

DOE Bioenergy Technologies Office (BETO) 2021 Project Peer Review

Upgrading of C2 Intermediates – ORNL

Catalytic Upgrading

March 10, 2021

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Oak Ridge National Laboratory



ChemCatBio Foundation – FY21

Integrated and collaborative portfolio of catalytic technologies and enabling capabilities

Catalytic Technologies

Catalytic Upgrading of Biochemical Intermediates

(NREL, PNNL, ORNL, LANL)

Upgrading of C1 Building Blocks (NREL)

Upgrading of C2 Intermediates (PNNL, ORNL)

Catalytic Fast Pyrolysis (NREL, PNNL)

Electrocatalytic CO₂ Utilization (NREL)

Enabling Capabilities

Advanced Catalyst Synthesis and Characterization (NREL, ANL, ORNL)

Consortium for Computational Physics and Chemistry(ORNL, NREL, PNNL, ANL, NETL)

Catalyst Deactivation Mitigation for Biomass Conversion (PNNL)

Industry Partnerships (Phase II Directed Funding)

Opus12 (NREL)

Visolis (PNNL)

Sironix (LANL)

Cross-Cutting Support

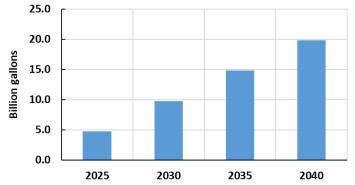
ChemCatBio Lead Team Support (NREL)

ChemCatBio DataHUB (NREL)

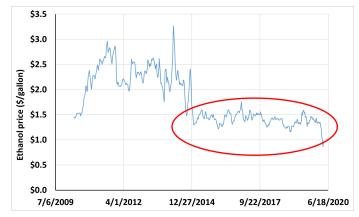
Project Overview: Important Platform Chemical - Ethanol

Ethanol is an attractive feedstock for middle distillate fuels and chemical products:

- Growing market of aviation biofuels
- Established ethanol market (~29.0 billion gallons globally in 2019)
- Lower ethanol prices over the last five years creates opportunities for jet fuel production from ethanol
 - Chemical coproduct from ethanol can further enhance the cost competitiveness
- Factors expected to **expand ethanol availability**:
 - Fthanol "blend wall"
 - Advancement in ethanol production efficiency
 - Feedstock diversification



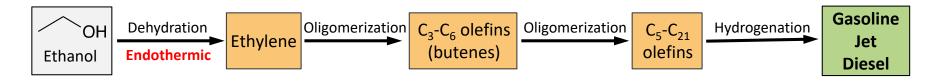
Aviation biofuel consumption in IEA Sustainable Development Scenario



US historical ethanol selling price (source: Iowa State University)

https://www.iea.org/commentaries/are-aviation-biofuels-ready-for-take-off Renewable Fuels Association, ethanolrfa.org/statistics/annual-ethanol-production

Project Overview: Conventional Ethanol to Jet (ETJ) - Benchmark



Limitations for conventional ETJ:

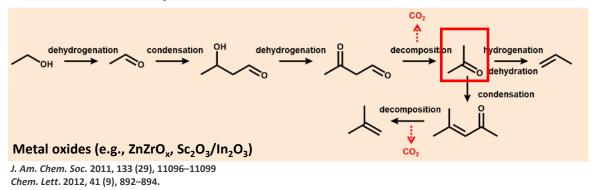
- Two-step process operation for C₃₊ olefin production
- Ethanol dehydration is endothermic (requires significant energy input)
- Large unit operation temperature differences → increase heat management duty
 - O Dehydration, 1st-stage and 2nd-stage oligomerization: e.g., 200-350, ~85 and 200-300°C
- Higher CapEx and OpEx

Opportunities for developing one-step ethanol to C_{3+} olefin (ETO) technology:

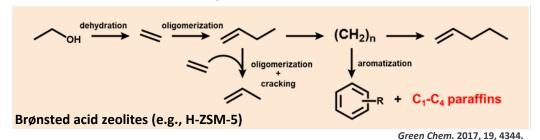
- Reduce the number of unit operations
- Minimize external energy inputs and reduce the demand of heat management
- Reduce CapEx and OpEx

Project Overview: Literature Available One-Step ETO Pathways

ETO over oxide-based catalysts via acetone:



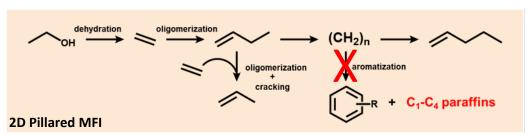
Brønsted acid-catalyzed reactions to C₃₊ olefins:

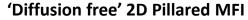


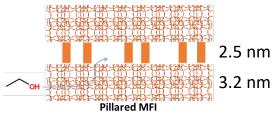
Common challenge: significant side product formation (e.g., light paraffins, CO₂)

Project Overview: 2D Pillared MFI to Optimize C₃₊ olefins (ORNL, FY17)

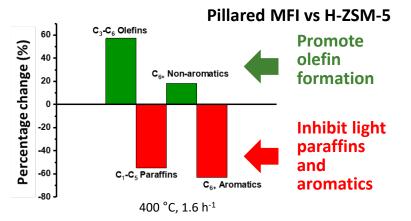
Minimize downstream aromatization and light paraffin formation to promote C_{3+} olefin formation by significantly reducing the diffusion length:

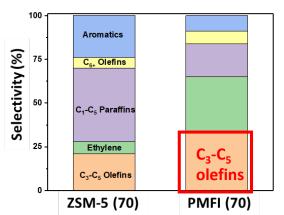






Yuk et al. J. Phys. Chem. C 2020, 124, 28437.





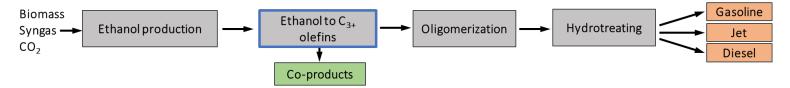
Set FY17 baseline:

- 100% ethanol conversion
 - 35% C₃₊ olefins
- 65% total olefins

Challenges in inhibiting ethylene

• Strong R&D need in new catalysis and reactions for one-step ETO to selectively produce C₃₊ olefins

Project Overview: Goal Statement



Goal: Develop efficient **one-step ETO technology** to enable high carbon efficiency to liquid hydrocarbon fuels at lower cost, and enable the development of a **market-responsive biorefinery** concept through C₂ platform providing control over gasoline, diesel, and jet fuel, as well as high value co-products

Outcome: Advance C₂ upgrading technology and reduce commercialization risk by addressing catalysis and process challenges and demonstrating liquid fuel production at a modeled minimum fuel selling price (MFSP) of \$3.0/GGE

Differentiator

Conventional ETJ

- Two-step ethanol to C₃₊ olefins
- Higher energy demand due to endothermic dehydration and temperature differences between unit operations
- **Higher CapEx and OpEx** (e.g., ethanol upgrading cost is \$1.19/GGE based on two-step Ziegler reaction*)

*Tao et al. Green Chem. 2017, 19, 1082-1101.

ETJ via One-step ETO

- One-step ethanol to C₃₊ olefins
- Reduced energy management burdens:
 - Shift to slightly exothermic reaction
 - ETO and oligomerization are operated at similar T
- Lower CapEx and OpEx (ethanol upgrading cost reduced to \$0.5-0.6/GGE, based on our process)

1-Management Approach

Joint milestones to tackle challenges associated with catalyst development and deactivation mitigation

Advanced catalyst synthesis and characterization (ACSC)

Understand catalyst structures and deactivation mechanisms; leverage characterization capabilities developed for C1 project

Consortium for Computational Physics and Chemistry (CCPC)

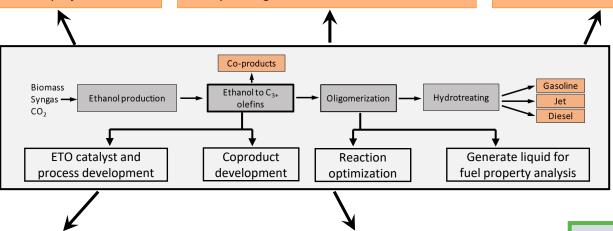
Computational modeling to understand the catalysis and chemistry, and predict new catalyst design

Catalyst deactivation mitigation (CDM)

Probe catalyst deactivation mechanism and mitigate catalyst deactivation

BETO peer review CCB industry advisory board Industry outreach

Inputs



Publications, conference presentations, webinars, IP, tech transfer

Outputs

C₂ upgrading – PNNL

Collaborate on olefin oligomerization and coproduct development

Analysis team (NREL & ANL)

Perform process design, TEA, and LCA

TEA-informed metrified milestones and Go/No-Go to relate catalyst advancement to cost

End goal is to transfer R&D discoveries to industry and promote US bioeconomy

1-Management Approach: Risks and Mitigation

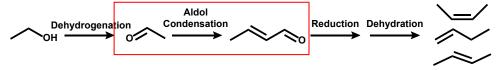
Risks	Mitigation
New ETO catalyst being developed requires investigation of long-term catalyst durability and regeneration.	Catalyst characterization, deactivation and regeneration studies are planned as part of new catalyst testing throughout the course of the project. Accelerated catalyst deactivation may be considered.
Achieving \$3/GGE distillate production may be difficult with the current fermentation-derived ethanol feedstock and process model.	TEA scenario analysis will be conducted for new concepts with coproducts from either ethanol or lignin .
The compositions of C₃₊ olefins are not ideal for making high-quality aviation fuels.	Oligomerization of the C_{3+} olefins derived from ETO will be performed and optimized to generate liquid hydrocarbons. Fuel property analysis will be carried out to guide the optimization of C_{3+} olefin composition.

Milestones Associated with Risk Mitigations

- FY21 Q2 (C2-CDM): Determine impact of steam on Y/BEA stability and possible deactivation mechanism
- FY21 Q3 (C2-ACSC-CCPC-CDM): Develop a regeneration procedure and/or new catalyst formulation to optimize regenerability by characterizing deactivation mechanism(s) of Cu-Zn-Y/Beta for ETO
- FY21 Go/No-Go: Evaluation of pathway(s) to meet FY22 carbon efficiency and cost targets

2-Technical Approach: Research Challenges and Success Factors

Target one-step ETO via acetaldehyde & crotonaldehyde as intermediates



Research Challenges

- **Design and balance multiple active sites** to catalyze cascade reactions to selectively produce targeted C_{3+} olefins
- *Minimize side reactions*: ethanol dehydration, C-C cleavage to CO₂, olefin over hydrogenation
- Characterize and understand the catalyst structures, reaction mechanisms for the purpose of designing next-generation catalyst
- Maximize catalyst lifetime

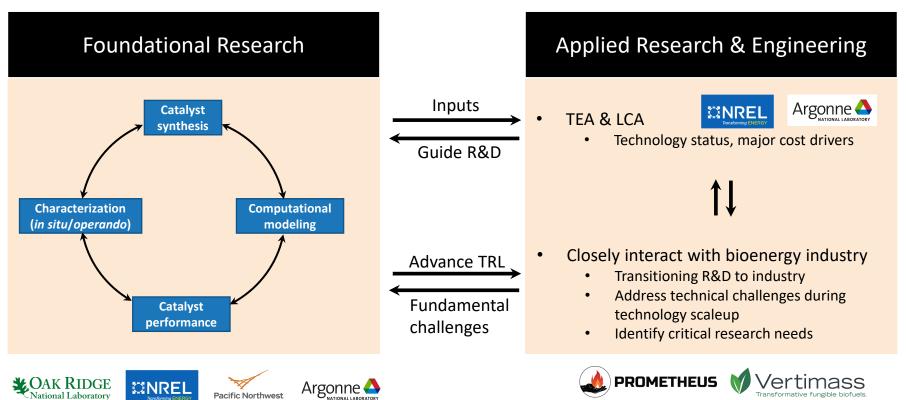
Critical Success Factors

- Maximize carbon efficiency to liquid hydrocarbons via active and selective multifunctional catalyst design
- *Understand deactivation and develop regeneration/mitigation approaches* though collaborating with ChemCatBio enabling projects (CDM, ACSC, CCPC)
- Advance technologies with bioenergy industry partnerships

2-Technical Approach: Integrated Foundational and Applied Research

Argonne 📤

Collaborative efforts to enable fundamental catalysis findings and advance technology





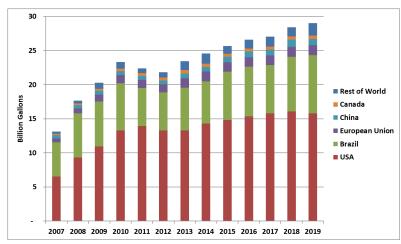
≅NREL

Pacific Northwest

3-Impact: Relevance to Bioenergy Industry

New ETJ technology addresses the needs for both ethanol producers and growing markets of middle-distillate fuels

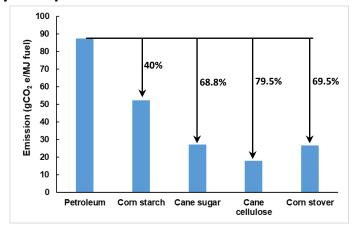
Increasing ethanol production, ethanol 'blend wall' and decreasing ethanol price urge for **new applications**



Global Ethanol Production by Country or Region

ETJ present a great opportunity to expand the ethanol applications, diversifying the product portfolios for ethanol producers

Middle distillate fuels from renewable ethanol can help to address the **decarbonization** challenges for **heavy duty transportation and aviation**.



Well-to-wake analysis for jet production from different feedstocks

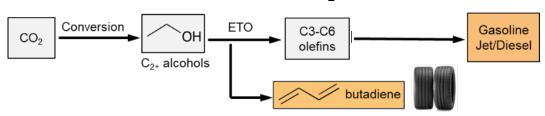
Vertimass ethanol upgrading technology licensed from ORNL shows significant opportunities for GHG emission reduction (joint study with Vertimass*)

*Hannon et al. PNAS. 2020, 117 (23), 12576-12583.

3-Impact: Relevance to Bioenergy Industry

ORNL licensed new ethanol-to-butadiene-jet (ETBJ) technology to Prometheus to expand the applications to CO₂-derived alcohols



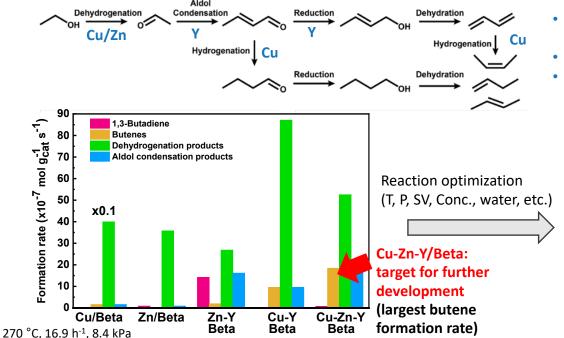




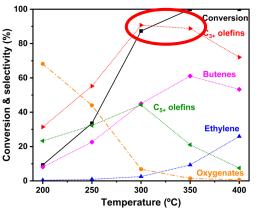
- ORNL ETBJ technology in converting alcohols to liquid fuels and chemicals provides:
 - High yield of liquid hydrocarbon fuels
 - Lower CapEx and OpEx
 - Flexibility to produce chemical co-product
- Additional IPs developed on ethanol upgrading technologies to expand the collaboration opportunities with industry
 - US Patent 10,696,606, and additional three US patent applications
- Disseminate scientific learnings on catalysis for bioenergy through peer-review publications
 - Hannon et al. PNAS. 2020, 117 (23), 12576-12583; Yuk et al. J. Phys. Chem. C 2020, 124, 28437.
 - Sun et al. Nat Commun 2018, 9, 4454; Cordon et al. ACS Catal. Under revision.

4-Progress and Outcomes: One-Step ETO Catalyst Design

Objective: develop multifunctional Lewis acid zeolite-based catalyst(s) to catalyze cascade ethanol conversion to butene-rich C_{3+} olefins



- Beta zeolite: leverage pore confinement effect, high surface area for anchoring metal sites
- Cu/Zn: ethanol dehydrogenation
- Y sites: 'softer' Lewis acid for aldol condensation, minimize ethanol dehydration



Oxygenates include acetaldehyde, diethyl ether, C4 oxygenates

0.53 h⁻¹, 7.3 kPa ethanol, 97.5 kPa H₂

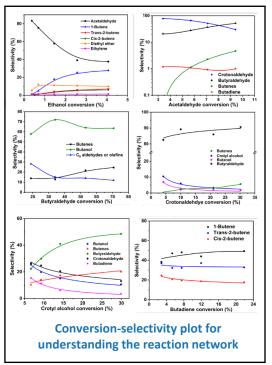
Cu-Zn-Y/Beta can selectively produce butene-rich C₃₊ olefin

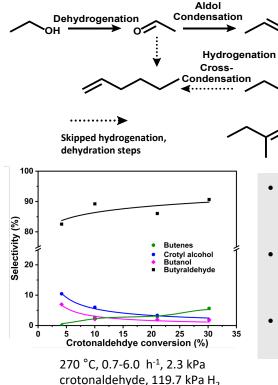
89% C₃₊ olefin, 98% total olefins at ~100% conversion (350 °C)

ethanol, 113.6 kPa H₂

4-Progress and Outcomes: Reaction Network Understanding

Objective: understand ETO reaction network over Cu-Zn-Y/Beta catalyst by feeding ethanol and various reaction intermediates





• Presence of several competitive pathways for

Dehvdration

Hydrogenation

Reduction

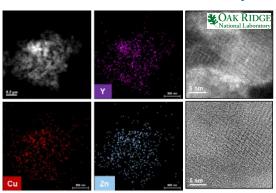
- crotonaldehyde to butenes; formation of butyraldehyde is the major one
- Opportunities for new catalyst design to tune different olefin targets (e.g., 1-butene, 1hexene)
- **Future work:** tune and balance catalyst sites to provide control on this ETO chemistry

Junyan Zhang, et al. In preparation.

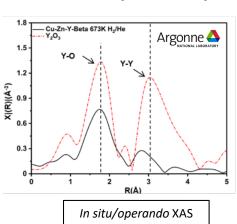
4-Progress and Outcomes: Catalyst Structures

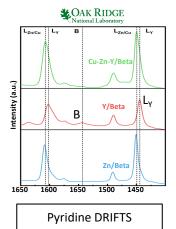
Objective: accelerate understanding of catalyst structures via collaborations with ACSC and CCPC, providing guidance for next-generation catalyst design

Catalyst characterizations (with ACSC)







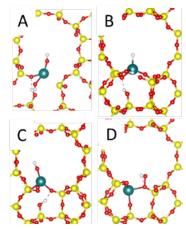


Outcomes:

- Characterizations showed atomically dispersed metal sites over Cu-Zn-Y/Beta
 - Isolated Y induced both L acid sites and B acid sites
- Joint efforts with CCPC and ACSC provided structural information for further catalyst design
 - Eliminate B acid sites to inhibit ethanol dehydration; alternative L acid centers for condensation

Future work: further establish structure-function relation for new catalyst design

Modeling (with CCPC)



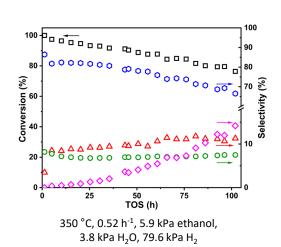
Potential Y/Beta structures identified via modeling (PNNL)

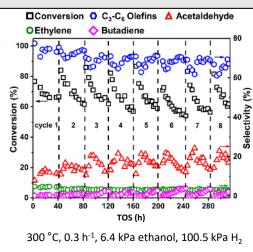
- Catalyst structures
- Reaction mechanism
- Guide catalyst design

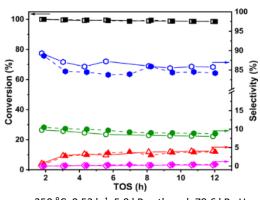
Junyan Zhang, et al. In preparation.

4-Progress and Outcomes: Catalyst Stability

Objective: evaluate Cu-Zn-Y/Beta catalyst stability, regenerability and water tolerance







 $350~^{\circ}\text{C},\,0.52~\text{h}^{\text{-}1},\,5.9~\text{kPa}$ ethanol, 79.6 kPa $\text{H}_2;\,0.52~\text{h}^{\text{-}1},\,5.9~\text{kPa}$ ethanol, 79.6 kPa $\text{H}_2,\,2.3~\text{kPa}$ water

Stephen Purdy, et al. In preparation.

Outcomes:

- Cu-Zn-Y/Beta was operated for >100 h TOS in one cycle, regenerable for multiple cycles via oxidative regeneration
- Water does not show significant impact on ethanol conversion and product selectivity for short TOS (Preliminary)
- Offer potential for feeding wet ethanol to cut cost by reducing separation burden

Future work:

 Collaboration with CDM, ACSC, CCPC to further understand catalyst deactivation (i.e., steam stability, coke formation, metal sites stability) in FY21 Q2 and Q3, providing insights for long-term catalyst stability

4-Progress and Outcomes: Performance at Varied H₂ Partial Pressures

Objective: understand Cu-Zn-Y/Beta performance at different H₂ partial pressures or without H₂

H ₂ Partial Pressure	Product Selectivity (%)									
(kPa)	(%)	AA	C ₂ =	C ₃ =	C ₄ =	C ₅₊ =	C ₃₊ =	Butadiene	Oxygenates	Paraffins
101	96.4	6.3	17.0	5.0	56.7	10.8	72.6	0.3	9.4	0.6
75	95.8	6.6	17.0	5.0	56.9	11.1	72.9	0.5	9.0	0.5
50	94.5	6.9	16.9	4.9	57.2	8.5	70.6	1.7	10.5	0.3
25	93.0	6.5	16.0	5.0	52.9	7.3	65.2	8.3	10.3	0.2
0	90.6	6.5	16.9	5.1	12.9	6.8	24.8	47.5	10.7	0.1

350 °C, 0.52 h⁻¹, 7.4 kPa ethanol

AA: acetaldehyde; oxygenates include diethyl ether, AA, C4 oxygenates

Junyan Zhang, et al. In preparation.

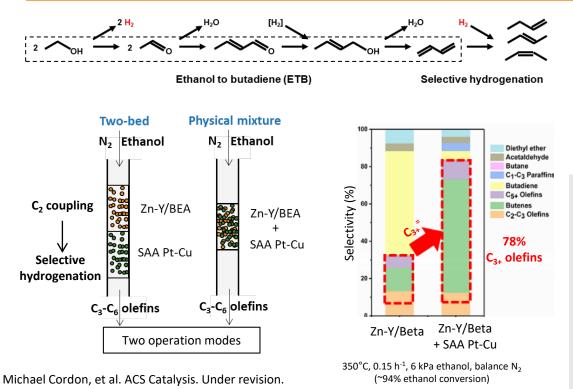
Outcomes:

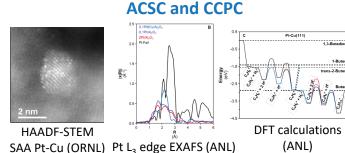
- H₂ partial pressure (<50 kPa) can be adjusted to tune butadiene/C₃₊ olefins
- At low H₂ partial pressure or without H₂, this process can produce middle distillate and butadiene simultaneously
- Preliminary TEA indicates coproduction of butadiene can significantly reduce MFSP
- How to achieve selective ETO without hydrogen cofeeding? (industry interests in this approach)

4-Progress and Outcomes: Ethanol to Butenes without External H₂

Objective: utilize in situ generated H₂ to achieve selective ethanol to butene-rich olefins

Challenge: require active and selective catalyst that can utilize these H_2 at ETO conditions (>300 °C)





Outcome: composite catalyst (Zn-Y/Beta + SAA Pt-Cu) can achieve selective ETO without H₂ cofeeding (**78% C**₃₊ **olefin selectivity**, 94% conversion)

 Characterizations and DFT calculations demonstrate SAA Pt-Cu ensemble sites are critical for BD hydrogenation at ETO conditions

Impact: avoid external H₂ supply, separation and recycle; reduce OpEX and CapEx

4-Research Progress Summary

Joint catalysis R&D significantly advanced the state of technology for ETJ via single-step ETO over ORNL catalysts

	FY17	FY18	FY20	Target
ETO Catalyst	Pillared H-MFI	Cu-Zn-Y/Beta	Cu-Zn-Y/Beta	Improved design
ETO Single-pass EtOH Conversion	100%	~99%	100%	100%
C ₃₊ Olefin Selectivity	33%	87%	89%	95%
Total Olefin Selectivity	65%	92%	98%	98%
Liquid Hydrocarbon Yield (GGE/gal. EtOH)	0.27	0.54	0.58	0.62
Demonstrated Time on Stream		~40 h	>300 h	>300 h*
EtOH Feed	Pure EtOH	Pure EtOH	Aqueous EtOH (up to 60 wt.% water)	Biomass/CO ₂ derived EtOH
EtOH Upgrading Cost (\$/GGE)	1.35	0.94	0.60	0.50
Technology Readiness Level	1	1	3	

^{*}Long-term durability testing may be achieved via other opportunities

Future work on catalysis R&D:

- Improve C₃₊ olefin selectivity via new catalyst design, leading to increased liquid hydrocarbon yield
- Understand and improve catalyst stability
- Develop **coproduct(s)** from ethanol based on TEA guidance

Summary

Project Goal

Develop efficient one-step ETO technology to enable high carbon efficiency to liquid hydrocarbon fuels at lower cost, and enable the development of a market-responsive biorefinery concept through C₂ platform providing control over gasoline, diesel, and jet fuel, as well as high value co-products

Approach

- Developing *multifunctional Lewis acid catalysts* to catalyze cascade reactions to selectively produce C_{3+} olefins
- **Integrated foundational and applied research** to accelerate the catalyst development

Impact

- New ETJ technology addresses needs for both ethanol producers and growing markets of middle distillates
- Transitioning R&D discoveries from national labs to industry

Research progress

- Demonstrate a new Cu-Zn-Y/Beta catalyst for selective ETO (~89% C₃₊ olefins, 98% total olefins)
- Cu-Zn-Y/Beta is *regenerable* for multiple cycles via oxidative regeneration, and *stable in the presence of water*
- Collaboration among ACSC, CCPC and C₂ upgrading enables the understanding of catalyst structure and chemistry, providing guidance for further catalyst development
- R&D efforts have significantly advanced the state of the technology

Quad Chart Overview

Timeline

- 10/01/2019
- 09/30/2022

	FY20	Total Planned Funding (FY20-Project End Date)
DOE Funding	\$400K	\$1.2M

Barriers addressed

Ct-F: Increasing the Yield from Catalytic Processes

 Developing catalysts that enable processes with higher carbon efficiency and yield

Ct-E. Improving Catalyst Lifetime

 Exploring deactivation via characterization, and developing regeneration/mitigation strategies

Project Goal

Develop *efficient one-step ETO technology* to enable high carbon efficiency to liquid hydrocarbon fuels at lower cost, and enable the development of a *market-responsive biorefinery concept* through C2 platform providing control over gasoline, diesel, and jet fuel, as well as high value co-products

End of Project Milestone

Demonstrate *improvements to the direct ethanol to* butene-rich olefins catalyst technology thereby enabling a new, market-responsive biorefinery pathway through C2+ oxygenates providing control over gasoline, diesel, jet, and co-products, with the ability to obtain a distillate MFSP of at least \$3/GGE.

Funding Mechanism

AOP Project (Direct-Funded Lab Project)

Acknowledgement

ORNL team

Zhenglong Li
Michael Cordon
Kinga Unocic
Shiba Adhikari
Bruce Adkins
Junyan Zhang
Stephen Purdy
Brian Davison
James Parks
Todd Toops

NREL team

Ling Tao Susan Habas
Dan Ruddy Qiyuan Wu

PNNL team

Roger Rousseau Gregory Collinge
Vanda Glezakou Mal-Soon Lee
Robert Dagle Vanessa Dagle
Huamin Wang Fan Lin

ANL team

Ted Krause Evan Wegener Rajeev Assary Mingxia Zhou

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James Harris (UA)
Dongxia Liu (UMD)









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Additional Slides

Responses to Previous Reviewers' Comments

- "In general, the project has very focused tasks to develop a wide range of potential full candidates from low molecular weight oxygenates. The team successfully leverages interactions with many groups within ChemCatBio to enhance the project. The majority of the project has seen activity and selectivity metrics met, though room to improve catalyst reusability remains. In general, future studies appear to build on prior successes and will leverage capabilities in ChemCatBio to attempt to further catalyst performance. The project is productive in both connecting with fundamental science, publications in field-leading journals, technology, and several patents and a successful technology transfer with one of the processes."
 - We agree that catalyst reusability remains an important part of our research. This is the focus of our endof-year goal, where we will explore deactivation and regeneration in all three of our oxygenate conversion pathways.

Publications, Presentations, and Patents

Publications

- 1. S.F. Yuk, M. Lee, G. Collinge, J. Zhang, A. B. Padmaperuma, Z. Li*, F. Polo-Garzon, Z. Wu, V. Glezakou, R. Rousseau*. Mechanistic Understanding of Catalytic Conversion of Ethanol to 1-Butene over 2D-Pillared MFI Zeolite. Journal of Physical Chemistry C, 2020, 124, 28437.
- 2. J.R. Hannon, L.R. Lynd*, O. Andrade, P.T. Benavides, et al. Technoeconomic and life cycle analysis of single-step catalytic conversion of wet ethanol into fungible fuel blendstocks. PNAS, 2020, 117, 12576-12583.
- 3. M.J. Cordon, J. Zhang, S.C. Purdy, E.C. Wegener, K.A. Unocic, L.F. Allard, M. Zhou, R.S. Assary, J.T. Miller, T.R. Krause, J. Kropf, C. Yang, D. Liu, Z. Li*. Selective butene formation in direct ethanol to C3+ olefin valorization over Zn-Y/Beta and single-atom alloy composite catalysts using in situ generated hydrogen. *ACS Catal*. Under revision.
- 4. J. Zhang, L. Tao*, B.H. Davison, Z. Li*. Decarbonize Aviation via Cost-Competitive Middle Distillate Fuels within a Market-Flexible C2 Platform-based Biorefinery Concept. In prep.
- 5. J. Zhang, E.C. Wegener, S.C. Purdy, K.A. Unocic, L.F. Allard, M. Zhou, R.S. Assary, J.T. Miller, T.R. Krause, J. Kropf, C. Yang, D. Liu, Z. Li*. Isolated metal sites over Cu-Zn-Y/Beta for selective butene-rich C3+ olefins production from ethanol. In prep.

Publications, Presentations, and Patents

Presentations

- 1. R. Dagle, V. Dagle, Z. Li. "Single-step catalytic conversion of ethanol to n-butene-rich olefins and 1,3-butadiene chemical product", DOE BETO ChemCatBio Webinar, July 31, 2019.
- 2. J. Zhang, S. Adhikari, C. Yang, N.J. Libretto, J. Miller, T. Krause, K. Unocic, D. Liu, Z. Li. "Multifunctional Zeolite Catalyst for Cascade Conversion of Ethanol to C3+ Olefins: A Pathway to Renewable Jet Fuel", NAM26 2019 North American Catalysis Society Meeting.
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Publications, Presentations, and Patents

Patents

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- 2. Zhenglong Li, "Zeolitic catalytic conversion of alcohols to olefins" United States Patent Application No. 16/530,397 filed on August 2, 2019.
- 3. Zhenglong Li, "Method for alcohol upgrading to jet, diesel, gasoline and valuable co-products." United States Patent Application No. 17/083,437 filed on October 29, 2020.
- 4. Zhenglong Li, Junyan Zhang, Brian Davison. "New catalyst materials for ethanol conversion to high olefins." United States Provisional Application Serial No. 63/141,996.

Technology Transfer

 ORNL licensed new ethanol-to-butadiene-jet (ETBJ) technology to Prometheus Fuels: https://www.ornl.gov/news/prometheus-fuels-licenses-energy-saving-ornl-ethanol-jet-fuel-process