Catalytic Upgrading of Pyrolysis Products for the Production of Sustainable Aviation Fuel

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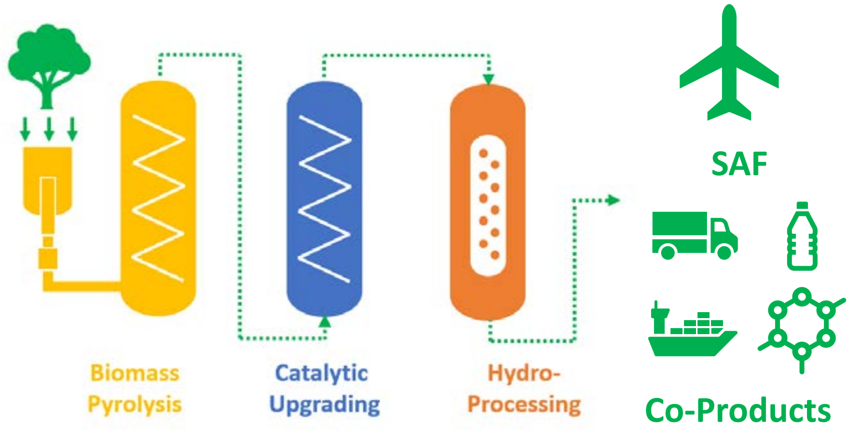
Catalytic Upgrading Session
BETO Peer Review
April 3-7, 2023

NREL/PR-5100-85850

Project Overview

Project Goal: Develop a technology pathway to convert woody biomass into sustainable aviation fuel (SAF) and other biogenic products via catalytic fast pyrolysis (CFP) and hydrotreating



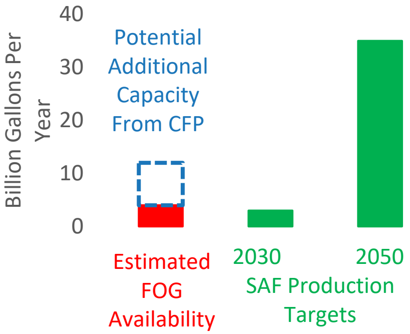
Key Advantage: Catalytic fast pyrolysis generates a stabilized bio-oil that de-risks down-stream hydroprocessing

Market Trends: Repurposing refinery hydroprocessing infrastructure for the production of renewable diesel and sustainable aviation fuel from fats, oils, and greases

Phillips 66: Rodeo, CA

Shell: Rotterdam, Netherlands

Existing approach is constrained by the availability of waste fats, oils, and greases



This research opens pathways for SAF production from forest resources and woody wastes

Estimated Availability of Forest Resources and Woody Wastes
133 Million Dry Tons/Yr
8 BGPY Hydrocarbon Fuel Potential

DOE Billion Ton Report, 2016, ORNL/TM-2016/160

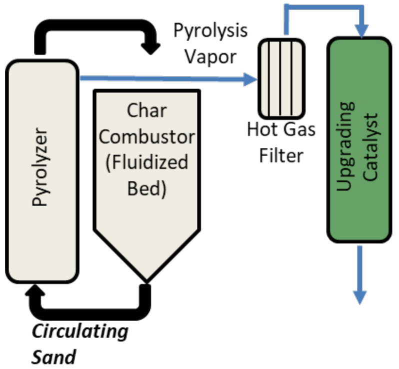
Project Overview

Previous Research: Fixed Bed Catalytic Fast Pyrolysis

- Demonstrated potential to achieve 78% GHG emissions reduction with a modelled minimum fuel selling price (MFSP) < $3.00 GGE
- Evaluated durability over 100+ reaction regeneration cycles
- Identified opportunities to valorize high value co-product streams

Challenges:

- **Requires co-fed hydrogen and utilizes noble metal catalysts**
- **Rapid deactivation necessitates multiple reactors for continuous upgrading**
- **Poor heat transfer in fixed bed**



Dutta. A., et al., J. Chem. Eng., 2023, 451, 138485
French, R. J., et al, ACS Sustain. Chem. Eng., 2021, 9, 1235
Griffin, M.B., et al. Ener. Environ. Sci., 2018, 11, 2904

2021 Pivot

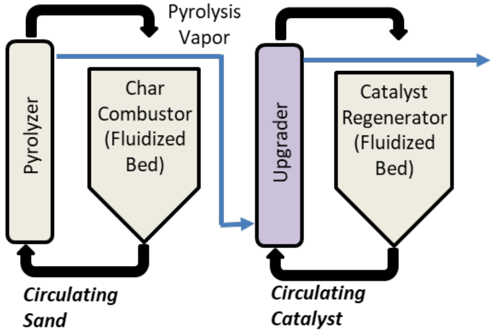
Current Research: Fluidized Bed Catalytic Fast Pyrolysis

Advantages:

- **Can be performed without co-fed hydrogen using zeolite catalysts**
- **Rapid deactivation can be managed through continuous regeneration**
- **Improved heat transfer in fluidized bed**

Research Needs:

- Benchmark performance for SAF production
- Establish critical material attributes for CFP-oil
- Increase carbon efficiency and fuel yield



Approach: Schedule

Kickoff 10/1/21	Phase 1: Current Work	*Go/No Go 06/30/23*	Phase 2: Future Work	*End Date 9/30/24*

Objectives:

Benchmark performance for SAF production and establish critical material attributes for CPF-oil

Technical Targets:

- Demonstrate the production of **SAF that meets ASTM D4054 guidelines** for density, viscosity, boiling point, heating value, flash point, freeze point, and volatility.
- Demonstrate **≥70% GHG reduction** without requiring noble metal catalyst or co-fed hydrogen during CFP
- Establish **three critical material attributes** (CMAs) for CFP bio-oil

Objectives:

Increase process carbon efficiency and fuel yields

- Modification of technical CFP catalysts to reduce coking and increase bio-oil production
- Process optimization to effectively utilize byproduct streams (e.g., gasses, volatile condensables, char)

Technical Targets

- Demonstrate a **≥ 20% increase in carbon efficiency to SAF** while maintaining the ability to meet the fuel property, GHG reduction, and CMA targets outlined in the project's GNG.
- Demonstrate hydroprocessing durability for **≥ 500 h time on stream**

Approach: Integrated Experimental Campaigns

Biomass Handling and Preprocessing (INL) → **Ex-Situ CFP** Fluidized Bed ZSM-5 SAR 30 → **Hydrotreating** Fixed Bed Sulfided NiMo/Al_2O_3 → *Fractionation and Fuel Property Analysis*

- Continuous CFP experiments performed **without requiring co-fed hydrogen or noble metal catalysts**
- **Realistic biomass feedstocks** including clean pine and waste forest residues
- Sufficient product volume for **fuel property assessment**
- **Technical catalyst formulations** for CFP and hydrotreating steps
- Comprehensive analytical capabilities to achieve **material balances of 100 ± 5%**

Supporting Analysis

Pre- and Post-Reaction Characterization of Technical Catalyst Formulations

Bio-Oil Thermal Stability Assessments

Technoeconomic and Lifecycle Analysis

Approach: Management Plan

Task 1: Project Management and Industry Engagement
Lead: Mike Griffin

Task 2: Catalytic Fast Pyrolysis of Biomass
Lead: Cody Wrasman

Task 3: Hydroprocessing of CFP-Oil to SAF
Lead: Kristiina Iisa

Task 4: Thermal Stability of CFP-Oil
Lead: Mark Nimlos

Task 5: Characterization of Catalyst Properties
Lead: Susan Habas

Task 6: Refinery Economic and Optimization Modelling
Lead: Michael Talmadge

Diversity, Equity, and Inclusion
Lead: Anne Starace

- Dedicated funding to support DEI efforts
- DEI-position on the project management team
- Inclusion of DEI milestones as project deliverables

Partnering with Minority Serving Institutions

Supporting Minority Owned Businesses

Engaging through workshops and 'DEI-minutes'

Collaborating on 'JUST-R' Metric Development*

*Dutta, N. S., et al., "Just-R metrics for considering energy justice in early-stage energy research", Joule, 2023, in press

Approach: Communication and Collaboration

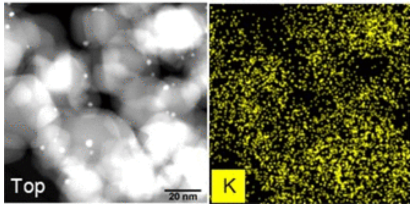
Coordination with Enabling Projects Provides Fundamental Insight into Catalyst Deactivation and Regeneration

Catalyst Deactivation Mitigation

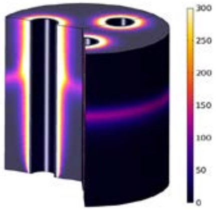
Advanced Catalyst Synthesis and Characterization

Consortium for Computational Physics and Chemistry

Catalytic Upgrading of Pyrolysis Vapors



Impact of Potassium Deposition on Catalyst Performance



∇T, °C/cm

Reactor Scale Modelling Informs Regeneration Methods

6 Collaborative Manuscripts with Enabling Projects Since 2021

Adkins, B., et al., *Reaction Chemistry & Engineering*, **2021**
Unocic, K.; et al. *Microscopy and Microanalysis*, **2021**, 27, 796-798
Unocic, K., et al. *Journal of Visualized Experiments*, **2021**, 173
Lin, F., et al., *ACS Catalysis*, **2022**, 12, 1, 465-480
Downes, C. A., et al., *Chemistry of Materials*, **2022**, 34, 6255
Unocic, K. A., et al., *Microscopy and Microanalysis*, **2022**, 28, 2472

Industry Engagement Promotes Commercially Impactful R&D Outcomes

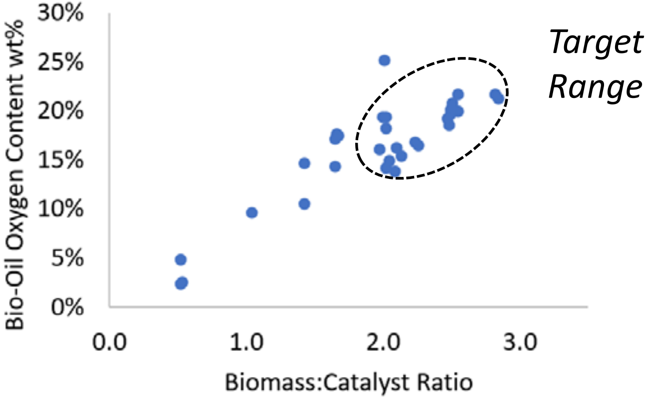
Johnson-Matthey
CRADA: Accelerating CFP catalyst and process development through collaborative R&D

ExxonMobil
CRADA: Advancing biomass pyrolysis technologies through collaborative R&D

Phillips 66
Project-level collaboration focused on hydroprocessing of biogenic oils

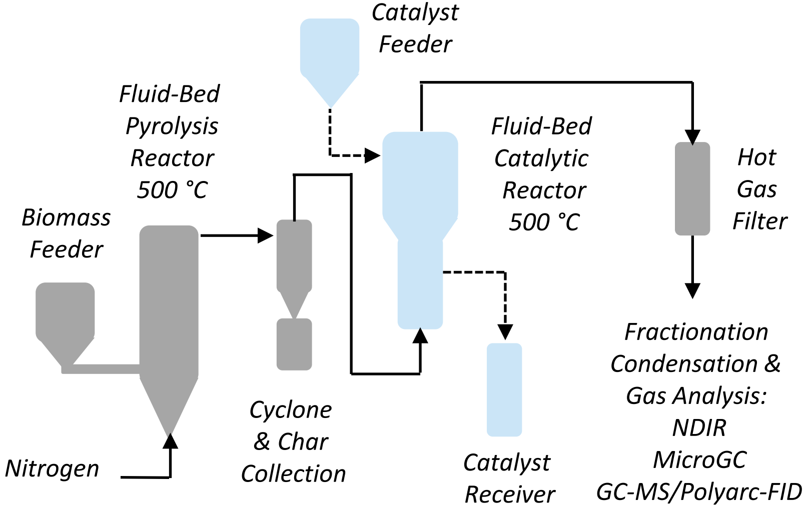
ChemCatBio Industrial Advisory Board
Consortium level guidance related to the conversion of biomass and waste carbon

Progress and Outcomes: Catalytic Pyrolysis



Progress

- Commissioned a fluidized bed reactor system with continuous pyrolysis and catalytic upgrading capabilities
- Completed an experimental campaign to determine product yield and composition at varying levels of catalytic upgrading



Feedstock: 50/50 Clean Pine + Forest Residues
Catalyst: ZSM-5 SAR 30 with Alumina Binder

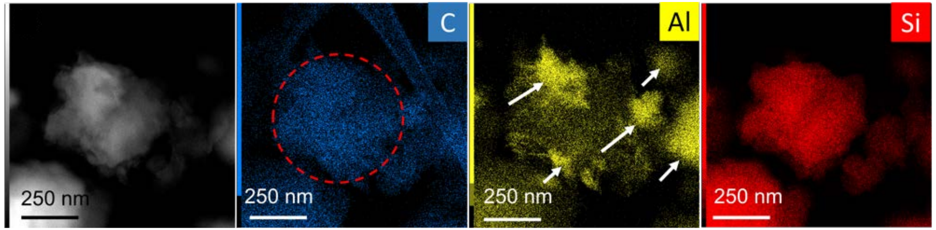
CFP-Oil Properties			
B:C, g/g	1.7	2.5	2.8
Carbon Efficiency, %	26	30	31
Oxygen Content, wt% db	17	20	22
Carboxylic Acid Number, mg KOH/g	19	29	30
Sulfur, ppm	32	32	27

Outcomes

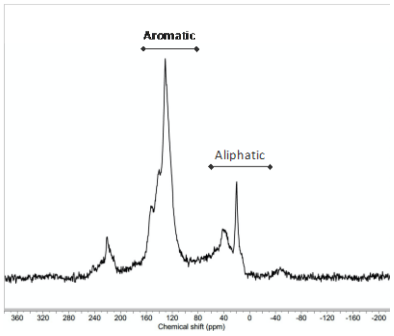
- Established benchmark product yield and composition data for CFP
- Achieved carbon balance closures of 100±5% through comprehensive analysis of the feedstock and product streams
- Generated three 500+ mL samples of CFP-oil with oxygen content ranging from 17-22 wt%

Progress and Outcomes: Catalyst Characterization

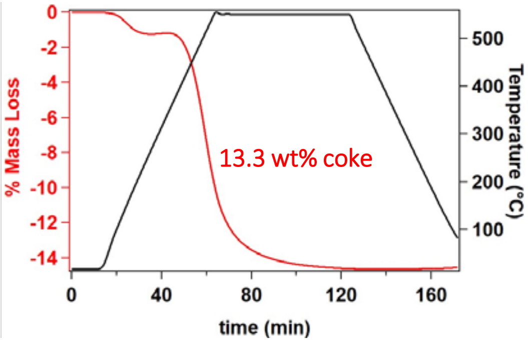
Progress: Applied physical and chemical characterization techniques to evaluate deactivation mechanisms and inform regeneration procedures for CFP catalysts



^{13}CNMR



TGA-IR



Sample	Surface Area (m^2/g)	Acid Site Density (μmolNH_3/g)
Fresh Catalyst	340	765
Post Reaction	51	516
Regenerated	336	722

Outcomes:

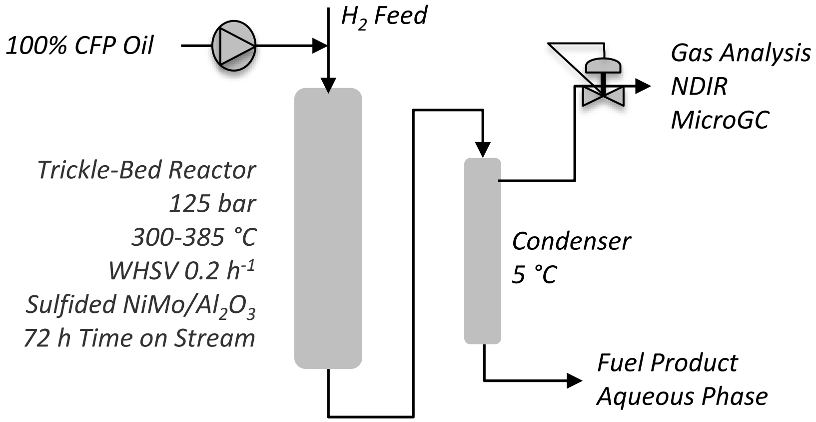
- Generated benchmark characterization data for technical zeolite catalysts with an emphasis on determining the unique composition and combustion properties of biogenic coke
- Confirmed efficacy of oxidative regeneration procedure to restore CFP catalyst active sites
- Ongoing: Pre- and post-reaction characterization of hydrotreating catalysts

Ongoing: Experimental/computational collaboration with CCB enabling projects to evaluate the composition, distribution, and combustion kinetics of biomass-derived coke (CFP, CDM, ACSC, CCPC)

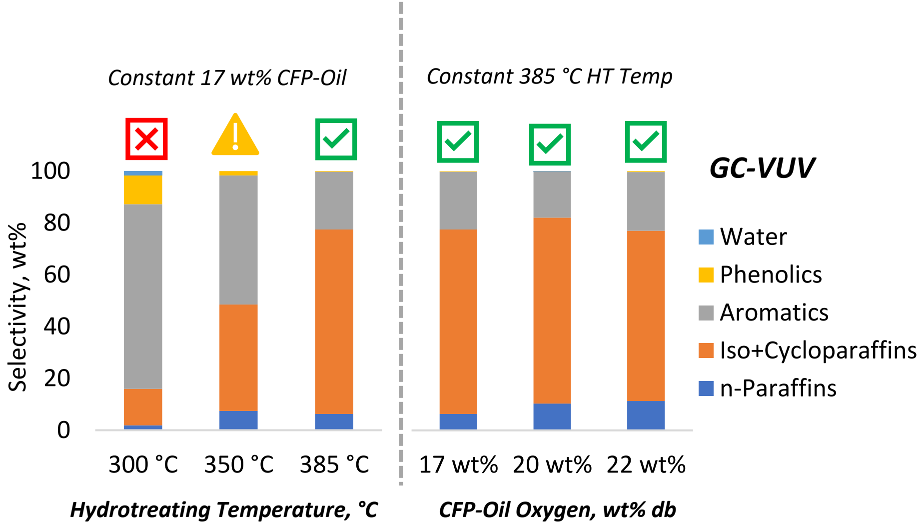
Progress and Outcomes: Hydrotreating

Progress

Completed an experimental campaign to determine the impact of CFP-oil oxygen content and hydrotreating temperature on product yield and composition



Hydrotreating Results, 385 °C			
CFP-Oil Oxygen Content	**17 wt%**	**20 wt%**	**22 wt%**
HT Carbon Efficiency	91%	92%	89%
Hydrogen Consumption	7%	8%	13%
HT-Oil Oxygen Content	<0.001 wt%	<0.001 wt%	<0.001 wt%
HT-Oil H:C Ratio	1.76	1.76	1.81



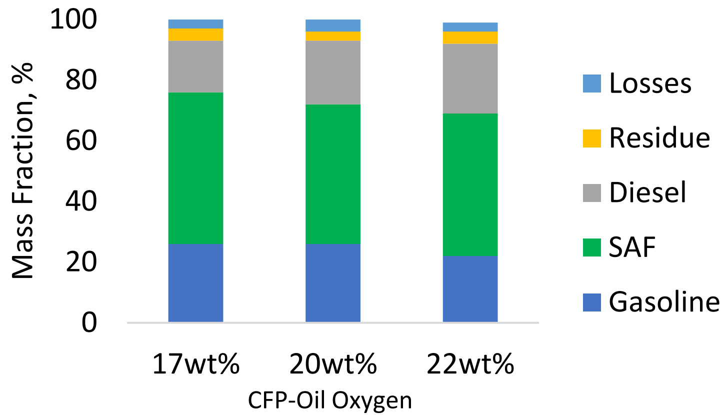
Outcomes

- Established benchmark yield and composition data for stand-alone hydrotreating
- Identified conditions suitable for the production of a high-quality hydrotreated oil with oxygen levels below detection limits

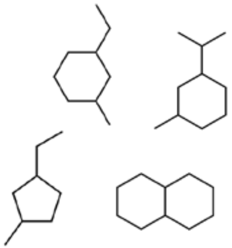
Progress and Outcomes: Fractionation and Fuel Properties

Progress

Fractionate the hydrotreated oils into gasoline, SAF, and diesel cuts using a micro-spinning band distillation system



SAF Properties				
CFP-Oil Oxygen Content, wt% dry basis		17 wt%	20 wt%	22 wt%
Density @15°C, 0.730-0.880 g/ml	✓	0.854	0.843	0.847
Flash Point >38 °C	✓	41.5	41.5	39.5
Freezing Point, <-40 °C	✓	<-80	<-80	<-80
Surface Tension 22°C, 25-29 mN/m[b]	✓	28	27	28
Lower Heating Value, >42.8 MJ/kg	⚠	42.5	42.7	42.6
D86 Simdis T10 150-205 °C	✓	162	162	161
D86 Simdis FBP <300 °C	✓	253	250	254



>75 wt% Cycloalkanes in SAF Fraction

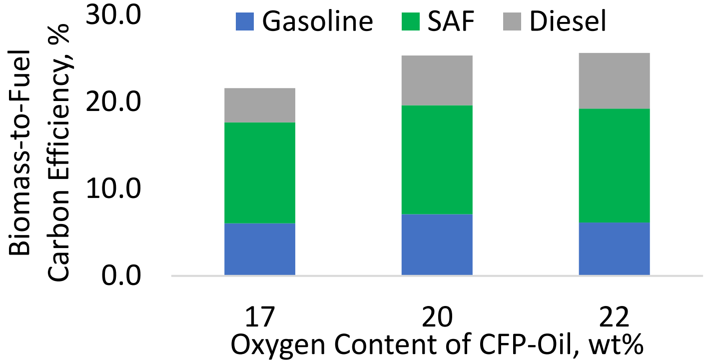
- Primary component in Jet A
- Difficult to access via other SAF pathways (HEFA, FT, ATJ)
- Increased energy density and cleaner burning than aromatics

Outcomes

- Established benchmark yield and compositional data for gasoline, diesel, and SAF fractions
- Generated a cycloalkane-rich SAF product that meets key ASTM D4054 guidelines for fuel properties

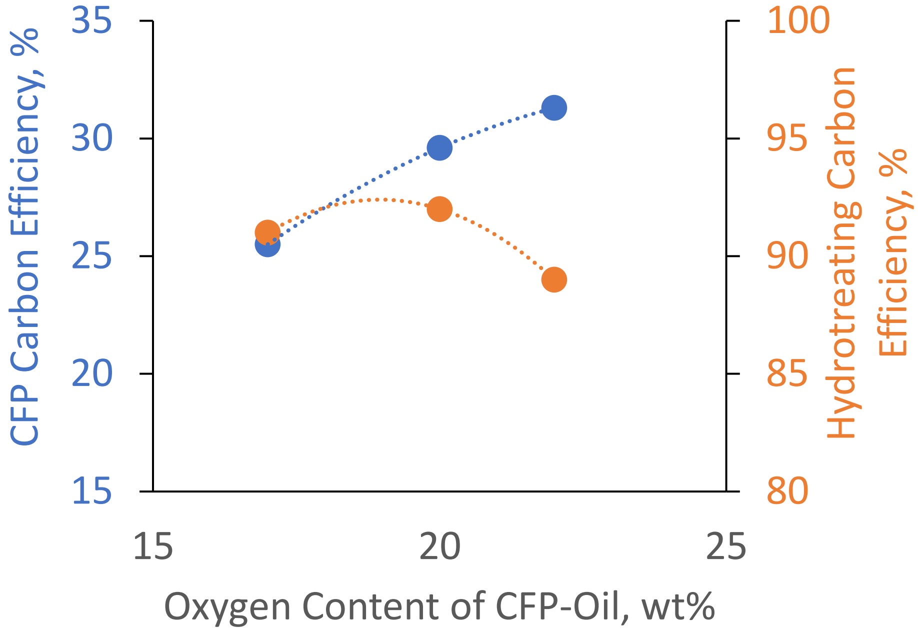
Holladay, et al (2020) , DOE/EE -2041 8292

Progress and Outcomes: Yield Structures



Progress

Determined carbon efficiencies and compositional data for the integrated biomass-to-fuels process



Preliminary TEA/LCA: Thermochemical Process Analysis 2.1.0.302

Oxygen in CFP Oil, wt%	17%	20%	22%
Estimated Minimum Fuel Selling Price	6.1-7.5	5.7-6.9	5.3-6.5
Estimated GHG Reduction, %	84	78	75

[1] Modelled *Minimum Fuel Selling Price* (MFSP) in 2016$, with ±10% variation
[2]Approximate GHG reduction over petroleum gasoline (93 g CO_2e/MJ) with coproduct electricity from usable excess energy not converted to liquid fuels.

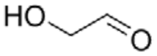
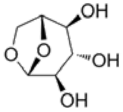
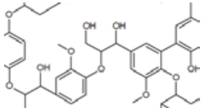
Outcomes

- Revealed correlations between process yield and bio-oil oxygen content for CFP and hydrotreating steps
- Provided experimental data to support technoeconomic and lifecycle analysis

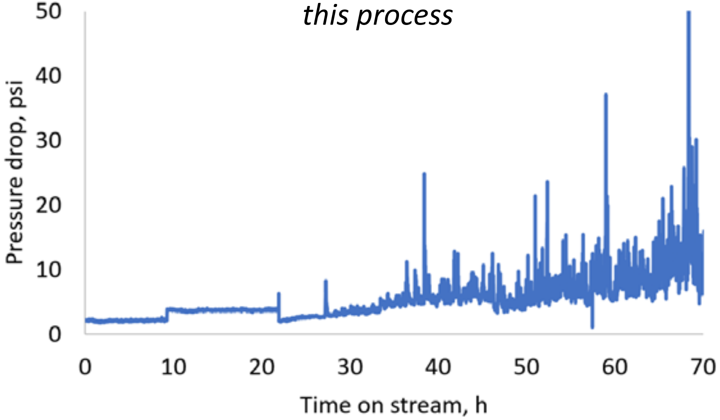
Progress and Outcomes: Critical Material Attributes

Progress

Identified CFP-oil compound classes with potential links to system plugging during hydrotreating

Compound Class	Proposed Plugging Mechanisms	Critical Material Attribute (CMA)	Detection Methods
Reactive Carbonyls	Condensation Reactions	 Hydroxyacetaldehyde	GCMS
Anhydrosugars	Cross-Linking & Gelation	 Levoglucosan	GCMS
High Molecular Weight Oligomers	Viscosity & Nucleation	 > 500 Dalton Heavies	GPC

Plugging of the hydrotreater represents an important risk and primary failure mode for this process



Data were collected during experiments performed under a collaborating project: 3.2.1.005 "Advancing the Development of Biofuels for the Maritime Sector"

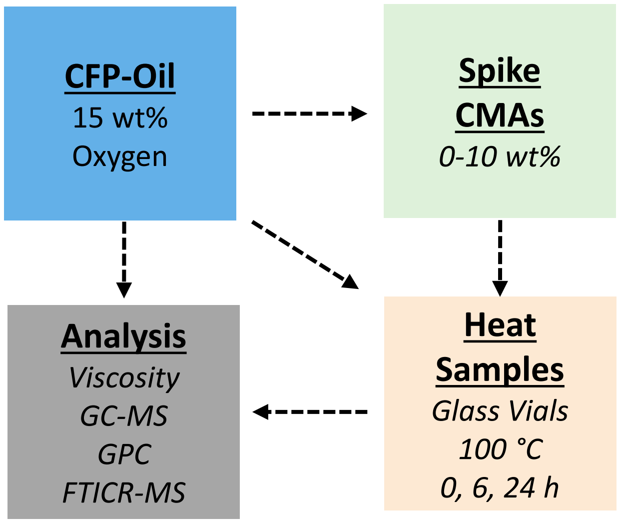
Outcome

Down-selected three critical material attributes (CMAs) for further analysis via chemical spiking and thermal stability experiments

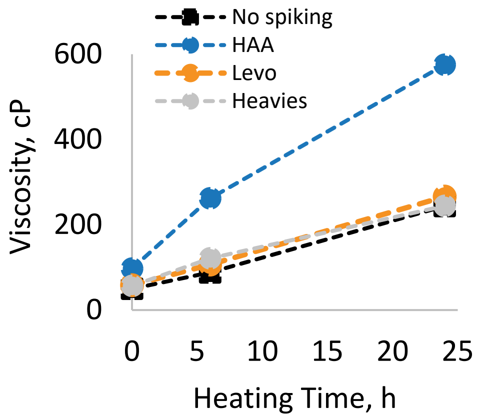
Progress and Outcomes: Critical Material Attributes

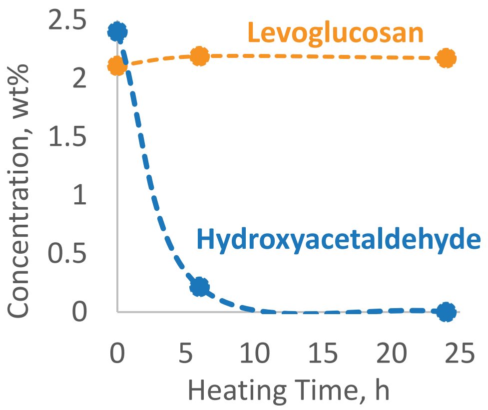
Progress

Completed a series of chemical spiking experiments to evaluate the impact of CMA concentration on thermally-driven increases in viscosity and molecular weight



High Throughput; < 10 mL Material Requirements





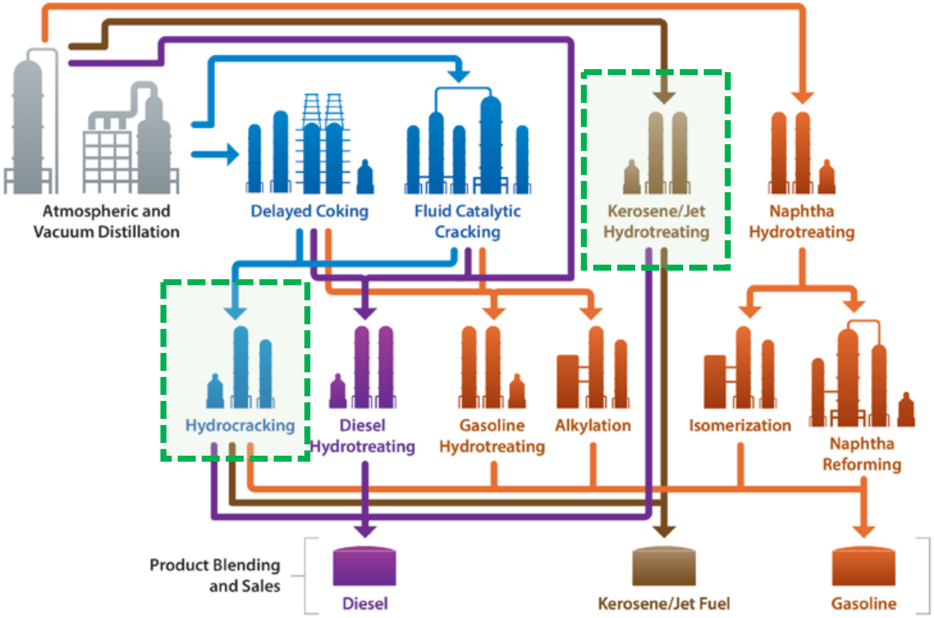
Outcomes

- Identified correlations between hydroxyacetaldehyde (HAA) concentration, viscosity, and molecular weight growth
- Identified reactive carbonyls as a primary species of concern

Ongoing: Follow-on thermal stability experiments and statistical analysis to further probe correlations between concentration, viscosity, and molecular weight for reactive carbonyls

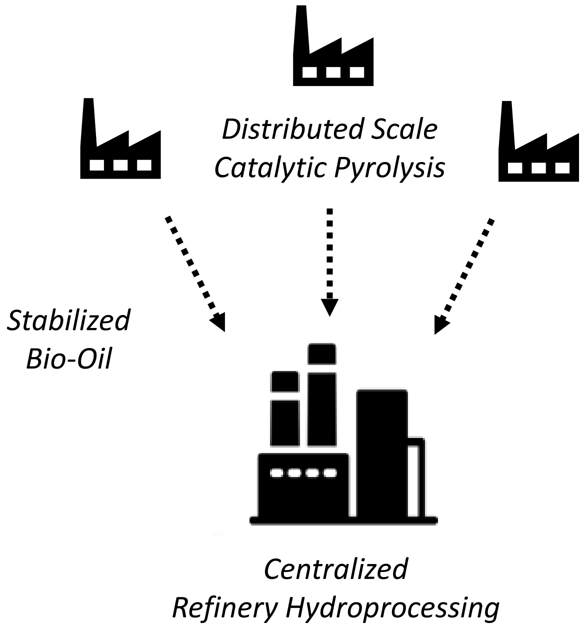
Impact: Pathway to Market

Refinery Hydroprocessing



- Refinery integration points with direct pathway to SAF
- 100+ BGPY current US hydrotreating/hydrocracking capacity
- Leverages existing workforces and institutional knowledge
- Potential for blending multiple biogenic and fossil streams

Hub and Spoke Production Model



US 2022 Refinery Capacity Summary	
Diesel Hydrotreater	**70 BGPY**
Hydrocracker	**37 BGPY**

https://www.eia.gov/petroleum/refinerycapacity/

Impact: Scientific Knowledge

12 Peer Reviewed Publications Since 2021
See Supporting Slides

12 External Presentations Since 2021
See Supporting Slides

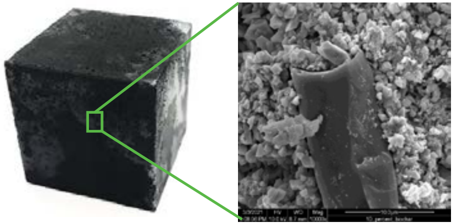
2 Issued Patents
6 Pending Applications
Novel catalysts, processes, and co-products

Synthesis and Characterization of Technical Catalysts
CCB Focus Area

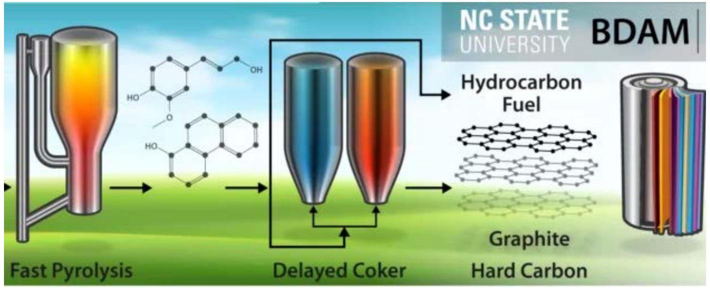
Spin-Off Projects Since 2021

High-Performing Carbon-Negative Concrete Using Low-Value Byproducts From Biofuels Production

Carbon Upcycling
Colorado School of Mines
University of Colorado Boulder



Biocrude Derived Anode Materials for Lithium-Ion Batteries



NC State University
Birla Carbon
Yale University
Battery Innovation Center
Ensyn Corporation

Summary

Approach

- **Supports BETO SAF production goals** through integrated pathway development that reduces technical risk, addresses feedstock constraints, and informs process scale-up
- Active project management **with integrated risk identification and mitigation efforts** as well as a **dedicated DEI effort**
- Established avenues for coordination with **multi-lab collaborators** and **industrial partners**

Progress and Outcomes

- Established **benchmark yield structures and compositional data** for three levels of CFP upgrading
- Provided first-of-its kind **evaluation of SAF fuel properties** and identified opportunities to produce a cycloalkane-rich product stream
- Generated benchmark **characterization data for technical zeolite catalysts** with an emphasis on determining the unique composition and combustion properties of biogenic coke
- Established **bio-oil critical material attributes** to mitigate the risk of plugging during down-stream hydroprocessing

Impact

- Informed the development of **TEA/LCA process models** through the generation of high-quality data sets
- Generated **broadly enabling scientific knowledge and multiple spin-off projects** to advance the state of technology
- Aligned with **a promising pathway to market** that addresses an emerging demand for biogenic oils

Acknowledgements

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Kinga Unocic (ORNL)
Biva Talukdar (ORNL)

ChemCatBio
Chemical Catalysis for Bioenergy

U.S. DEPARTMENT OF ENERGY | Energy Efficiency & Renewable Energy
BIOENERGY TECHNOLOGIES OFFICE

Questions

chemcatbio.org

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This research was supported by the DOE Bioenergy Technology Office under Contract no. DE-AC36-08-GO28308 with the National Renewable Energy Laboratory

Additional Slides

Quad Chart

Timeline
10/1/21- 9/30/24

	FY22 Costed	Total Award
DOE Funding	*1.7 MM + 300 k Carryover*	*2.0 MM*

TRL at Project Start: 3
TRL at Project End: 4

Project Goal
The objective of this project is to produce sustainable aviation fuel (SAF) from lignocellulosic biomass through catalytic fast pyrolysis (CFP) and hydrotreating

End of Project Milestone
Perform a series of integrated CFP and hydrotreating experiments to achieve a ≥20% relative increase in carbon efficiency to jet-range products compared to the baseline established in FY22. The CFP-oil will meet the CMAs and tolerance ranges identified previously, and the SAF fraction will meet ASTM4054 specifications for density, viscosity, boiling point, heating value, flash point, freeze point, and volatility. Hydroprocessing durability will be demonstrated via experiments performed in a realistic environment for a minimum of 500 h time on stream. Technoeconomic and lifecycle analysis will be performed to determine the minimum jet selling price and demonstrate a ≥70% reduction in GHG emissions compared to fossil-based pathways.

Funding Mechanism
FY22 Lab Call

Project Partners
- ExxonMobil (CRADA)
- Johnson Matthey (CRADA)
- Phillips 66 (Hydrotreating Advisors)

TEA/LCA– Additional **Preliminary** Information

Financial assumptions consistent with FY20-21 State of Technology report*

Description of Assumption	Assumed Value
Cost year	2016
Internal rate of return on equity	10%
Plant financing by equity/debt	40%/60% of total capital investment
Plant life	30 years
Income tax rate	21%
Interest rate for debt financing	8.0% annually
Term for debt financing	10 years
Working capital cost	5.0% of fixed capital investment (FCI) (excluding land purchase cost)
Depreciation schedule	7-year MACRS [a] schedule [9]
Steam plant depreciation	20-year MACRS schedule [9]
Construction period (spending schedule)	3 years (8% Y1, 60% Y2, 32% Y3)
Plant salvage value	No value
Startup time	6 months
Revenue and costs during startup	Revenue = 50% of normal Variable costs = 75% of normal Fixed costs = 100% of normal
Onstream percentage after startup	90% (7,884 operating hours per year)

[a] Modified accelerated cost recovery system

***Reference:** https://www.nrel.gov/docs/fy21osti/80291.pdf; **GGE:** gallon gasoline equivalent

CFP Oil Oxygen Contents →	17%	20%	22%
Feedstock cost* ($/dry US ton)	67	67	67
Capital investment total (million $)[1]	707	728	740
Chemical coproducts	No	No	No
Electricity credit (cents/GGE)	78	57	45
MFSP[2,3] ($/GGE)	6.1-7.5	5.7-6.9	5.3-6.5
GHG Reduction over gasoline (%)	84	78	75

[1] Capital and operating costs based on the *ex situ* case in the 2015 design report (https://www.nrel.gov/docs/fy15osti/62455.pdf). Current model at lower pressure than 2015 report, leading to larger equipment sizes and higher capital costs for CFP equipment.

[2] Model yields considered hydrotreated products heavier than diesel and other losses during product distillation as fuel products because the heavy ends from distillation are hydrogenated and stabilized, and can likely be used as marine fuel

[3] CFP catalyst replenishment is based on values in the 2015 design report (based on typical FCC values). The reactor heat balance is used to determine the flow rate. With a less active catalyst (reflected by different biomass to catalyst ratios in our experiments), cheaper catalyst diluents can allow CFP catalyst cost reduction.

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