular plastics economy. We have previously worked with other BETO-funded and DOE-funded efforts to examine plastic waste mass flows in the United States on a geographical basis (e.g., https://doi.org/10.1016/j.resconrec.2022.106363). Moreover, AMMTO funds efforts in various analysis-driven plastic supply chain modeling efforts that are complementary to BOTTLE's mission (e.g., see https://doi.org/10.1111/jiec.13423). Last, and especially as we intend to advance a few key deconstruction technologies up the TRL, we are also working with industry partners to source realistic plastic waste. The reviewers identified polymer processing as a current gap, and, during the Q&A, we discussed monomer synthesis and manufacturing R&D at a larger scale as another gap. Both gaps will be addressed directly as we move into the proposed renewal phase for the FY 2024–FY 2026 project cycle. The comments regarding how to balance early-stage research relative to pushing promising technologies up the TRL ladder is excellent, and we are carefully considering what the balance of these activities should be going forward. We mostly view the balance as a funnel, where multiple promising early-stage technologies should be evaluated in parallel, and only select technologies should be selected, guided by both technical promise/feasibility and analysis, to be advanced in TRL. Moreover, as we advance technologies along the TRL ladder, we will also actively pursue additional funding mechanisms, including industrial funding, to support and eventually off-board scale-up to industry partners and other support mechanisms. Doing this will allow us to balance our resources to maintain a "full pipeline" of early-stage, promising innovation and a select number of technologies that BOTTLE can advance to TRL 4–5 before transferring them to other support mechanisms.

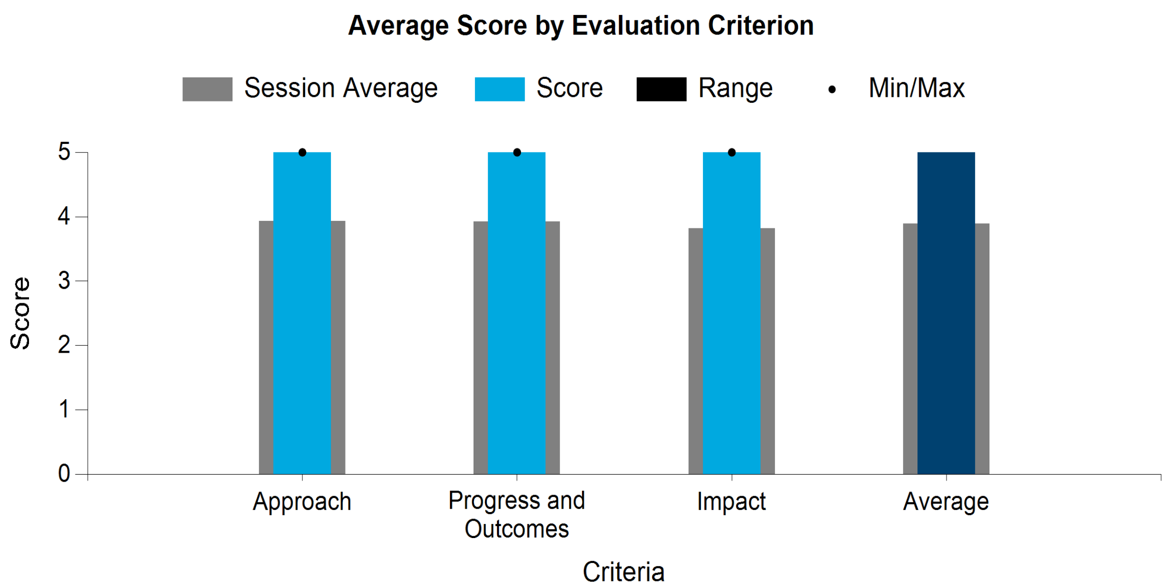
ANALYSIS

BOTTLE Consortium

PROJECT DESCRIPTION

WBS:	BOTTLE2
Presenter(s):	Jason DesVeaux; Michelle Reed; Taylor Uekert
Project Start Date:	10/01/2020
Planned Project End Date:	09/30/2023
Total Funding:	$400,000

The use of analysis is foundational to BOTTLE. In particular, we conduct TEA, LCA, and supply chain modeling for each new BOTTLE innovation, ranging from new deconstruction and upcycling technologies to redesigned polymers. These critical analysis tools are used in parallel with laboratory research to ensure that BOTTLE technologies can ultimately meet our key metrics. The BOTTLE team used analysis early in the current 3-year project cycle to identify research priorities from a scale, energy, and economics perspective (https://doi.org/10.1016/j.joule.2020.12.027), and recently we presented a comprehensive framework for comparing new circularity-focused approaches to linear, incumbent practices (https://doi.org/10.1146/annurev-chembioeng-100521-085846). This presentation will also review case studies in analysis for closed-loop PET recycling across a range of recycling methods, baseline studies that we have conducted for plastics pyrolysis and gasification as another comparator for BOTTLE technologies, and a comprehensive study of existing closed-loop recycling technologies from a full suite of environmental impacts and feedstock quality metrics. We have undertaken studies of circular polymers being developed in the Redesign task, and we will present an exemplary case for a bio-based acrylic polymer that can replace polymethyl methacrylate. Overall, analysis is critical to enable the BOTTLE Consortium to focus on impactful, realistic technologies.



COMMENTS

- Even if all the projects are not using the same LCA, it would be helpful for each to illustrate what is included in the analysis of what they are doing. For some, it appeared that it only included carbon footprint, which, while important, does not reflect the full range of impacts that plastic may have. It would benefit from including analysis of plastic additives, for example. The existence of this analysis effort is very reassuring for the overall effort.

- The analysis component of BOTTLE is one of its strengths and should be commended. By publishing transparent, consistent comparisons of different routes, it has the potential to influence both industry decisions as well as policy ones. As such, it is important that these analyses are periodically updated as more data become available or as improvements are made. It was particularly gratifying to hear that the analysis publications have resulted in industry reaching out to explore publishing high-quality LCA of their pilot or commercial processes. For increased impact, as the analysis activities identify actionable opportunities to decrease the impacts of different routes, it would be useful to collect these across the portfolio to identify which process improvements will have the greatest crosscutting benefits. The early and frequent engagement between experimental efforts and analysis should continue, as this is key to maximizing decarbonization potential at speed. The outreach efforts to socialize tools such as materials flow through industry at the American Physical Society short course are encouraging for widening the life cycle thinking within the polymer development community. Gathering feedback from industry and academia on how to make the analysis outcomes, tools, and data should be intentional to maximize impact. The interactive graphic with links to publications is a nice start, but more may be needed to make the results optimally useful for the community.

- This project provides a very sound and disciplined analysis approach that is consistently and uniformly applied to each program. The project fosters a transparent commitment to open-source practices and published a framework for plastics analysis in 2022. The case examples are powerful; these include the upfront sensitivity analyses that enabled a credible, specific target for the research focus (e.g., cheaper feedstock to achieve price parity of recycled (r-TPA) versus virgin (v-TPA) to accomplish a more impactful process. The application of this analysis framework has achieved the desired impact of enabling the deployment of technological routes with a higher commercialization potential.

- I think this project is absolutely fantastic. The team's TEA and LCA results are used in other parts of the consortium to inform go/no-go decisions. If possible, I would love other BETO projects to build on this very thorough analysis as well. I would use this analysis as a benchmark for everybody else's economic and sustainability analysis.

PI RESPONSE TO REVIEWER COMMENTS

- We are delighted to see that the review panel found the Analysis task to be a strength of the BOTTLE Consortium. We agree with the comment regarding updates to the analyses as new data and insights become available. We are indeed doing this now for PET enzymatic hydrolysis and waste plastics gasification, for instance. The idea about crosscutting benefits across analysis cases is good—we will consider how to do this carefully. We will continue to expand the functionality of the analysis-focused part of the BOTTLE website. Another reviewer remarked on the fact that we are not using the same LCA—we note that this confusion is an artifact of the short nature of the presentations. We have a self-consistent LCA approach across the BOTTLE Consortium, but due to time constraints, we were unable to show all the details. Further, we are actively working on incorporating additional impact categories—especially those focused on plastic additives, microplastics, and social effects—as more data become available. Overall, we were delighted to receive such a positive reception of the Analysis task from the review panel.

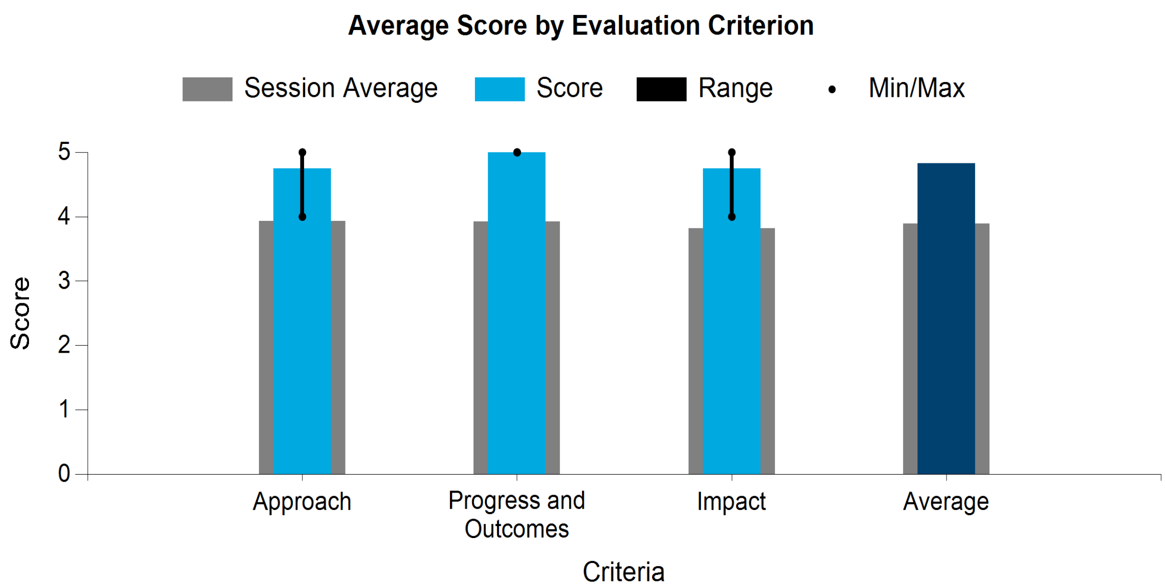
DECONSTRUCTION

BOTTLE Consortium

PROJECT DESCRIPTION

WBS:	BOTTLE3
Presenter(s):	Michelle Reed; Taraka Dale; Yuriy Roman-Leshkov
Project Start Date:	10/01/2020
Planned Project End Date:	09/30/2023
Total Funding:	$1,737,500

The Deconstruction task is pursuing the chemo-catalytic and biocatalytic depolymerization of today's plastics into molecular building blocks that can either undergo closed-loop recycling or valorization to higher-value products. For the FY 2021–FY 2023 project cycle, Deconstruction is the largest task in the BOTTLE Consortium due to the urgency of developing new recycling strategies for plastics that are not sufficiently recycled (or recycled at all) today. This two-part presentation will first focus on the development of chemo-catalytic methods to convert mixed polyolefin waste into small-molecule hydrocarbons, oxidative deconstruction of mixed plastic waste to oxygenates that can be converted microbially to a single product (in collaboration with the Upcycling task), and a catalytic glycolysis strategy for PET closed-loop recycling. The second part of this presentation will focus on innovations in enzymatic PET recycling through the development of process-advantaged enzymes that are specific to crystalline PET substrates and are able to operate at ~65°C and low pH. Exploratory efforts are also underway to use enzymes to depolymerize nylon and PUs. Together, the Deconstruction task in the BOTTLE Consortium has developed a suite of innovative technologies to depolymerize many commodity polymers, and we are using analysis now to determine the most impactful paths forward that warrant further scale-up and process integration.



COMMENTS

- This was very thoroughly explained and covered each evaluation bullet point. I appreciated that the first section highlighted additives to plastics as a major risk and had a mitigation strategy: controlled studies with known additives. I would have appreciated more explanation of what is meant by autoxidation. Oxidative funneling tolerates additives—are they getting included or released?

- The approach of starting with a wide range of technologies (hydrogenolysis, oxidation, glycolysis, photoelectrochemical, enzymatic) addressing key polymer classes of polyolefins and PET is appropriate. The down-selection to focus on the most promising options guided by analysis should be applauded. The results of these early TRL deconstruction efforts have excellent potential for innovation. Particularly promising is the autoxidation funneling route, which has the potential to allow the conversion of compositions used currently into more circular or benign systems. Particularly exciting are the preliminary results on the oxidative funneling of polyvinyl chloride systems. If successful, this opens a route to transform the materials of today to those of the future. The creation of a spinoff company, Tereform, based on this work is encouraging for future commercialization. The enzymatic degradation work is very well done, with a solid experimental approach. Compared to the other routes explored, it has less potential for impact. A narrowing focus will allow BOTTLE to achieve a higher impact potential. The hydrogenolysis deconstruction targeting lubricant oil would be worth benchmarking against the LOUPs project, which is targeting the same waste stream and application area.
- Deconstruction is at the heart of this technology area. The team deselected the photochemical and electrochemical approaches and diverted resources to more promising areas. The principals employ an excellent mix of model/mechanistic and engineering analyses to optimize. A nice outcome for all this work is a platform with select targets and a choice of the best feedstock and process to achieve GHG, yields, etc. The promising work with the autoxidation of mixed plastic waste streams has resulted in a spinoff company, Tereform. The crystalline PETase enzyme discovery work has the potential to be a useful preprocessing step for PET with a lower energy demand.
- It is very exciting that the team found a first enzyme that is preferential for crystalline PET. How does the very early work of Taraka Dale tie in with the rest of the BOTTLE group? Can you funnel these early TRL technologies through faster because you have all this expertise under one umbrella?

PI RESPONSE TO REVIEWER COMMENTS

- We thank the reviewers for the positive feedback on the Deconstruction task. In terms of the enzymatic hydrolysis work, we agree that this has a more limited scope relative to the other deconstruction technologies presented, and we will carefully consider this input going forward, toward maximum impact. In terms of how this fits in with the rest of the BOTTLE team, PET closed-loop recycling technologies—including enzymatic hydrolysis, glycolysis, and methanolysis—are all of keen interest to the BOTTLE Consortium, so enzymatic hydrolysis fits in under the umbrella of closed-loop PET recycling processes. For the comment regarding the oxidative funneling process tolerating additives: We did not have sufficient time to explain this in detail, but we note that organic additives are very likely oxidized in a similar manner to the polymers present in the systems. We are currently conducting further analytical chemistry work to this end to track the fate of additives in this process and others. On the point about moving these early TRL technologies faster because of the complementary expertise in BOTTLE: That is certainly our intention. We really appreciate the positive comments and the constructive feedback for the Deconstruction task.

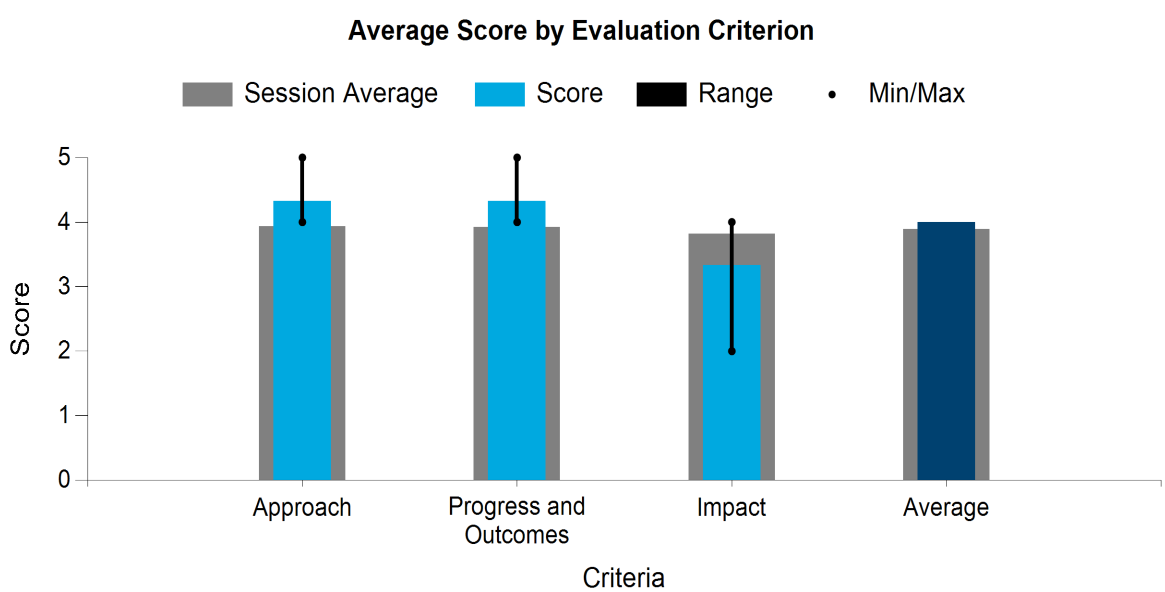
UPCYCLING

BOTTLE Consortium

WBS:	BOTTLE4
Presenter(s):	Adam Guss; Michelle Reed
Project Start Date:	10/01/2020
Planned Project End Date:	09/30/2023
Total Funding:	$800,000

PROJECT DESCRIPTION

BOTTLE's Upcycling task aims to convert intermediates derived from the deconstruction of today's plastics into molecular building blocks that can be converted into recyclable-by-design polymers with the Redesign task. Accordingly, the Upcycling task is a critical "bridging" task in BOTTLE. Efforts in the Upcycling task focus specifically on: (1) the discovery and engineering of new metabolic pathways and enzymes to convert intermediates from mixed waste plastics to central carbon metabolism; (2) metabolic engineering to convert mixed oxygenates into single compounds; and (3) the consolidated bioprocessing of PET, wherein a microbe secretes a PET hydrolase enzyme and converts the depolymerization products to value-added compounds in a single step. This work has led to the "biological funneling" of oxygenates from PET, polyethylene, and polystyrene into tunable bioproducts. The pathway discovery work has led to the discovery of pathways for PET co-monomers, including isophthalic acid, among others, which are now being engineered into production strains. For the consolidated bioprocessing of PET, we have successfully engineered a thermophilic bacterium to secrete thermo-tolerant PET hydrolases and consume both TPA and ethylene glycol, which are key steps toward enabling this concept. Overall, the Upcycling task is enabling the conversion of mixed plastics into PABPs and recyclable-by-design monomers through cutting-edge science.



COMMENTS

- Interesting question: If microbes break down waste plastic and poop out useful materials for making new plastic, does that mean the waste plastic is transformed into bioplastic by the participation of the microbes? That seems to be what this presentation is saying. This highlights the need for commonly shared definitions.

- The project could benefit from TEA to determine whether it is on target and sensitivity analyses to determine which factors to focus on to achieve the highest benefit. I am concerned about the practicality of a pure biocatalysis route to upcycling; it may not achieve practical production rates. The investigators should engage with industry to focus the upcycling scheme on reasonable metrics.

- This is a very integrated project that touches many other aspects of the consortium. I appreciate that the team is using products from the oxidative funneling process. I am excited to see the team scale up their production of monomers.

PI RESPONSE TO REVIEWER COMMENTS

- We appreciate the feedback on the Upcycling task. Whether a waste-based plastic can be considered a bioplastic is debatable, and we did not mean to convey that. Instead, the use of a microbial conversion process here enables funneling mixed-plastic-waste-derived intermediates into a single product, and bio-based substrates could also undergo similar transformations. We apologize for any confusion caused here. We are conducting TEA now on multiple upcycling pathways that involve biological transformations, and we will be able to report these analyses in the next Project Peer Review and in the peer-reviewed literature in the meantime, especially toward the synthesis of new monomers for recyclable-by-design/circular polymers.

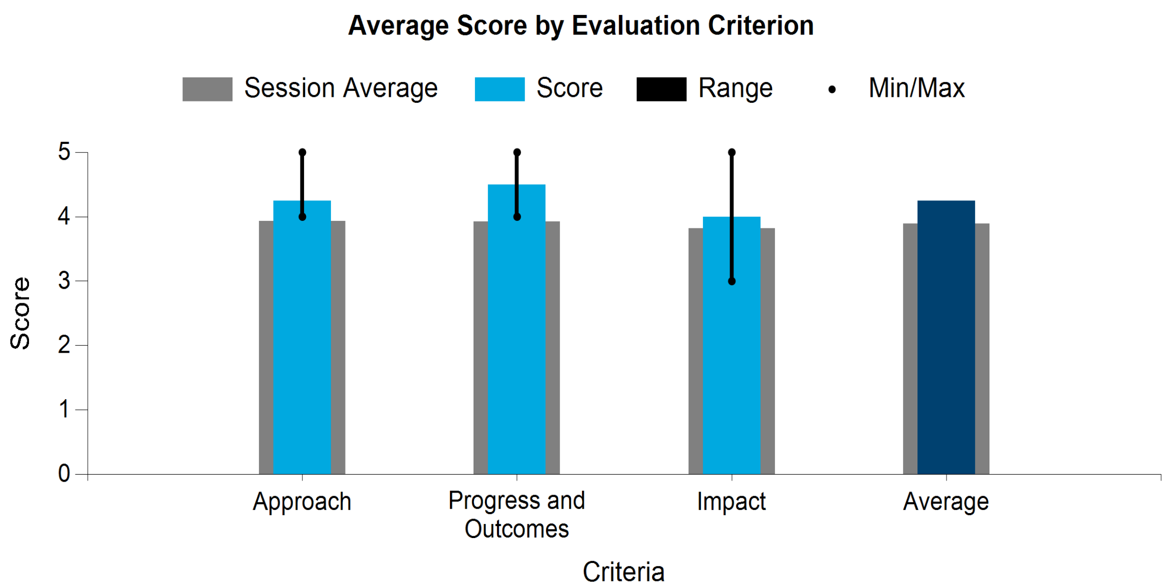
REDESIGN AND MODELING

BOTTLE Consortium

PROJECT DESCRIPTION

WBS:	BOTTLE5
Presenter(s):	Eugene Chen; Linda Broadbelt; Michelle Reed
Project Start Date:	10/01/2020
Planned Project End Date:	09/30/2023
Total Funding:	$895,000

The Redesign task in BOTTLE focuses on the synthesis of polymers that can be recyclable by design, sourced from waste-based or bio-based feedstocks, and can replace linear plastics. In close collaboration with the Redesign task, the Modeling task primarily focuses on predicting pathways (PickAxe) that can be used to efficiently manufacture building blocks, either biologically, chemically, or through hybrid approaches, coupled to an ML tool (PolyID) that can predict key properties of recyclable-by-design polymers before experimental work is initiated. These tasks have made multiple exciting innovations since FY 2021, including the development of key design principles for circular polymers; the demonstration of a bio-based, circular replacement for polymethyl methacrylate; and innovations in the "hybrid monomer" concept for polyester, nylon, and polyolefin replacements. Moreover, the Redesign task has greatly advanced the ability for PHAs to be chemically synthesized, enabling much greater control and tunability. This now includes the ability to be melt-processable to produce recyclable, bio-based alternatives to polyolefins. The Modeling task is directly addressing the cost of new monomers for these redesigned polymers through pathway predictions as well as supporting other BOTTLE efforts in deconstruction and upcycling. Going forward, the portfolio of new polymers from the Redesign and Modeling tasks will be a major focus area for the BOTTLE Consortium.



COMMENTS

- What is the thread connecting the graphs in the second half of this presentation? What is the story? The connections of the second half were not clear.

- The approach of the recyclable-by-design polymers guided by modeling is sound. Particularly strong is the integration and use of modeling to down-select what systems should be pursued from the huge potential design space; however, further model development is needed to move to a wider range of relevant properties, particularly those that will be strongly influenced by microstructure and crystallinity. An initial screening based on chain composition makes sense, but working more on developing chain chemistry–structure–property relationships and the influence of processing should be considered. Strong interaction with industry partners on the key property requirements for targeted applications (beyond just mechanical or barrier properties) should be considered, particularly as they relate to processing. As projects move toward higher TRL, a much more nuanced discussion around how performance is defined will be needed to identify areas of course. The planned activities around monomer scale-up to ensure sufficient volumes for evaluation will be critical and should be expanded moving forward. Because the early results are so impressive, starting initial TEA/LCA to help guide monomer and polymer selection is important.

- Programs like this recyclable-by-design program have benefited from early industrial guidance with realistic property targets for each major polymer class. The presentation on the bio-acrylics production of the monomer and its properties was compelling. I like that the team is building on the concept of "hybrid monomers"—e.g., 4,6-nylon and the two lactones (caprolactone and butyrolactone)—to generate polymer products that have the desired mix of recyclability and performance attributes. The outputs of performance, processability, and recyclability exceed the incumbents, and this approach highlights several good monomer targets. I like that the team has developed approaches to predict crystallization, glass transition, and melting temperature (Tc, Tg, and Tm, respectively). I appreciate developing the Monte Carlo framework.
- I am very impressed by the team's partnership with Amazon and that their materials are being used in industrial projects. I appreciate that PickAxe is freely available, and the number of PickAxe uses would be an interesting metric to track.

PI RESPONSE TO REVIEWER COMMENTS

- Thank you to the review panel for their positive input on the Redesign and Modeling tasks. We fully agree that additional metrics for predicting beneficial polymer properties are crucial, and as noted in the presentation, we are working actively to that end, including with industry input. We agree that monomer synthesis at larger scales alongside TEA and LCA will be critical in the redesign efforts. We apologize if the threads connecting the first and second parts of this presentation were not clear—the Redesign and Modeling tasks work very closely together, so we thought it would be beneficial to present them back-to-back. We share the reviewers' excitement about the future of circular polymers and the ability to predict their properties and the most efficient monomer synthesis pathways. We look forward to sharing the results to these ends in the next Project Peer Review.

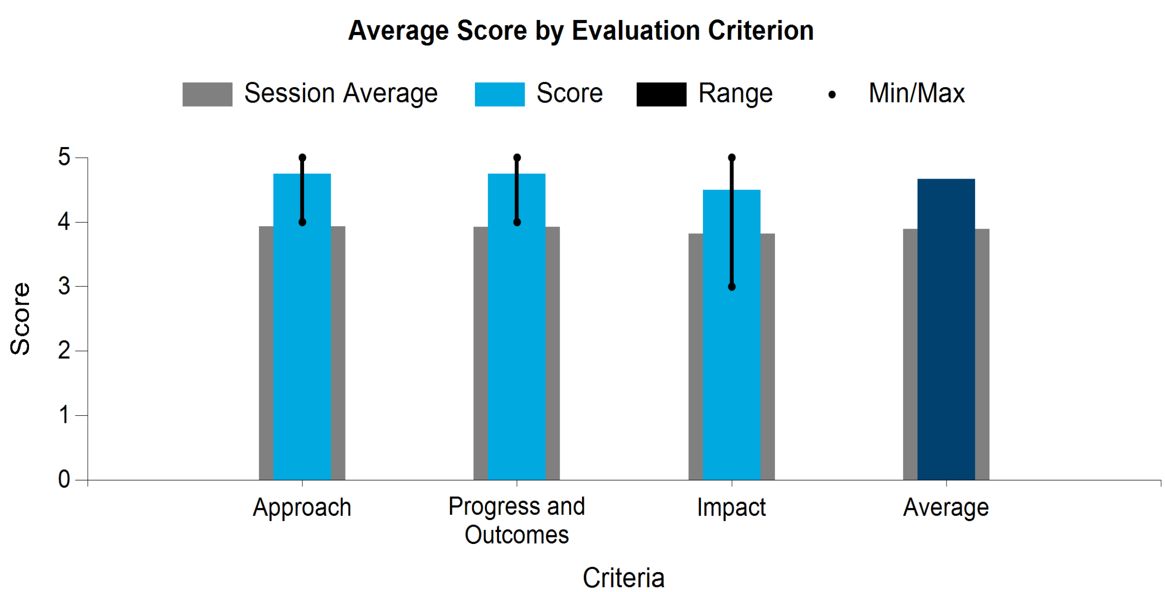
CHARACTERIZATION

BOTTLE Consortium

PROJECT DESCRIPTION

WBS:	BOTTLE6
Presenter(s):	Christopher Tassone; Meltem Urgun Demirtas; Michelle Reed
Project Start Date:	10/01/2020
Planned Project End Date:	09/30/2023
Total Funding:	$767,500

We use the synchrotron capabilities of the Stanford Linear Accelerator to characterize chemical and biological catalysts reacting with polymers with *in situ* and *operando* techniques with the Deconstruction task. This effort has developed multiple new approaches for *in situ* and *operando* measurements. These critical measurements directly inform reactor design and scale-up activities. In collaboration with the Redesign task, the Characterization task is applying both advanced scattering and spectroscopic measurements as well as conducting critical EOL measurements using ASTM-based methodologies. These key activities directly inform both polymer processability at scale and potential degradability if redesigned polymers leak into the environment. For EOL studies, we are developing new capabilities in the scale-down of biodegradation tests alongside strategies to determine the fate of redesigned polymers in realistic environments. Moreover, the Characterization task reported a framework for analyzing polymer deconstruction approaches to promote scientific reproducibility in the community (https://doi.org/10.1038/s41929-021-00648-4). We will soon report a comprehensive characterization of commercial plastics for the research community to understand the potential of additives and contaminants to affect deconstruction and upcycling approaches.



COMMENTS

- The science was solid and very interesting and gets full marks for what it is. The connections beyond interesting science (potential for use, scale-up, and commercialization) were not made clear within the presentation.

- The characterization efforts are well integrated into the projects and activities, which is a strength. Continued interaction to ensure a low barrier to interfacing as well as thoughtful decisions about when fast feedback versus deep dives are needed should be continued. Particularly nice is the characterization development allowing monitoring *in situ* in relevant conditions. Using such approaches to help identify or improve the robustness of catalysts to conditions and contaminants seems particularly important to ensure that approaches can be deployed. Integrating the EOL (degradation testing) results that monitor impacts beyond CO_2 to include methane monitoring with LCA is critical for matching systems/applications where recycling or degradation are most appropriate. Additionally, I advise the team to consider mapping inherently dissipative applications (paint wear, seed coatings) with biodegradable materials while prioritizing recyclability for applications with the best potential for material recovery and reprocessing. The work on monitoring degradation beyond CO_2 production has impact beyond just BOTTLE projects, and the dissemination of the methodologies and insights gained will benefit the entire field of biodegradable polymer development and mitigate potential unintended consequences, depending on the degradation products formed.

- The team is allocating appropriate resources (for both in-depth characterization and rapid high-throughput screening) to create reactors to mimic realistic conditions for assessing the deconstruction mechanisms. I applaud the commitment to scaling down existing ASTM test methods. Is it also possible to consider conceiving accelerated tests for internal guidance while not abandoning these long-term ASTM test measurements for promising candidates? The discovery work to elucidate the "plastisphere" microbiome is noteworthy. This will be very useful knowledge and will be a great asset for the biodegradation research community.
- It is fantastic that the characterization team is physically exchanging samples with all the other parts of the consortium and has worked with all the other groups in BOTTLE. It would be interesting to see if the team could assist other parts of the BETO portfolio that are producing plastics as well. In addition, all teams working on modeling and computation could benefit from having consistent data sets on different plastics. Are there standard operating procedures for characterization that have worked well within BOTTLE that could apply to other projects as well (for example, "Inverse Biopolymer Design Through ML and Molecular Simulation")?

PI RESPONSE TO REVIEWER COMMENTS

- Thank you to the review panel for their input on the Characterization task. In terms of "connections beyond interesting science," the work from the Characterization task overall was highlighted in multiple examples of direct impact to improve deconstruction-, upcycling-, and redesign-relevant processes and materials—indeed, these impacts were embedded in those three presentations as well (indicated by the characterization icon on the slides). These impacts have a direct impact on the potential for use, scale-up, and commercialization. The comment regarding scaled-down and accelerated testing mechanisms versus maintaining the existing ASTM methods is excellent, and we agree that we need to maintain the industry-accepted methods while also innovating for higher throughput. In terms of working with other parts of the BETO portfolio, many of the same researchers in the BOTTLE Consortium work on multiple projects resulting from FOAs, in the PABP project portfolio, and with academic collaborators (all this work is often in the same laboratory with the same equipment). Thus, there are already existing mechanisms for this transfer of knowledge, expertise, and data. We will also look to both formalize this information exchange and expand it as we can.

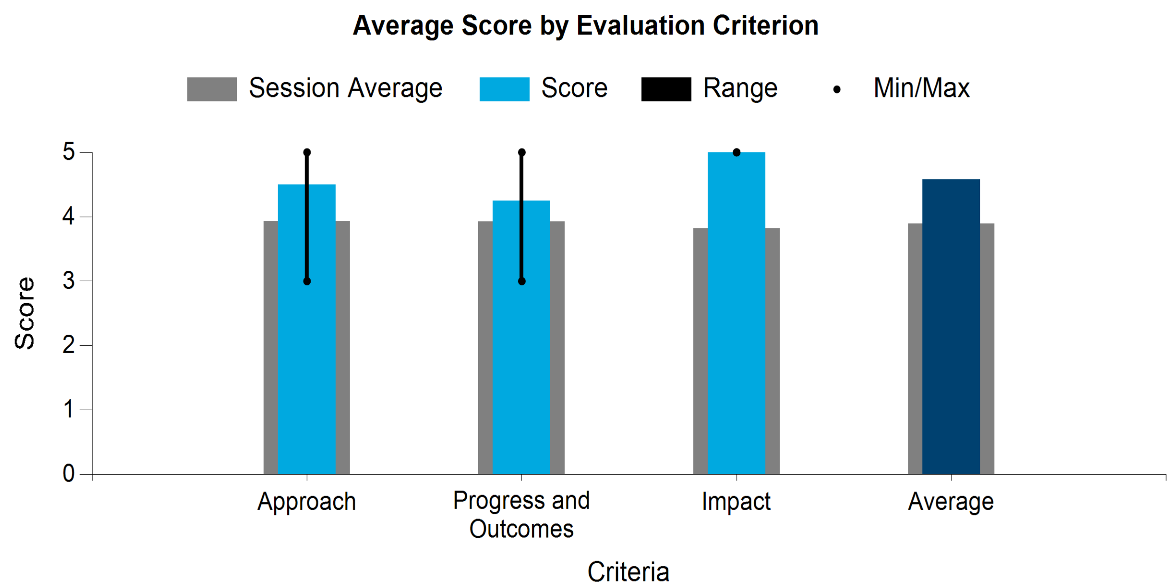
INDUSTRY PROJECTS AND ENGAGEMENT

BOTTLE Consortium

PROJECT DESCRIPTION

WBS:	BOTTLE7
Presenter(s):	Kat Knauer; Michelle Reed
Project Start Date:	10/01/2020
Planned Project End Date:	09/30/2023
Total Funding:	$150,000

One of BOTTLE's primary goals is to work with industry to catalyze new technologies toward our overall vision and mission using a centralized industry engagement plan. This plan aims to (1) solve real-world problems in plastics upcycling via targeted, company-funded projects; (2) promote industry engagement via streamlined access to BOTTLE partners and technologies; and (3) collaborate with companies to scale and deploy BOTTLE technologies into the economy. Our approach follows a five-stage model: prospecting, initial engagement, knowledge sharing, proposal and contracting, and opportunity won. Since FY 2021, BOTTLE has onboarded a full-time chief technology officer to spearhead industry engagement and lead industrially funded research projects. To date, BOTTLE has engaged with >150 companies, executed six industrial funds-in CRADAs, successfully completed two industry projects, extended two CRADAs with follow-on funds, and submitted more than 30 patent applications. Industry projects span the Deconstruction, Upcycling, and Redesign tasks; harness the capabilities of the crosscutting tasks; and take advantage of the broad IP portfolio developed through DOE funding. BOTTLE partners have an inventory of innovations that can inform the design of industry-specific collaborative projects with the highest probability of producing novel IP. Engaging with a diverse set of companies has also directly informed our R&D portfolio toward maximizing the impact and utility of BOTTLE innovations.



COMMENTS

- This is very effective at answering the impact question, so it gets full marks for what it is, but the presentation does not relate well to the other rating questions.

- Overall, BOTTLE's approach to industry engagement appears highly successful. Streamlining contracts upfront, making information easily accessible via the website, and proactively addressing staffing challenges is working. The approach of targeting consumer-facing brands with sustainability pledges is generating interest and engagement, and the belief that the brands can drive other supply chain actors to be involved should be monitored as the CRADA projects progress. Given the early-stage research at BOTTLE as well as the massive system shifts needed to deploy at scale to meet BETO targets, it is worthwhile to explore how BOTTLE can act as a convening mechanism to achieve precompetitive development with a critical mass of industry partners to create the scale needed for system shifts.

- The new chief technology officer has gotten off the ground running and is wisely using resource dollars by allocating CRADAs for established entities that invest their own dollars and reserving FOAs for investment with smaller and midsize companies that likely lack deep research organizations. It seems like a savvy and reasonable move to create market pull by building relationships with recognizable brand companies with the expectation of moving beyond these brands in the near future and deepening relationships with the polymer supply chain companies. The engagement pathway is very reasonable, with realistic estimates of negotiation times (lengthier than desirable) and obstacles to sharing knowledge by potential companies. I anticipate that if there are more successes, the current barriers will be lower—there will be a built-in community with advocacy/advertising that will promote the value of a BOTTLE alliance and accelerate opportunities.

- I believe the centralized business development position is fantastic and should be implemented in the other projects of this size as well. Kat being the central contact point for these companies helps speed up contact as well as getting to meaningful contracts. I think having a publicly available CRADA form on the website is fantastic. In terms of licensing, I strongly believe that government-funded research into recyclability should not result in exclusive licenses. The technology should be used widely and without barriers to have the strongest impact. I am very impressed that there have been two startups that have spun out of BOTTLE.

PI RESPONSE TO REVIEWER COMMENTS

- We were thrilled to see the positive reception for BOTTLE's industry engagement model, which we also think is working quite well thus far. Regarding monitoring the shift from brand owners to more upstream supply chain actors: This is an excellent point, and fortunately we have started to see the onset of this transition, with BOTTLE acting as a "nucleation point" for multiple supply chain partners. We believe we will be able to report on this transition more concretely and quantitatively in the next Project Peer Review. Regarding the comment on the "impact question … but the presentation does not relate well to the other rating questions"—we apologize for any confusion. We attempted to highlight the progress and outcomes as well as the approach explicitly in the slides. Regarding the comment on IP licensing ("I strongly believe that government-funded research into recyclability should not result in exclusive licenses. The technology should be used widely and without barriers to have the strongest impact."): National labs and universities developing recycling inventions with DOE funding must balance broad (nonexclusive) access with the need to provide returns (through exclusive licenses) to industry players who have to invest significant sums of money to bring these inventions to market. Industry partners have provided consistent feedback that they will not invest in technologies that are nonexclusively available to their competitors. Universities and national labs have robust protections in place to ensure broad impact of DOE-funded technology through license agreements, including performance requirements and clawback provisions, to ensure the technology is diligently commercialized for strong impact.

OVERVIEW, PROJECT MANAGEMENT AND INTEGRATION, AND DEI

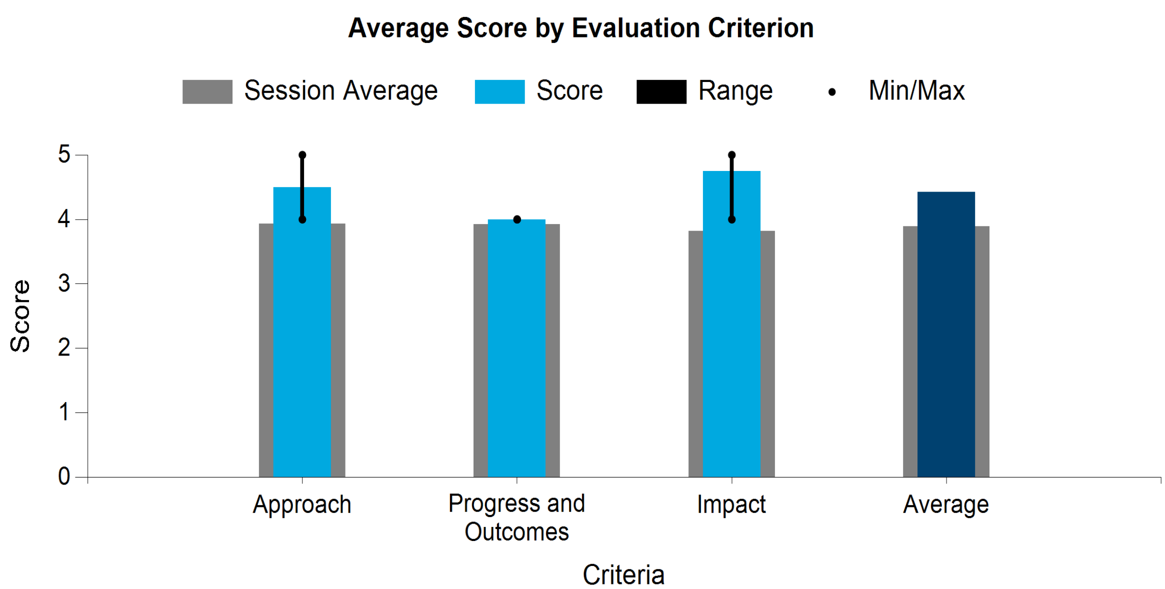
Separations Consortium

WBS:	SEP1.0
Presenter(s):	Lauren Valentino
Project Start Date:	10/01/2022
Planned Project End Date:	09/30/2025
Total Funding:	$390,000

PROJECT DESCRIPTION

Separations often account for the largest energy, economic, and environmental footprint in chemical processes, including bioprocessing. The Bioprocessing Separations Consortium brings together teams from six national laboratories to address these key challenges by advancing energy-, cost-, and carbon-efficient separations to support the decarbonization of transportation and industry. The consortium is organized into four tasks: (1) Project Management and Integration, (2) Core Experimental Projects, (3) Analysis and Computation, and (4) DEI.

The Project Management and Integration task provides technical guidance, facilitates communication among teams, tracks progress and impacts of the consortium, and guides the go/no-go decision process. This task also manages external communications, particularly with the advisory board, which provides feedback to help the consortium maintain an industrially relevant focus. A key outcome of the Project Management and Integration task is shaping the consortium's multiyear research portfolio by identifying BETO- and industry-relevant separation challenges, assessing consortium capabilities, and considering potential economic and environmental improvements. Finally, the DEI task focuses on fostering a welcoming, diverse, and inclusive environment within the consortium as well as supporting future workforce development for the biofuels and bioproducts industry.



COMMENTS

- The project is on track. The presenter gave an excellent overview of bioprocessing separations and consortia. There were clear objectives and emphasis areas for 2023. I need clarification on the IP rights for all participants. The selection of the IAB is critical, and the PI's engagement with the IAB should be strengthened. It was great to see the integration of TEA, LCA, and computation at the early stage of the project. The approach to DEI looks very strong. Is there any way to target an audience of high school students from under-resourced communities and/or within traditionally underrepresented groups?

- The Separations Consortium as a whole will advance the SOA with regard to bioprocessing. Improving the efficiency of separations specific to bio-based processes is critical for advancing the establishment of materials for transportation (fuel) and other industrial applications, including new materials. A broad range of separation technologies are being developed within the consortium. Management of the projects was standardized across the portfolio with monthly consortium meetings, biweekly TEA/LCA meetings, biannual meetings with the IAB, strong DEI initiatives, and both a laboratory executive board and an advisory board. Advisory board members come from a mix of deep technical expertise and business backgrounds and viewpoints. Implementation strategies for the technologies are less defined. It might be good to have clearly defined off-boarding objectives for commercialization so that once projects are off-boarded, new ideas can begin to be investigated. Off-boarding should be viewed as the ultimate success. Communication and collaboration among the various labs and efforts seemed to be top-notch—it appears that the LCA, TEA, and computational studies are having a significant impact on determining how the projects are executed, which is impressive. Additionally, the consortium has shown flexibility in that some projects have been rescoped to optimize the chances of success. A robust DEI plan, including the Bioenergy Bridge to Career Program with a community college focus, will increase exposure to bioenergy careers and encourage seeking candidates from underserved communities. Overall, the projects looked to be progressing on schedule. For some projects, it may be too early to have a clear connection to commercialization, but all projects had at least some contact with industry collaborators. Some projects may have too many industry voices and should consider down-selecting the number of partners at some point to provide focus.

- Separations are one of the most challenging aspects of bioproduct manufacturing, from both a technical and commercialization perspective. This is because the costs associated with separations can determine whether a bioproduct will achieve market acceptance, whether as a fuel or as a chemical. This program has identified that energy- and water-intensive processes will not allow for the successful reduction of carbon intensity for bioproducts and has identified innovative project areas that will advance the SOA. The consortium is well organized and well designed and appears to foster good cooperative work between the various laboratories. Potential areas for improvement are, first, industry collaboration. The IAB appears to meet regularly and provide good advice on the industrial application of the program efforts, but specific collaborative work with industry partners seems to be limited. A more focused effort on bringing industry collaborators on board to participate in projects would enhance the technical strength of the projects and help to ensure their relevance to practical problems. Including TEA and LCA at early stages is welcome and is an excellent way to provide metrics for assessing the value of technological developments; however, both analyses will necessarily evolve as the technology develops further through scale-up and implementation, so they should not be viewed as static scores. The sensitivity analyses being performed should help mitigate the risk of an overly optimistic early-stage analysis that does not reflect the realities as a project progresses toward implementation.

- It is fantastic that TEA/LCA is essential in focusing this project's efforts and guiding development and that the consortium meets on a monthly basis to ensure that everyone is aligned on tasks and milestones. An overall comment on the consortium: I believe that this group is at a point where the technologies are ready for the next steps in commercialization. I would suggest that the team put additional effort into business development—the BOTTLE Consortium created a business development position to centralize conversations with industry partners and licensing. I would suggest creating general templates for your CRADAs and making them available to your researchers as well as potential new partners via your website. I appreciate that the team is collaborating with other BETO portfolio efforts and industry partners. I believe that the IP on these projects should be centrally handled as well so that the researchers are not stuck with negotiating with several technology licensing offices.

PI RESPONSE TO REVIEWER COMMENTS

- We would like to express our appreciation for the thoughtful, valuable, and supportive feedback provided in the reviews. Regarding IP and business development, IP rights for any work done by the consortium are managed in accordance with the prime contract between DOE and the respective Federally Funded Research and Development Centers in the consortium. Collaborative projects may be established by any consortium laboratory and an external collaborator through a CRADA, which includes IP management. The consortium is committed to the ongoing development and availability of general CRADA templates; this includes revisiting our current CRADA development package. We aim to make the template readily available on our website. We also appreciate the reviewer's feedback on business development, but we would like to respectfully clarify that the consortium's primary focus is on technology development. Regarding the high number of industry collaborators for some projects, we believe that multiple industry partnerships bring diverse perspectives that can enhance the applicability of our projects; however, the reviewer's point about balancing collaboration with clear direction is valid. As projects evolve, we will consider strategically down-selecting the project collaborators, considering the expertise and value that each partner brings to ensure that critical perspectives are not lost in the process. We absolutely agree that the selection of the advisory board is critical because the board serves as a link between the national laboratories and industry, providing valuable guidance, expertise, and industry perspectives to researchers. Although not presented in detail due to time limitations, in 2023, we initiated an effort to update our advisory board. This selection process targeted individuals who can actively contribute to the consortium's goals and provide meaningful guidance to researchers. As of May 2023, the advisory board includes representation from the following entities: Amyris, ADM, BioMADE, AMMTO, Genomatica, Honeywell UOP, ICM Inc., New Culture, Propharma, Siemens, and Virent Inc. As noted during the presentation, the consortium meets with the advisory board twice per year and asks that board members provide advice, review results and progress in comparison with work plans, and provide feedback on the prioritization of research projects (experimental and analytical). In addition to our biannual meetings, in June 2022, we invited the board's feedback on the consortium's 3-year plan to BETO. In July 2021, we hosted a listening day to engage stakeholders on challenges and opportunities in bioprocessing separations. The consortium will continue these regular interactions with the advisory board. Regarding the target audience for our educational workshop, we have chosen to focus on community college students for a few reasons. First, we aim to provide targeted support and resources to facilitate a smoother transition from 2-year programs to 4-year institutions or the workforce. Second, community college students are a better audience for our content. Having completed a high school education, they have a foundational understanding of scientific principles and are better equipped to engage with the content. Finally, with their greater focus on entering the workforce, we seek to provide these students with specific knowledge, networking opportunities, and career guidance in the bioenergy field to enhance their employability.

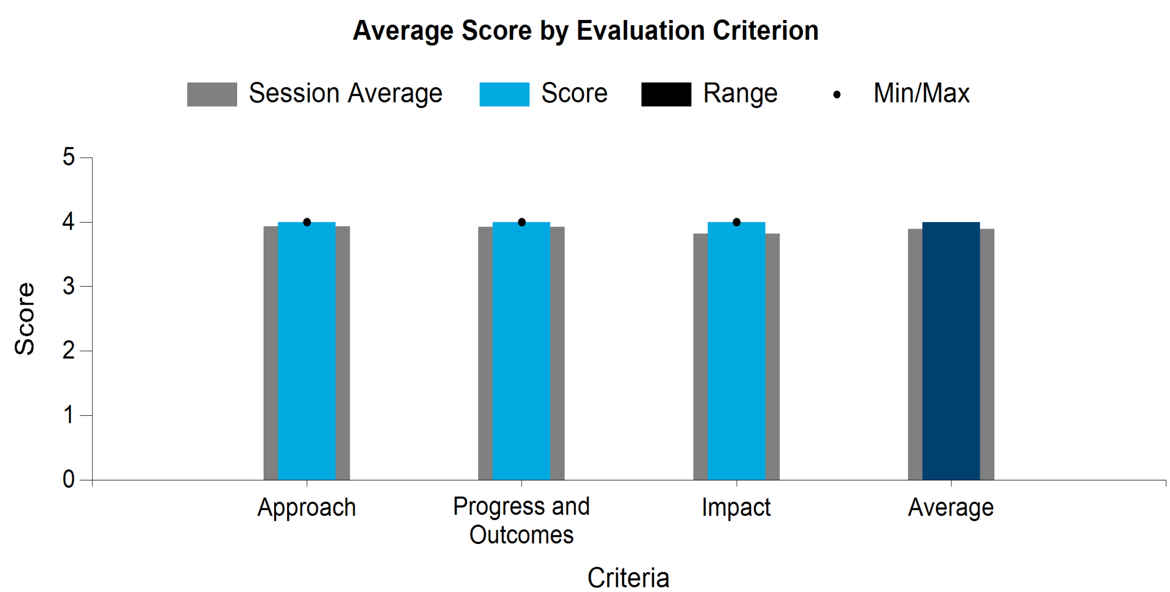
ADSORPTION BASED ISPR FOR ABF PRODUCTS

Separations Consortium

PROJECT DESCRIPTION

WBS:	SEP2.1
Presenter(s):	Gregg Beckham
Project Start Date:	10/01/2022
Planned Project End Date:	09/30/2025
Total Funding:	$500,000

This task focuses on the development of adsorption-based ISPR integrated with simulated moving bed chromatography for the recovery and purification of carboxylate products that are relevant to BETO. ISPR has been pursued previously in the Separations Consortium to recover carboxylic acids near or below their pKa values with liquid-liquid extraction coupled with downstream distillation; however, there are many acid products in the BETO portfolio that require neutralization well above their pKa values wherein ISPR could still be a major benefit to the bioprocess performance, including muconic acid, beta-KA, 3-hydroxypropionic acid, itaconic acid, butyric acid, and others. In this task, we are combining dynamic filtration with a rotating ceramic disk, resin capacity measurements, tailored resin synthesis, and simulated moving bed chromatography into an ISPR system that can be used to recover BETO-relevant carboxylates from bioreactor cultivations. We are working across process scales and using computational modeling where applicable alongside TEA and LCA to understand major cost, energy, and GHG emissions drivers. The impact of this project will be a bench-scale integrated approach to recovering carboxylate products *in situ*, which will reduce the waste generation from biological carboxylate production processes and improve the productivities of biological systems.



COMMENTS

- The project is on track. The team is developing an ISPR system for the recovery of muconic acid, aconitic acid, 3-hydroxypropionic acid, and beta-KA. Is there any feedback from the IAB on the approach and scalability of this ISPR system? The team needs a strong and detailed DEI plan.

- The success of this project depends on a high level of collaboration with other BETO projects, and it looks as if this is happening. The project started in October 2022, so it is still quite early. Risks and mitigation strategies are sufficiently defined. Is this equipment inherently difficult to keep up and maintain? It would be good to get industry feedback on this. The Quarter 2 milestone of collecting data on the uptake of various acids on the resins was achieved. Industry partners can help refine the project objectives and provide information on their relevance. The slide on nanostructured adsorbents is quite interesting, and it can help tailor the filtration of specific acids. The DEI plan with five interns (are they from underrepresented groups?) and participation in the Bioprocessing Separations Consortium is sufficient for the DEI objective.

- This new project area has the potential to provide an innovative set of technical solutions to the separation problems that reduce the rate of adoption of new bioproducts. The approach aimed at reducing the number of unit operations in the downstream processing of bioproduct manufacture is well thought out and has the potential to significantly advance the SOA in the topic area. Collaborations among the laboratories appear to be well managed and well considered. The progress is impressive given the recent start date of the project. The primary technical risk is that the metrics for the final product qualification are not universally understood. Purity requirements are defined by the end-use application, so assessing the performance of the quality of a separation process may not capture the specific requirements of the end use. This could be improved with more industry involvement for specific project areas that would allow for input on performance requirements. The scope of the project is broad, with many different separation techniques and targets. The use of proxies at this stage is necessary, but the team needs to ensure that translatability across target molecules is maintained. For example, data generated for lactic acid may not be universally applicable when the target molecule changes.
- I appreciate that the team is targeting scales of 10 milliliters all the way up to 100 liters. I greatly appreciate that the team is working with other BETO projects and industry partners to select high-impact targets to focus on. It is very impressive that the team has three CRADAs, one with each industry partner. I would want to see continued collaboration with industry partners in picking high-value targets.

PI RESPONSE TO REVIEWER COMMENTS

- We thank the reviewers for their positive feedback. We are committed to supporting a sustainable bioeconomy through advancing the recruitment, development, and retention of a diverse and talented workforce. In addition to participating in each lab's DEI plan, including individuals from underrepresented groups in our research team, and providing student internships, the consortium is supporting future workforce development in bioenergy technology fields by creating the Bioenergy Bridge to Career Program. This was presented in the overview presentation for the entire Separations Consortium. We emphasize that the project overall has an exceptionally strong DEI plan. Our equipment is not difficult to maintain, but we wanted to make the point that we have dedicated staff who are experts in its use and upkeep. We find that having dedicated staff to maintain specialized equipment makes the collection, quality, and efficiency of data more consistent, and we were merely attempting to convey that we have been able to staff the project with dedicated experts to focus solely on this effort for maximum impact. In terms of purity requirements, we are working with the PABP projects and industry partners to understand the purity requirements, e.g., for the condensation polymerization of diacid products. We acknowledge the enthusiasm of the reviewers for industry engagement, and we will continue to emphasize this in our R&D activities. The consortium also meets with our advisory board twice per year to gather feedback on the potential commercial viability of all consortium technologies, and we will continue to integrate this into our approach.

CO-OPTIMIZATION OF SCALABLE MEMBRANE SEPARATION PROCESSES AND MATERIALS

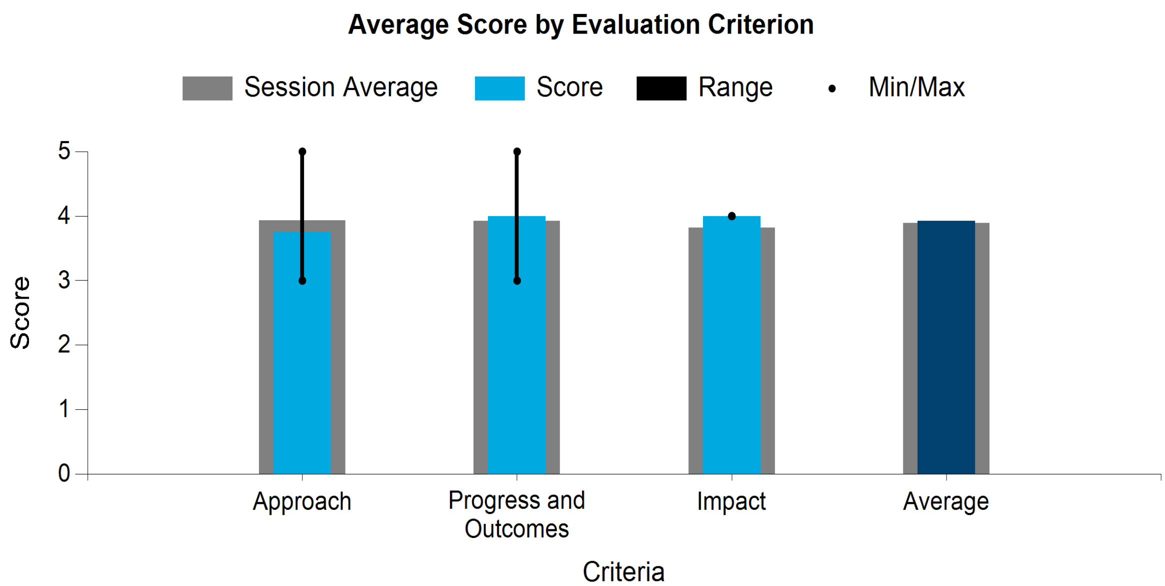
Separations Consortium

WBS:	SEP2.2
Presenter(s):	Meltem Urgun Demirtas
Project Start Date:	10/01/2022
Planned Project End Date:	09/30/2025
Total Funding:	$385,000

PROJECT DESCRIPTION

Membranes can reduce the energy, carbon, and space intensity of traditional separation processes (i.e., distillation and other thermal-based processes) for organic solvents. Specifically, organic solvent nanofiltration (OSN) and organic solvent reverse osmosis (OSRO) are pressure-driven technologies that require less energy than a phase change; however, owing to similar chemistries and properties among commercially available nanofiltration and reverse osmosis membranes (the majority of which are designed for aqueous-based separations), these thin-film composite membranes are highly susceptible to organic solvent degradation, limited permeance of hydrocarbons, and operational problems, including fouling. This task will focus on simultaneous membrane material and process development, targeting a TRL of 4, for OSN/OSRO. Industry input highlighted the need for high-flux membranes with sharp separation cutoffs. At the same time, manufacturing methods must be based on scalable synthesis reactions. In addition to the characterization of commercially available and surface-modified membrane materials, this task will address gaps related to process engineering and scale-up, with the overall goal of transitioning membrane technology into more challenging separation environments.

By project end, this task will demonstrate scalable membrane surface modification techniques and evaluate membrane performance at the pilot scale. Argonne National Laboratory's roll-to-roll facility will be used to scale up the surface-modified membranes. The membranes will be evaluated in a pilot-scale (140 cm^2) membrane unit using real bio-oil and biocrude samples.



COMMENTS

- There has been great progress in this review period. The project is on track. The team engaged membrane manufacturers, technology developers, and end users; integrated TEA and LCA; and demonstrated scalable surface modification techniques using Argonne's roll-to-roll facility. The coating of parts was not clearly described in the presentation. There was a clear plan on DEI initiatives. The team should continue to work with their industry partners, especially on the TEA and LCA sections at the beginning of their journey.

- The team met with a number of relevant companies to refine the scope of the project—this is great. There is a very strong DEI component for the project, including a diverse team, outreach activities, two interns/years from underrepresented groups, preferred vendor and procurement from disadvantaged-owned businesses, and Bridge to Career program participation. The project is just getting underway but holds promise in replacing high-energy distillation with OSN/OSRO. Current challenges are the lack of material strength and performance stability and fouling. The project leaders intend to improve these issues with surface modifications of the membranes for performance and durability. The team has already acquired commercial membranes, set up bench-testing units, and discussed LCA/TEA pathways with the analysis team. The method has the potential to make an impact in bioprocessing separation.

- This early-stage project has a valid technical approach to solve complex separation problems with an innovative plan. It was not clear where the new membrane technologies will be targeted and how that targeting should inform the overall technical effort. The risks associated with scale-up and operability seem to be addressed, but without better target information, it is difficult to assess the mitigation strategy. The project seems to be more focused on membrane fabrication than on the separation problem, which may reflect the TRL of the project more than the technical approach. The project's starting TRL is listed as 3, with a target of advancing the TRL to 4. It appears that the starting TRL may be earlier. It is critical to have an accurate TRL in order to assess progress.

- The team could reach out to Via Separations (https://viaseparations.com/technology/), a membrane-based startup out of the Massachusetts Institute of Technology that has done extensive research into what industrial streams exist and are currently not separated by membranes. I would encourage the team to test the stability of their novel systems at the temperatures at which they will run in the industrial system. A lot of polymeric-based membranes fail at these elevated temperatures and have limited operating lifetimes. If possible, I would suggest that the team build a model where potential partners with separation problems can see which one of the team's membranes would be useful.

PI RESPONSE TO REVIEWER COMMENTS

- We thank the reviewers for taking the time to review our work. We appreciate their supportive comments on our DEI and industry engagement efforts. We greatly appreciate this recognition and encouragement of our technical plan, as well as the recommendation of the separation membrane company, Via Separations. To clarify, the focus of this project is to address both membrane material development and process engineering innovations. By addressing both aspects simultaneously, we aim to achieve synergistic improvements that can lead to enhanced performance, efficiency, and cost-effectiveness. As acknowledged by the reviewers, this is a very early-stage project. Initial efforts in Year 1 are focused on conducting experiments to evaluate the SOA in OSN applications. This systematic assessment, along with a literature review, will identify the challenges, research gaps, and potential solutions for organic solvent separation using existing commercial membranes, and it will guide the team's selection of the best technical solutions for modifying and/or functionalizing the commercially available membranes to enhance their performance. Meanwhile, the team will also explore emerging alternative membrane materials and designs that can surpass the commercial membranes. For example, a membrane with an ultrathin hydrophobic polyamide skin layer will achieve significantly higher permeance and selectivity. Regarding the targeted applications, we have reached out to industry to identify specific applications where membrane technologies can provide significant benefits. We identified OSN as a result of this industry outreach. We are actively working to characterize the SOA membrane materials and will continue to engage with industry experts as performance results become available to further narrow the specific application within the biorefinery. We also recognize that different applications may have unique requirements, including operating temperature. Our current membrane test cell can be operated up to 121°C, so our team is prepared to conduct experiments at elevated temperatures if needed. In addition, the team has experience in fabricating polymeric materials that can withstand higher operation temperatures (e.g., up to 300°C). We will leverage this knowledge for our customized membrane development within this project.

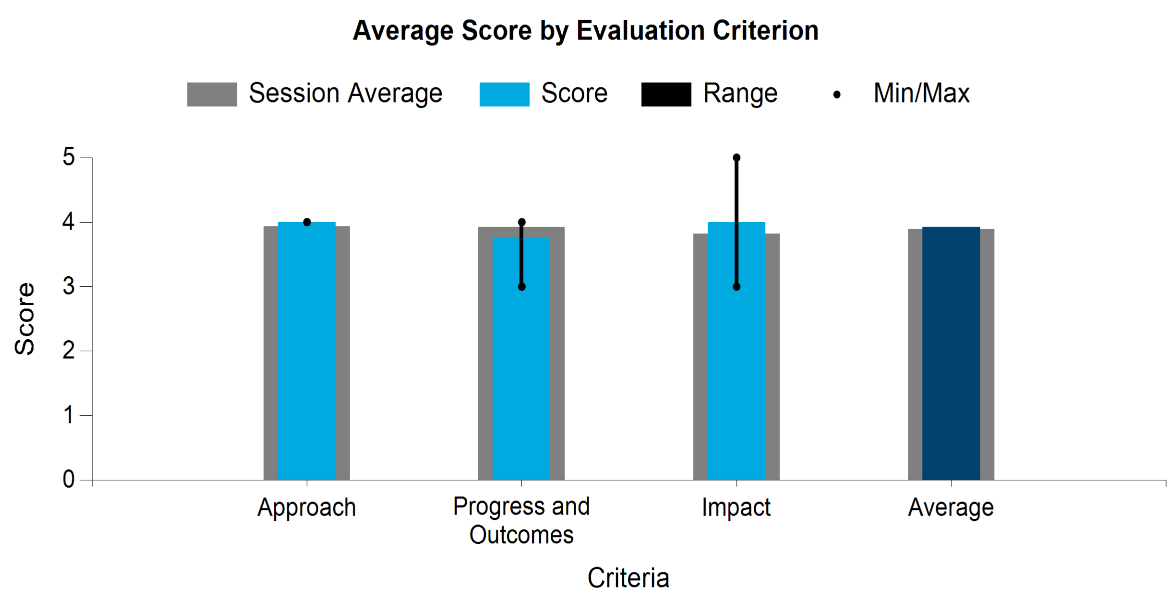
CONTINUOUS COUNTER CURRENT CHROMATOGRAPHY

Separations Consortium

PROJECT DESCRIPTION

WBS:	SEP2.3
Presenter(s):	Gregg Beckham
Project Start Date:	10/01/2022
Planned Project End Date:	09/30/2025
Total Funding:	$650,000

This task in the Separations Consortium focuses on the development of CCC, which is an advanced liquid-liquid chromatography method. Today, CCC is primarily practiced in batch mode operation, but continuous operation will be required for at-scale deployment in a biorefinery setting. In this task, we are developing a continuous CCC process in collaboration with a company that builds CCC units. This work will be demonstrated on the biorefining challenge of lignin valorization. Specifically, we demonstrate the use of continuous CCC for both monomer-monomer and monomer-oligomer separations in multiple lignin streams of relevance to the biorefining industry. Breakthroughs in this area would be useful for BETO goals in both SAF and biochemicals production, including directly contributing to BETO's 2030 lignin valorization goal. Our approach includes developing new computational modeling approaches to optimize both batch and continuous CCC processes, conducting experimental work to determine optimal solvent systems for multiple lignin streams sourced from BETO-funded projects and industry collaborators, and using TEA and LCA to identify the most impactful areas to ultimately enable this approach in the biorefinery. This task overall will enable high-resolution, multicomponent, continuous separations at scale, demonstrated on a grand challenge biorefining problem.



COMMENTS

- The team focused on lignin monomer-monomer and monomer-oligomer separations with continuous CCC up to the kilogram scale. There has been some progress in this review period. The project is on track. There is great collaboration among NREL, Dynamic Extractions, and Lignolix. I am not sure that this continuous CCC has the potential to create wider and more significant impact. The team should seriously think about feasibility and scalability. The PI needs to develop a DEI strategy and show the progress in the next review period or at the end of the project.

- It is early in the project—the project was initiated October 2022. There is a good DEI plan with five undergraduate internships (hopefully some going to underrepresented groups) and participation in Separations Consortium DEI activities. The risk mitigation plan is good for the project, including hiring a technician for CCC maintenance (keep an eye out for ways to reduce processing maintenance for scale equipment). There is a solid technical approach and risk mitigation. Progress has been made in identifying lignin streams for testing, computational modeling for process optimization, and defining solvents for extractions. Thus far, predictions from the model match experimental profiles. The team is working closely with an industry partner that is interested in enabling continuous CCC, which will increase the odds for eventual commercialization.
- This is an early-stage project that, if successful, could provide access to a broad range of new molecules useful for either fuel or chemical product applications. The project approach is sound and makes good use of computational methods to reduce risk and facilitate more rapid development. Although there is a good strategy to identify and mitigate risks, the risks associated with scale-up may not be adequately captured. For early-stage projects such as this one, I think that articulating the vision for success through commercial deployment has value, even if it can only be conceptual at this stage of the technology development. In this case, there has been a great deal of progress on the basic separation, but a clear statement of what this process might look like at a full-scale biorefinery would have value for understanding and assessing the approach. The project shows good collaboration with both lab and industry partners.
- I appreciate that the team is collaborating with Dynamic Extractions to develop a continuous CCC process. I appreciate that the team is using real-world lignin oils from its partnering BETO projects.

PI RESPONSE TO REVIEWER COMMENTS

- We appreciate the reviewer feedback. All project teams within the consortium are engaging in the following DEI activities: (1) including individuals from underrepresented groups in STEM as researchers, (2) leveraging ongoing programs at each national laboratory to provide student internships, and (3) participating in diversity-focused education and outreach programs. The project team will join researchers and educators from across the consortium laboratories to educate and support the next generation of bioenergy students and researchers through the Bioenergy Bridge to Career Program. This DEI plan was presented by the Separations Consortium PI on behalf of the entire consortium in the overview presentation. In terms of the feasibility of CCC, we stress that this is an early-stage technology, as was mentioned in the presentation. For a frame of reference, a similar method to CCC, centrifugal partitioning chromatography, is now being scaled industrially (but it typically delivers lower product yields and purities than CCC, which is why we are not using that approach). In line with the high-risk, high-reward R&D that is currently at low TRL, to our knowledge, no other separation method available today enables facile separations of multiple target compounds outside of hydrocarbon separations through distillation, which is not appropriate for most biomolecules due to their high oxygen content. A similar reviewer comment was made that the “risks associated with scale-up may not be adequately captured”—this is one of the main reasons we are working with an industry partner through a CRADA on this project. This ensures that we have a direct line of sight to scale up for CCC when we are able to make the system continuous in nature. We will also soon publish a detailed process model of CCC integrated into a full-scale biorefinery to show a clearer picture of how this could fit into an overall process.

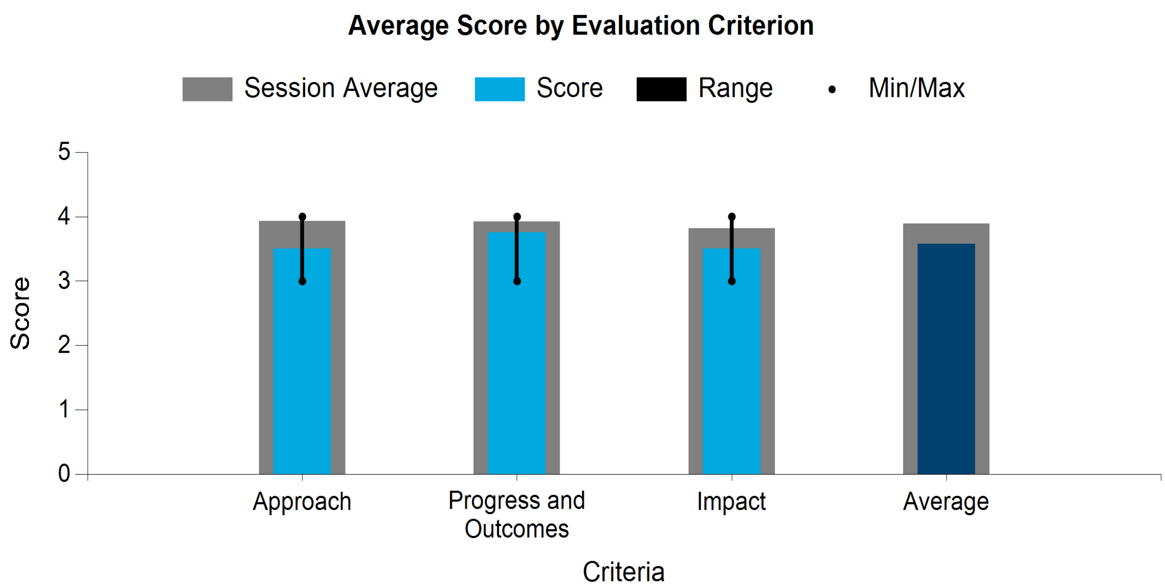
DIOL SEPARATIONS

Separations Consortium

WBS:	SEP2.4
Presenter(s):	Ramesh Bhave; Syed Islam
Project Start Date:	10/01/2022
Planned Project End Date:	09/30/2025
Total Funding:	$400,000

PROJECT DESCRIPTION

2,3-butanediol (BDO) is gaining attention in the global market as an intermediate product for several applications, such as liquid fuel. It can be produced through fermentation in low concentrations (<10 wt %). A common commercial separation/concentration technology for BDO involves evaporation and multistage distillation, both of which are energy-intensive. The goal of this work is to develop low-cost and energy-efficient separation technologies—supported by TEA, LCA, and computational modeling—to recover BDO from fermentation broth. To achieve this goal, we are developing a membrane-assisted liquid-liquid extraction-based process to separate BDO from fermentation broth. The membrane contactor provides a high surface area for intimate interfacial contact of the aqueous and organic phases. It is a single-step continuous process. Its modular design allows for linear scale-up. Further, it offers several other advantages, including low chemical usage, low waste generation, low energy consumption, and low capital and operating cost. Moreover, it prevents emulsion formation. Organic solvent oleyl alcohol and 1-Hexanol have been identified as the potential solvents to extract BDO with a yield of >95% 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 an aqueous solution using hexanol in a continuous mode of operation.



COMMENTS

- The team explored three new approaches prior to selecting an approach for experimental development. The project is on track, and it seems that the team successfully changed their direction. Quantum and classical mechanics will help the screening and identification of proper solvent candidates. The team identified porous hydrophobic polypropylene hollow fibers as a membrane contactor to separate BDO from aqueous media using hexanol. The PI did not present their DEI strategy and progress.

- No DEI plan was provided. The project aims to separate BDO from fermentation broth with high efficiency and low energy impact. The project started in October 2022, so it is very early. The project has successfully changed direction in response to previous reviewer comments. The risks of general BDO separations were outlined, but risks and mitigation plans for the new process were not specifically outlined in the presentation. The team has achieved progress in identifying two potential solvents, hexanol and oleyl alcohol, through modeling. Are other solvent candidates being considered? It was unclear how the team settled on those two solvents. PP hollow fibers were selected as the microporous membrane, but I am wondering if polyvinylidene difluoride (PVDF) fibers should also be tried because of their increased chemical resistance and thermal stability. The project is on target to achieve its goals. The team should increase their engagement with industry partners within the next year.

- This project has made significant progress since the change in approach developed after the last Project Peer Review. The redirection and improved focus have resulted in what appears to be an initial demonstration of a practical, cost-effective means of separating 2,3-BDO from a dilute fermentation broth mixture, a technical problem that has resisted solution. Validation by an industry partner would improve the impact of the project and better demonstrate that the technical achievements illustrated to date have merit. The technology appears to have a valid pathway to scale-up and makes good use of computational methods to mitigate risk and speed up the development cycle in the solvent selection. More work needs to be done on the solvent recovery portion of the project, but the technical risks associated with that element of the project are low.

- I appreciate that the team is realizing the suggestions from the computational team to use hexane for the separations. It is always great to see computational models making suggestions that can have a real-world impact. The team should work closely with feedstock partners and offtake partners to ensure that their approach is relevant to industry, and they should check their assumptions in the TEA/LCA.

PI RESPONSE TO REVIEWER COMMENTS

- Comments: The team explored three new approaches prior to selecting an approach for experimental development. The project is on track, and it seems that the team successfully changed their direction. Quantum and classical mechanics will help the screening and identification of proper solvent candidates. The team identified porous hydrophobic polypropylene hollow fibers as a membrane contactor to separate BDO from aqueous media using hexanol. The PI did not present their DEI strategy and progress.

- Response: The PIs thank the reviewer for the positive comments on the technical achievement. Regarding DEI, the consortium is supporting future workforce development in the bioenergy technology field by hosting the Bioenergy Bridge to Career Program. Researchers from across the consortium laboratories, including this project team, will participate in educating the next generation of bioenergy students and researchers. This was presented in the overview presentation for the entire Separations Consortium. We also strive to include individuals from underrepresented groups in our research team and provide student internships. To that end, we have made an offer for a postdoctoral research associate position to a woman scientist to promote the diversity and inclusion of underrepresented groups in STEM. Additionally, we are planning to host an intern from a local community college (e.g., Pellissippi State Community College).

- Comments: No DEI plan was provided. The project aims to separate BDO from fermentation broth with high efficiency and low energy impact. The project started in October 2022, so it is very early. The project has successfully changed direction in response to previous reviewer comments. The risks of general BDO separations were outlined, but risks and mitigation plans for the new process were not specifically outlined in the presentation. The team has achieved progress in identifying two potential solvents, hexanol and oleyl alcohol, through modeling. Are other solvent candidates being considered? It was unclear how the team settled on those two solvents. PP hollow fibers were selected as the microporous membrane, but I am wondering if polyvinylidene difluoride (PVDF) fibers should also be tried because of their increased chemical resistance and thermal stability. The project is on target to achieve its goals. The team should increase their engagement with industry partners within the next year.

- Response: The PIs thank the reviewer for the positive comments on the technical achievement. In terms of risk mitigation: One of the risks of the proposed process is the identification of a hollow fiber contactor with appropriate properties, such as wettability and pore size. We have successfully identified porous polypropylene hollow fibers. The preliminary results showed that the membrane contactor can bring the aqueous and the organic phase together without emulsion formation. After establishing the proof of concept, we plan to investigate the long-term performance process in a continuous mode of operation. Regarding solvent selection: Based on our preliminary investigation from molecular dynamic simulations and basic properties of organic solvents reported in the literature, we have identified two solvents; however, we plan to investigate other solvents with the support of molecular dynamic simulations. Regarding the hollow fiber selection, we have past experience in porous PP hollow fibers, as they have small pore size with high surface area, have hydrophobicity, and are relatively inexpensive. The PIs thank the reviewer for suggesting PVDF hollow fibers. We plan to identify and evaluate PVDF hollow fibers with comparable properties to PP as we progress with the project, especially processing real-life fermentation broth. After establishing the proof of concept in the first year of the project, we think it will be more appropriate to engage with industry partners.

- Comments: This project has made significant progress since the change in approach developed after the last Project Peer Review. The redirection and improved focus have resulted in what appears to be an initial demonstration of a practical, cost-effective means of separating 2,3-BDO from a dilute fermentation broth mixture, a technical problem that has resisted solution. Validation by an industry partner would improve the impact of the project and better demonstrate that the technical achievements illustrated to date have merit. The technology appears to have a valid pathway to scale-up and makes good use of computational methods to mitigate risk and speed up the development cycle in the solvent selection. More work needs to be done on the solvent recovery portion of the project, but the technical risks associated with that element of the project are low.

- Response: The PIs thank the reviewer for the positive comments. We will work on the solvent recovery process with the support of the computational team. The TEA/LCA work is in progress to evaluate the trade-offs for using hexanol versus oleyl alcohol. The computational team uses an ML approach and molecular dynamics simulation to identify organic solvent that has high affinity toward BDO. Previously, the computational team investigated the effectiveness of hexanol. Currently, the team is running a molecular dynamics simulation on oleyl alcohol as the organic solvent based on the thermodynamic properties, including BDO solubility and free energy. Lawrence Berkeley National Laboratory also performed a wiped film evaporation with hexanol and BDO and successfully recovered the hexanol. They plan to perform a wiped film evaporation for the separation of BDO from oleyl alcohol.

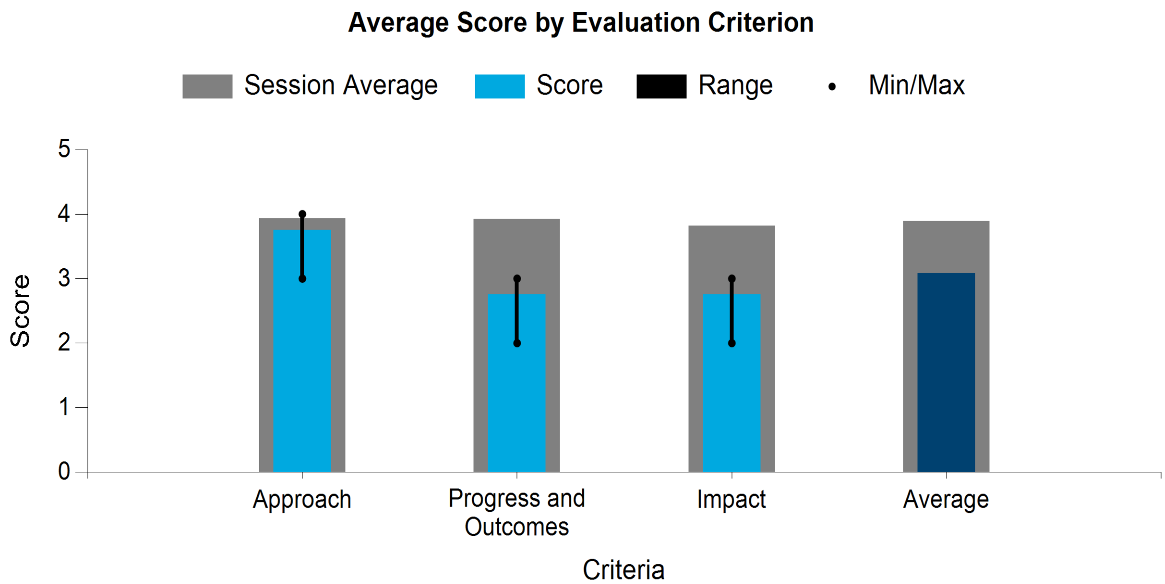
- Comments: I appreciate that the team is realizing the suggestions from the computational team to use hexane for the separations. It is always great to see computational models making suggestions that can have a real-world impact. The team should work closely with feedstock partners and offtake partners to ensure that their approach is relevant to industry, and they should check their assumptions in the TEA/LCA.

- Response: The PIs thank the reviewer for the positive comments. We plan to work with feedstock partners and offtake partners. Our team members have experience working with feedstock producers. We intend to work with more closely with these partners as the project progresses.

ELECTROCHEMICAL SEPARATION TECHNOLOGIES TO EXTRACT INTERMEDIATE ORGANIC COMPOUNDS

Separations Consortium

WBS:	SEP2.5
Presenter(s):	Yupo Lin
Project Start Date:	10/01/2022
Planned Project End Date:	09/30/2025
Total Funding:	$400,000



COMMENTS

- The project is on track. The team is trying to separate and recover medium-chain carboxylic acids from bioconversion streams for SAF and/or biochemical production using electrochemical separations. The previous and planned DEI activities look good. The team should address the main challenges (cost, process efficiency and selectivity, and the complexity of short- and medium-chain carboxyclic acids (SCCA and MCCA, respectively)) with electrochemical separation technologies. Is the process powered by renewable energy?

- It is very early in the project—the kickoff was in October 2022. The project has one of the strongest DEI plans, with four undergraduate interns, a separation activity for grade seven, participation in "Introduce a Girl to Engineering Day," and participation in Science Careers in Search of Women. Kudos for this! The project aims to design, fabricate, and operate a scalable shock wave electrolysis stack and scale a capacitive deionization system (by 10 times) to recover medium-chain carboxyclic acids from bioconversion streams. There are advantages to the electrochemical separation technique, including reduced fouling and resistance and lower GHG and use of water. Progress to date includes a shock wave electrolysis stack that has been successfully operated. Reversible electrosorption using capacitive deionization has also been demonstrated. The technology could be important in eliminating the fouling and transport resistance that is experienced with other membranes, with lower environmental impact.

There are good partnerships set up with the Agile BioFoundry and the industrial company Visolis to ensure the success of the project.

- This project would greatly benefit from some conceptual engineering to better illustrate a vision for how this technology could be deployed at scale. The pathway for commercialization is very unclear, and without some techno-economic guidance, the benefits of the approach are not clear. If we assume that the vision has been developed and the preliminary costs are defined, the approach appears to be valid and well thought out. The project is organized and well managed and shows good collaborative efforts across the labs. The project milestone states that the intent is to develop a "game-changing" technology for separations that, if successful, would have a tremendous impact on the bioproducts industry. The articulated risks are incomplete given the nascent state of development for this process and should be expanded, along with a more complete description of the mitigation strategy.
- The team is planning to do its TEA in the third year of the project. I would suggest that the team test the economic and environmental viability of the technology as soon as possible. This is a very early project, and the team is currently running their experiments with a potassium chloride salt solution. I would love to see this system use a more application-relevant feed stream to reach the goal of using real bioprocessing streams in the timeline of this project. To this end, the team should work with industry partners to source feed streams and enter into conversations with industry partners on the offtaker side. I believe that the self-assessment of TRL 3 at the project start is too high when the team is still using a model stream (potassium chloride salt solution) at bench scale.

PI RESPONSE TO REVIEWER COMMENTS

- We sincerely thank the reviewers for their thoughtful reviews and positive feedback. We appreciate the recognition of our efforts related to DEI. This positive feedback validates our ongoing commitment to promoting an inclusive environment, driving innovation, and achieving long-term success. We recognize the importance of addressing cost considerations to ensure the feasibility and adoption of these technologies. Previous internal consortium analyses indicated that the economic feasibility of another electrochemical technology (resin wafer electrodeionization) is comparable with that of other consortium technologies. As experimental performance results are generated in this task, we will feed these data into both techno-economic and sustainability analyses. We will explore potential scenarios in which electrochemical separations are powered by renewable energy to evaluate the cost and sustainability implications. Regarding process efficiency and selectivity, continuous R&D efforts are dedicated to refining these technologies to achieve higher efficiency and selectivity in the electrochemical separation processes. The progressive separation efficiency targets are listed in each milestone as measurement metrics. We fully understand the significance of conceptual engineering in providing a vision for the deployment of electrochemical separation technologies at scale. Although these are early-stage technologies, as mentioned during the presentation, slide 3 illustrates the biorefinery block flow diagram. We will work to develop a more detailed process flow diagram illustrating the key steps, equipment, and material flows involved in the electrochemical separation processes. The process flow diagram will serve as the basis for TEA to provide insights into the commercial viability of these technologies. Engaging with our industry partner, a potential end user, will help validate the technology's value proposition and guide a potential commercialization strategy. Regarding the risks, shock wave electrolysis has been technically demonstrated by Martin Bazan's group at the Massachusetts Institute of Technology, resulting in more than a dozen published articles for water desalination applications. Similarly, capacitive deionization is used at the industrial scale for water treatment but has not been demonstrated for biorefinery applications outside of the consortium. As noted in the Consortium Overview and Analysis task presentation, TEA and LCA are integrated throughout the R&D cycle to identify potential challenges and uncertainties. We agree with the reviewers that it is important to work with process-relevant streams; however, as these technologies are at an early stage, we are using synthetic mock solutions to provide initial insights into separation mechanisms, helping us understand the underlying science and optimize the technologies. As the work progresses, we will use mock solutions of increasing complexity and real-world process streams in our separation experiments.

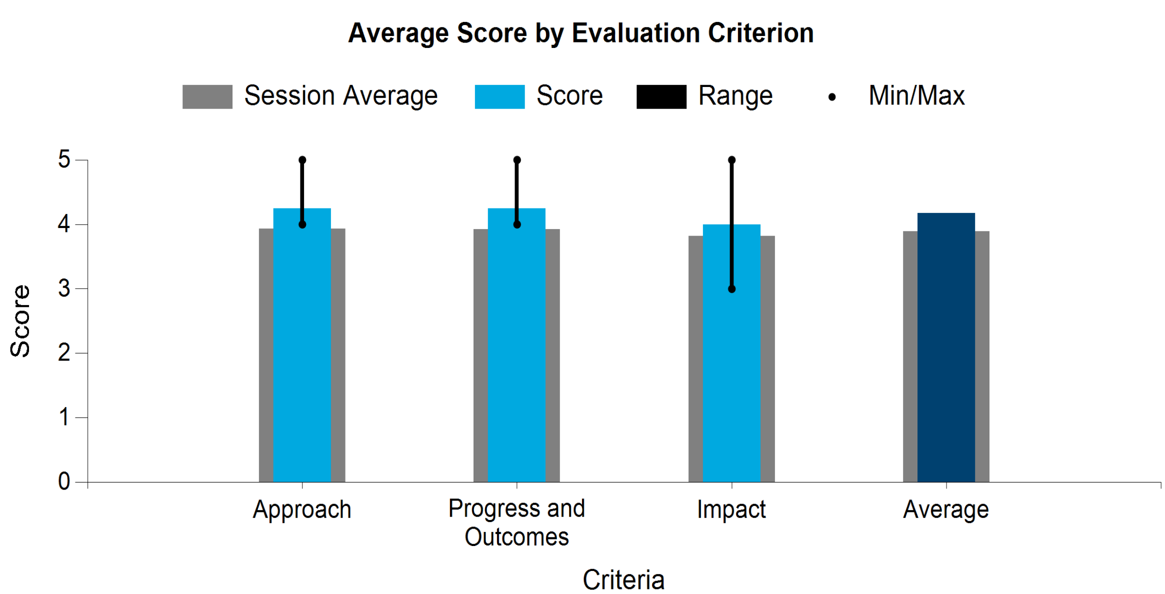
ENABLING SAF PRODUCTION BY ADSORPTIVE DENITROGENATION

Separations Consortium

WBS:	SEP2.6
Presenter(s):	Daniel Santosa
Project Start Date:	10/01/2022
Planned Project End Date:	09/30/2025
Total Funding:	$400,000

PROJECT DESCRIPTION

Hydrothermal liquefaction (HTL) of biocrude from wet waste biomass such as algae, manure, food, and sewage sludge inherently contains high amounts of nitrogen due to the starting protein content. Thus, nitrogen in hydrotreated biocrude includes a large variety of nitrogenated compounds, such as amides and amines, but also non-basic and more refractory indoles that require removal to achieve a <2-parts-per-million nitrogen level. This is a critical step to obtain ASTM approval of the wet waste HTL pathway for producing SAF. The HTL pathway can produce 3.9 billion gallons/year of SAF (>20% of the U.S. aviation demand in 2019) from wet wastes while reducing GHG emissions by >70% at a projected selling price of $3.15/gallon gasoline equivalent (2022); however, the current nitrogen removal method requires a severe hydrotreating step and consequently harsher operating conditions (increased temperature and pressure and/or lower space velocity) with increased H_2 requirements. This results in yield losses from undesired hydrocarbon cracking, and not many refiners have this capability. Alternatively, adsorbent development can be tailored for high selectivity toward small and refractory nitrogen molecules in complex biomass-derived liquids, coupled with regenerability and minimal yield loss. This technology is projected to be cost-competitive with the current SOA hydrotreating. In addition, this process requires no H_2 input and can improve the sustainability of refinery processes that adopt an HTL pathway. In this work, we will demonstrate an engineered sorbent system with tailored pore size, specific surface area, and selectivity targeted for high affinity and capacity for the sorption of target nitrogen molecules in a surrogate jet fuel, resulting in <2-parts-per-million nitrogen fuel. Future work will also demonstrate the selective adsorption of nitrogen-containing species from hydrotreated biocrude as well as minimal yield loss by continuous regeneration and lower carbon intensity due to lower hydrogen consumption.



COMMENTS

- The project is on track. There is huge potential to utilize 76 million tons/year of wet waste in the United States for conversion to ~400 thousand barrels per day SAF (~25% U.S. jet fuel demand). Are there any requirement(s) or limitation(s) for the incoming materials (wet waste) to optimize value from other HTL streams? TEA/LCA of an additional purification step using this method is critical. The PI needs to develop a DEI strategy and show the progress in the next review period or at the end of the project.

- The project started in October 2022, so it is in the very early stages. The DEI plan is multifaceted and includes three outreach activities, one student intern, and participation in Bioenergy to Bridge Program. The team has consulted with refineries to understand their concerns with adsorptive denitrogenation and gain their support, increasing the chances of project success. A good amount of data generated shows that Amberlyst resins have potential. The team is on track to down-select the most promising adsorbent materials. In summary, this is a solid project to remove nitrogen in HTL to produce lower-impact SAF.

- This is an impactful project that has shown significant potential to improve the manufacturability of SAFs in an industrially practical manner, with low energy inputs and a readily available adsorbent system that is scalable. A potential future technical challenge relates to the variability of wet waste and the impact of that variability on the separation technology employed here. Additionally, the successful use of a model biocrude, while valuable, could lead to underestimating the technical risk associated with a real-world stream that is variable. The state of the interaction with industry partners was also hard to ascertain. Some are listed on the collaboration slide, but their contributions and how they may be involved in the projects was not transparent from the presentation. Overall, the project is organized and well managed with a well-described technical approach.

- I would suggest that the team start using real-world wet waste and increase the capacity of the hydrotreated biocrude so that they can run this on more realistic samples. I would like the team to work with an offtake partner to confirm that its final fuels meet SAF requirements.

PI RESPONSE TO REVIEWER COMMENTS

- First, we thank the reviewers for taking their time and providing their thoughtful and insightful comments. Regarding the requirement for HTL, the current work on wet waste pretreatment for HTL includes dewatering to 25% or 20% and grounding/different feedstock mixing to make the stream homogeneous for pumping. Experimental tests are being conducted to de-ash the feedstock to avoid the potential engineering challenges of dealing with solids in the continuous process, such as plugging, fouling, vibration, and mechanical stress on the equipment. We believe that no additional pretreatment steps are needed to control the nitrogen content or make the adsorption technology suitable for denitrogenation, provided that the adsorbent can work for high nitrogen content as handled by hydrotreating. Our initial screening shows that candidate adsorbents have been proven to be able to work with high nitrogen content (up to 2,000 parts per million) in our simulated feed. The TEA and LCA effort will focus on identifying the adsorbent system with both the lowest environmental impact and the lowest capital and operational expenses. As for the key efforts to address DEI, we thank the reviewers for emphasizing its importance. Our team is actively engaging in three different outreach activities, including participating in the Bioenergy Bridge to Career Program, onboarding one summer student intern from an underserved community, and hosting a full-day virtual workshop this summer with seminars, panel discussions with our team of scientists, and hands-on learning sessions in computer programming for bioenergy science. This is part of the consortium-wide plan for this program, which was presented in the overview presentation. We are also in full agreement with the reviewer on using real-world wet waste. Our initial aim is to continue using model biocrude solutions to isolate specific variables, control experimental conditions, and gain a deeper understanding of underlying adsorption mechanisms. Subsequently, in the near future, we will apply the understanding gained above to test adsorbents and developed regeneration methods with real SAF fractions from upgraded biocrude derived from food waste. We would also like to thank the reviewer for their emphasis of the importance of collaboration and the transparency of our partnerships. Our partnership with Oak Ridge National Laboratory is critical in developing a scalable process. Using their continuous flow system will enable us to generate real-world adsorbent performance data for commercial process scale-up. We would also like to clarify that our partnership with industry is through gathering their critical feedback. They are concerned with recalcitrant nitrogen species that will require a severe hydrotreating step, which requires high-pressure hydrogen and special unit operations that are limited to larger refineries. Thus, the research is aimed at addressing an alternative that will enable smaller refiners to meet fuel quality requirements. We thank the reviewers for their encouragement of working with an offtake partner to confirm that the final fuels from biocrude meet the SAF requirements. We will continue to explore strategic collaborations to maximize the real-world impact of our work; however, we would also like to emphasize that the consortium’s core strength is in technology development. This project, in addition to several other BETO-funded projects, has been collaborating with SAF analysis experts such as Joshua Heyne from Washington State University to conduct a two-tiered prescreening process to demonstrate the suitability of the upgraded biocrude for SAF requirements, providing solid groundwork for future approvals and larger-scale demonstrations.

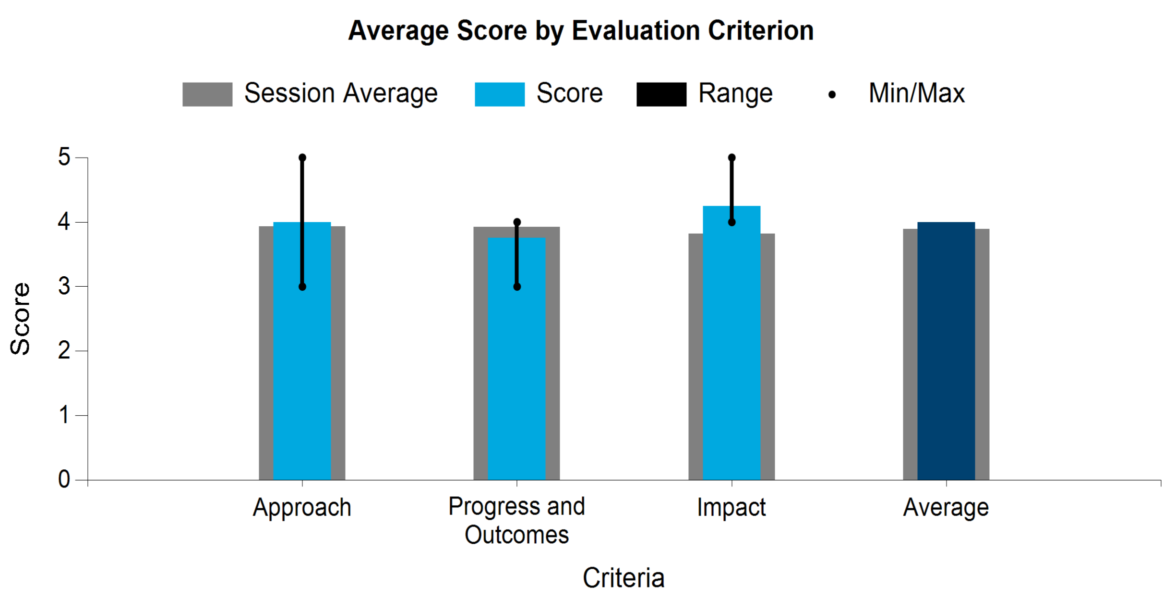
VOLATILE PRODUCTS RECOVERY

Separations Consortium

WBS:	SEP2.7
Presenter(s):	Phil Laible
Project Start Date:	10/01/2022
Planned Project End Date:	09/30/2025
Total Funding:	$650,000

PROJECT DESCRIPTION

Advances in strain engineering have enabled the biochemical production of a wide array of products. During aerobic fermentation, many of these molecules partition readily to the fermenter off-gas. Many of these are known to volatize fully during fermentation, while others volatilize less—but significantly—under typical conditions. This project utilizes advanced, high-surface-area materials for the recovery of volatile SAFs (and their precursors) by adsorption, with specificity achieved through tunable surface chemistries. This strategy thereby eliminates the energy-intensive steps (condensation/distillation) traditionally required to separate volatile organic products. Scaled adsorbent syntheses incorporating (1) macroporous structures capable of handling a high flow of fermentation off-gases while (2) retaining selective product recovery from bioreactors (= 10 liters) have been achieved. Performance with select volatile products has shown reproducible recoveries of ~85% of product from the vapor phase. Product is desorbed through compression with low energy forces (~20 psi, with materials reusable for >50 cycles). TEA and LCA demonstrate significant cost and environmental gains. Multiscale modeling enables mechanistic insights to optimize the recovery process for current and future products. Simple, inexpensive cartridge designs have facilitated successful, scaled validations—key metrics for industry stakeholders.



COMMENTS

- The project is on track. The team successfully demonstrated the use of xerogels (nanostructured adsorbents) with tunable surfaces in scalable, inexpensive capture cartridges to recover specific volatile products with little to no water. There was little or no info about xerogel chemistry. Scalability is a common challenge in structuring xerogels with specific structural profiles, such as mesoscale pores, high porosity, and high specific surface area. The team should integrate TEA for the scalability at the current stage. The previous and planned DEI activities look good.

- The team itself is diverse and has a very strong DEI plan in place to provide internships and learning experiences for DEI students. The development of vapor phase recovery will broaden the available products recovered from biorefining, so the project is important. The use of 3D printing to prototype cartridge geometry accelerates progress. There is good integration with the computational team and with other BETO projects so that specific volatile products can be retrieved, improving the overall success of the consortium. The industry collaboration looks really strong—there must be a lot of interest in recovering volatiles. The risks and mitigation strategies are clearly outlined. Milestones are on track. Are you thinking about ways to recycle or reuse the xerogel cartridges at EOL?
- This project has the potential to reduce the operating costs related to difficult bioproduct separations and has shown promise that good recoveries and purity can be obtained at a small scale. The project is well managed and appears to have good engagement with industry partners to meet the department's goals toward SAFs. The added complexity introduced by 3D printing does not seem to be adding significant value to the effort, and the scalability of that approach to meet the production volume goals for SAF does not seem to be well connected to the targets of the project. The overall research approach is rational, but a path toward scale-up is not clear. The project could benefit from a focus on the manufacturing of xerogel substrates, including surface modification, as well as the engineering requirements of the desorption step. The risk mitigation strategies for these scale-up issues could be strengthened.
- I appreciate that the team is actively collaborating with industry partners to use their capture technology within their systems to directly separate the volatile products of real reactions. I would like to see the team test their separations using real-world feedstocks and conditions, ideally in collaboration with their industry partners.

PI RESPONSE TO REVIEWER COMMENTS

- We thank the review team for their interest in our technology, and we appreciate both their recognition of the success of our approach and their suggestions for future improvement. We agree that this project should be kept as industry-forward as possible, and we note that (1) it was originally born from discussions with industry across many levels, and (2) these discussions continue to inform our technical strategy. With regard to the concerns raised by the review team, we share an interest in demonstrating, via both modeling and experimental validation, that xerogel synthesis and use can be scaled beyond the bench scale. We have tested synthetic strategies for production and adsorbent gels with the capacity to recover several liters of product, and we have deliberately utilized approaches in these experiments that can easily be expanded to pilot scale and beyond. This data was used in scaled cost evaluations indicating that the xerogel off-gas strategy would be cost-competitive for the capture of commodity products, especially with the cycle resilience of the materials and their ability to be reused hundreds of times. We will focus more on these demonstrations in the future, including a goal of achieving pilot-scale adsorption at the Advanced Biofuels and Bioproducts Process Development Unit (ABPDU) by Quarter 4 of FY 2025. In addition, we have an FY 2023 Quarter 3 milestone that revolves around the engineering requirements of the desorption step. We concur with the review team that this unit operation is central to the long-term viability of the xerogel strategy and will become one of the most critical demonstrative requirements as the approach scales further in the future. Last, we have engaged with a variety of industry partners and are planning validation of this off-gas capture approach with a range of products and processes over the next 18 months. These industrial products will be screened utilizing bench-scale workflows within the Separations Consortium, with the most promising products spun out as independent demonstration projects.

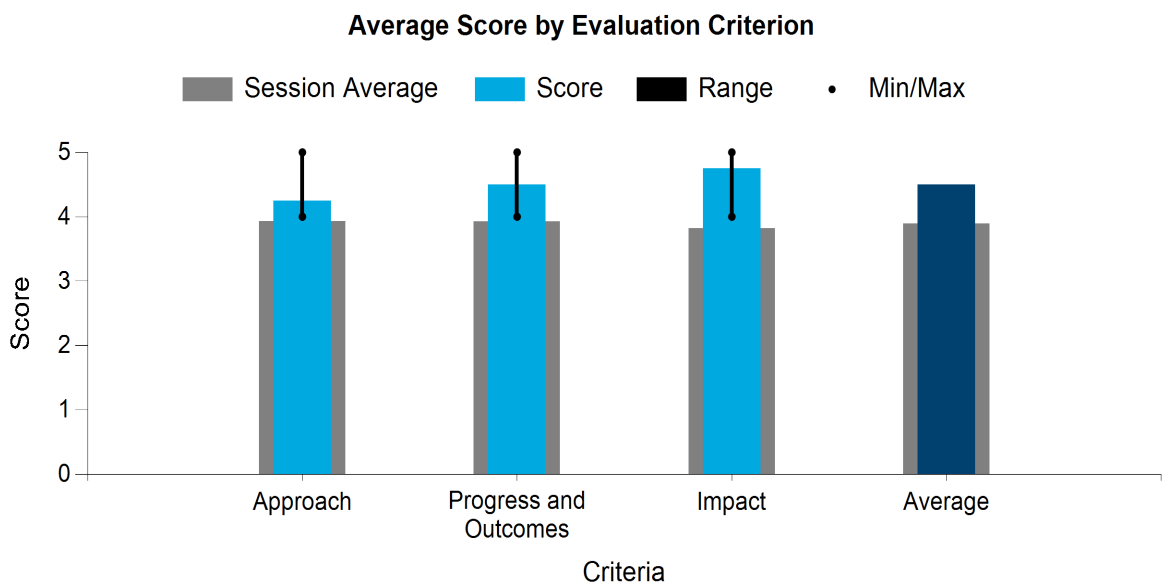
R&D-GUIDING TEA AND LCA

Separations Consortium

WBS:	SEP3.1
Presenter(s):	Jian Liu; Thathiana Benavides
Project Start Date:	10/01/2022
Planned Project End Date:	09/30/2025
Total Funding:	$825,000

PROJECT DESCRIPTION

TEA and LCA are used to guide and quantify the impacts of the Separations Consortium's R&D. The analysis team interfaces with experimental teams to illuminate the separation challenges that most influence the cost and environmental impacts of producing biofuels and bioproducts. This task quantifies the contributions to the cost and environmental footprint per fuel gallon equivalent or kilogram of product that these challenges are posing and compares them to defined baselines to assess the merits and drawbacks of the proposed technology. TEA is built on performance models developed in Aspen Plus and cost models in Excel using a discounted cash flow methodology. LCA uses biorefinery-level life cycle inventory data from the Aspen Plus models to determine sustainability impacts via calculations in the GREET model. During FY 2021 and FY 2022, the analysis team worked with the experimentalists addressing different challenges within the consortium and modeled the impacts of the various research efforts. Relative costs and sustainability outcomes were reviewed with the experimentalists. In this presentation, we describe the methodology around TEA/LCA for the Separations Consortium activities and present results for three different solutions of 2,3-BDO separations in support of the go/no-go milestone. A high-level challenge stream analysis was also conducted to identify separation challenges with high priority in the bioenergy processes and to reveal the maximum potential impact of using "ideal" separators.



COMMENTS

- The project is on track. The team collaborated with other BETO projects and industry partners. Understanding the trade-off between economic and environmental performance is crucial for sustainable practices, and integrating TEA and LCA is really important for evaluating emerging technologies at early TRLs. The team should be careful about any lack of consistent methodological guidelines, inconsistent system boundaries, limited data availability, and uncertainty for the integration of LCA and TEA. The team needs a clear plan on DEI initiatives.

- TEA and LCA were employed to assess and guide (recast) the BDO separations project and to optimize the research plan for the Separations Challenge project. Several significant project contributions resulted from the TEA/LCA, which highlighted cost and performance drivers for the processes. This is valuable feedback for the projects. Although this project functions as support to R&D, it provides critical guidance as projects progress to increase the likelihood of commercialization. Good and frequent communication between the LCA/TEA and experimental teams looks to be happening, which improves the progress and outcomes of all the projects. Milestones for BDO separation efficiency and the revised separation challenge streams were met. The DEI plan includes student internships, participation in the Bridge Program, and outreach activities.

- These analyses are essential to quantifying the technical merit of each project as well as ensuring that the outcomes satisfy the BETO strategy for technology development. It is apparent that these analyses are a key part of the other projects being reviewed and that the approach is widely adopted and considered a key element of the criteria for success for each project. The risks associated with performing these analyses at early stages of technology development are captured, and there appears to be a well-thought-out mitigation plan to address some risks. Additional risks to consider for these analyses are the fact that geography will often be a key driver for, e.g., energy costs or generation type, both of which will have a profound impact on the calculated cost and life cycle impact. The analyses were applied to great effect on the analysis of the 2,3-BDO separation process, which showed how the correct definition of the boundary conditions for the analysis drove the results. The impact greatly modified the approach applied to refining the development of the separation technology for that potentially valuable bioproduct.

- I appreciate how integrated the TEA/LCA work is in the go/no-go decisions for the rest of the projects. The example of how the TEA and LCA guided the development of the diol separations is a wonderful example of how to tie analysis into technology development. I think it would be helpful for the review to share the analysis team's milestones and timelines to better judge whether this team is on track as well. If the milestones are publication-based, for example, it would be great to share those numbers (goal versus actual) at the review.

PI RESPONSE TO REVIEWER COMMENTS

- "The team needs a clear plan for DEI initiatives": We thank the reviewer for this comment. Regarding the DEI initiative, we mentioned at the Project Peer Review that "we plan to participate in the Bioenergy Bridge to Career Program in FY 2023." We started working with the education programs at the different consortia labs to participate in the workforce development activities that will be conducted during summer 2023. This virtual workshop will occur every Friday starting July 14 and ending August 4. Among the activities we plan to participate in are selected topic webinars, panel discussions, and speed networking sessions, where we will interact with students from different backgrounds. Topics of discussion will include an introduction to the Bioprocessing Separations Consortium, bioenergy, LCA, and maximizing your digital and presence impact in social media, among others. Our team is also proud to have a strong representation of women, who have been traditionally underrepresented in STEM fields. This task has one female co-PI and supports a female postdoctoral researcher in her knowledge and skill development within bioenergy technologies.

- "I suggest the team include the impact of geography on the results of TEA and LCA": We agree with the reviewer regarding the point that regionalization will drive the results. Our current models are based on U.S. average costs, electricity grids, energy supplies, etc., and through our results, we can identify the key drivers of the cost and environmental metrics. We can vary parameters determined by the region, and this could be done by performing sensitivity analysis. In addition to sensitivity analysis, if necessary, TEA and LCA studies can also consider uncertainty characterization.

- "I suggest the team include milestones and a timeline for evaluation": We understand the reviewer's suggestion. Although economics and sustainability are strong considerations in the consortium's R&D, the consortium's deliverables to the project sponsor are primarily set based on the technical progress. As described in the overview presentation for the consortium, the analysis team worked closely with experimental researchers to develop internal analysis plans that align with the experiment timelines for all the tasks in the Separations Consortium at the beginning of the projects. We also track the TEA/LCA progress during biweekly meetings to ensure that the work is progressing toward our goals.

COMPUTATIONAL STUDIES SUPPORTING EXPERIMENTAL DESIGNS

Separations Consortium

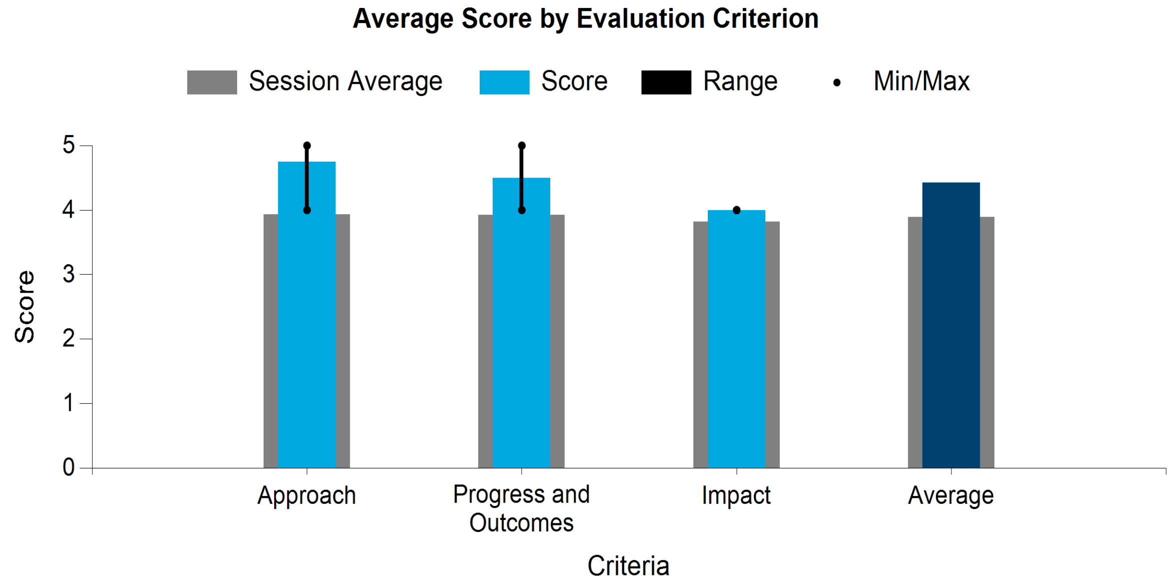
WBS:	SEP3.2
Presenter(s):	Difan Zhang
Project Start Date:	10/01/2022
Planned Project End Date:	09/30/2025
Total Funding:	$400,000

PROJECT DESCRIPTION

The separation of valuable compounds from complex biomass is hampered by high energy demands. Theoretical modeling and simulations can effectively accelerate the development of new materials and processes for efficient separation applications. Building on our collaborative work with the experimental consortium activities in FY 2020–FY 2022, we will combine atomistic-level modeling with data science tools to guide material selection and process optimization toward the design of novel separation technologies in collaboration with experimental teams.

The crosscutting computational task will interface with three experimental tasks to provide predictive guidance for down-selecting sorbent candidates and optimizing sorption processes. To achieve our goal, we will take input from our experimental partners on the specific separation task, including the target species; the desired properties of the sorbent materials; and other relevant information to establish the technology gaps that can be investigated by atomistic modeling. Atomistic-level modeling will be employed to characterize the sorbent/sorbate interactions and the key properties of sorbent materials corresponding to experimental conditions. Using these key properties as material features, we will apply high-throughput calculations to computationally screen candidate materials. Global optimization algorithms and ML technology will help accelerate our screening modeling. This screening will generate a list of potential material candidates tailored to target molecules and will inform fundamental insights into the governing factors in promising candidates. This knowledge will help the experimental teams narrow material candidates without needing to explore a wide experimental space, which is time-consuming and costly. In addition, TEA/LCA domain knowledge can be considered to further enhance our screening results by introducing more constraints in materials selection. On the other hand, our modeling results can refine the chemistry-guided parameters needed for TEA/LCA modeling.

The following specific tasks will be performed in collaboration with experiments: (1) computational screening of sorbent materials to understand the controlling factors of sorbents for deep nitrogen removal in experiments and to expedite the development of adsorption-based denitrogenation processes; (2) identification of new target volatile compounds and suitable functional groups in xerogels via computational modeling for the efficient capture of volatile compounds from off-gas streams; and (3) development of suitable solvent and membrane systems by computational modeling for large-scale 2,3-BDO separation from fermentation broth. We have identified three major challenges across these three tasks: the lack of high-quality data for complex sorbent systems, the trade-off between time cost and fidelity in the computational modeling of the bio-separation process, and the deficiency of fundamental knowledge for new technologies being developed in experimental teams. To address these challenges, we will carry out key molecular calculations and simulations as needed to fill the gap in data availability. To balance the trade-off between high accuracy and low cost, we will apply AI as the "last mile" tool to bridge the molecular modeling and experimental measurements. We will also pursue extensive collaborations with other BETO projects as well as industry partners to develop more fundamental knowledge about novel bio-separation technologies. By the end of the project, we will develop predictive modeling that expedites the search of solvent candidates for the solvent extraction of 2,3-BDO. Such methodology will be transferable to solvent solubility prediction in a wide range of industrial/BETO interests. We will also develop a molecular database to identify promising surface functional groups in resins for the efficient removal of both basic and non-basic nitrogen-containing compounds. Further, we will explore new research opportunities for functionalized xerogels to capture volatile compounds and guide the experimental synthesis of new xerogel materials. We expect that our computational modeling will offer guidance to expedite the advance of novel materials and processes in bio-separation. Theoretical tools will be made publicly available to interrogate bio-separation-relevant questions (e.g., solubility prediction for bio-separation process) and will pioneer new techniques for materials/processes in the decarbonization of transportation or industry. We will also disseminate our accomplishments in high-impact journals.



COMMENTS

- The project is on track. The team used the computational task to strategize and accelerate material/process discovery and design in bio-separation processes for three experimental tasks. A practical application of the computational screening/ML approach requires real-world case studies with sufficient data, which may be challenging for researchers; however, it could be a useful tool for the Performance-Advantaged Bioproducts and Bioprocessing Separations-related applications. Other than finding proper solvents, sorbents, and advanced materials with tailored surface groups, can we use ML to fill the data gaps of LCA? The PI did not present their DEI strategy and progress.

- This project is very new, kicking off in October 2022. The project will use computational modeling and AI to guide three projects and expedite the advances of materials and processes in bio-separations. The DEI plan includes topic development and the Bioenergy Bridge to Career internship. The team has already made progress in isolating promising solvent candidates for BDO extraction. In addition, the team has found that the Amberlyst resin could be a promising sorbate for denitrogenation and that long-chain alkyls on xerogels could provide better separation for limonene and isoprenol. These findings should help guide the experimental teams to accelerate their success.

- The project is a good example of how applied computational methods can reduce risk and increase the speed of the development cycle by providing better targeting and better fundamental understanding to improve the overall technical quality of multiple projects. In this case, applying these methods to solve problems in diol separation and adsorptive denitrogenation is particularly well thought out and impactful. The project efforts appear to have substantially advanced the SOA in both of those projects. The collaborative nature of this work is evident in the results. This work cannot proceed without excellent collaboration and work across diverse project teams. Excellent progress has been made, and a means of translating the successes of this effort to other projects should be developed and implemented to further advance both this project and the collaborative projects.

- I appreciate that the team is collaborating with the teams of Task 2.4 (Diol Separations), Task 2.6 (Enabling SAF Production by Adsorptive Denitrogenation), and Task 2.7 (Volatile Products Recovery). I would suggest that the team make their tool publicly available, if possible, to increase the impact of the fantastic work done here. Has the team explored industry collaborations for this model to work on industry-relevant problems? For the graphs of the computational screens, could you please mention what the desired feature is? On slides 6–8 in the presentation, please mark the desired quadrant, how close to the diagonal, etc.

PI RESPONSE TO REVIEWER COMMENTS

- The computational team thanks the reviewers for their thoughtful comments, valuable feedback, and encouragement. We appreciate that the reviewers value computational modeling, empowered by AI technology, in providing valuable insights to help guide experimental work and accelerate the technology goals set within the consortium. We have demonstrated good examples of how our computational modeling improves fundamental understanding in various bio-separation projects and how these new insights mitigate the risks of accelerating R&D in these projects. We are delighted that the reviewers found our computational work impactful and beneficial to advance the SOA in different projects. We strongly agree with the reviewers that our work cannot be achieved without excellent collaboration across diverse teams, and we remain determined to continue that in the future. We appreciate the constructive feedback on our presentation slides, and we will incorporate the suggested modifications in our future presentations to improve clarity. Several other specific comments are addressed below.

- The first reviewer asked if we could use ML to fill the data gaps of LCA. This is an excellent question! This is something we already considered several months ago, and we have initiated conversations with the TEA/LCA teams in the consortium. The idea would be to link the output from molecular modeling to AI tools to bridge the data gap and introduce more molecular-level insights in the TEA/LCA frameworks. With the reviewer's encouragement, we will continue developing this approach in the consortium.

- The first reviewer also commented that our DEI strategy and progress were not presented; however, the second reviewer noted our DEI approach. To clarify this, in slide 3 in our presentation, we indicated that we are deeply involved in the Bioenergy Bridge to Career Program by contributing to topic development and supporting student internships, as pointed out by a reviewer. The computational team members at PNNL and Oak Ridge National Laboratory will host a full-day virtual workshop with seminars, panel discussions, and hands-on learning sessions for programming in bioenergy science, and we will encourage the participation of underrepresented groups and students/postdocs. We have also reached out to local schools to host a Bioenergy Sciences for Students event. In addition, Vanda Glezakou is cochairing the next Gordon Research Conference in Chemical Separations on Jan. 21–26, 2024 (Galveston, Texas). The Gordon Research Conference is a premier conference with a very strong focus on DEI as well as mentoring of postdocs and early career scientists. We have requested a small contribution from the consortium (from FY 2024 funding) to support travel and registration for students/postdocs/early career scientists and underrepresented minority participants.

- The second reviewer's comment that the project is very new is only partially correct. The computational task was established in the previous cycle of the consortium (FY 2019–FY 2022) under the leadership of Vanda Glezakou, who also serves as the liaison between this consortium and the Consortium for Computational Physics and Chemistry. After Glezakou's move to Oak Ridge National Laboratory, Difan Zhang became the task lead, to provide the opportunity for leadership to an early career scientist.

- The third reviewer commented on the translation of our current success in the project to the other collaborative projects. We have strengthened the communication between different projects to ensure that the computational team's focus is on the core questions of our collaborators. We have also invited industry partners to join our advisory board to hear feedback from external voices and improve our connection to the other collaborative projects.

- The fourth reviewer suggested making our computational tool publicly available. In fact, we have been planning to make these tools available to the public once our paper is published. These tools will be shared via open-access platforms such as GitHub under the GNU license. This will also help us transfer our success in the current projects to the other projects, as the third reviewer commented.
- Further, a question was asked about whether we have explored industry collaborations for more industry-relevant problems. This activity is under development with the help of our experimental collaborators. For example, in our cooperation with the Volatile Product Recovery project, we have reached out to industry partners to identify new volatile compounds of interest, and the proper modeling for these compounds is currently under development.