Catalytic Upgrading of Pyrolysis Vapors
2.3.1.314

Mike Griffin
2021 BETO Peer Review
### Project Overview: ChemCatBio

#### 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)
Catalytic fast pyrolysis is a versatile technology pathway for the direct liquefaction of biomass and waste carbon sources

- Potential for high carbon yields to fuel blendstocks
- Ability to control the product slate through vapor phase catalytic upgrading
- Opportunities for co-processing using existing refinery infrastructure

Advantage over non-catalytic fast pyrolysis:
Generates a stabilized bio-oil with lower acidity, lower viscosity, and reduced oxygen content

Advantage over petroleum fuels:
Reduces greenhouse gas emissions and qualifies for advanced regulatory incentives

Project Overview

Project Objectives

• Maximize yields and minimize costs through integrated catalyst and process development

• Expand market responsiveness by developing routes to produce co-products

• Provide experimental data to inform process modelling and scale-up activities

• Support BETO goals of meeting 2022 verification cost and carbon intensity targets: ≤$3/GGE MFSP, ≥60% reduction in GHG emissions.

Vision:
A circular carbon economy in which biomass and waste carbon sources can be readily recycled into renewable fuels, chemicals, and materials.

GGE: Gasoline gallon equivalent, MFSP: Minimum fuel selling price, GHG: Greenhouse gas
The management plan leverages an integrated task structure that spans key elements of CFP catalyst and process development.

**Task 1: Project Management**
Lead: Mike Griffin

**Task 2: Catalyst Synthesis and Characterization**
Lead: Susan Habas

**Task 3: Performance Evaluation**
Lead: Calvin Mukarakate

**Task 4: Catalyst and Process Durability**
Lead: Matt Yung

**Task 5: CFP-Oil Production using FCC-Systems**
Lead: Kim Magrini

**Task 6: CFP-Oil Fractionation**
Lead: Kristiina Iisa

**Task 7: Co-Product Formation**
Lead: Mark Nimlos

The implementation strategy combines advancements in foundational science and applied engineering to meet overarching project objectives.

**Integrated risk assessment and stage-gates** to track progress and inform strategy.

**Risk Assessment:** March 2020 Comprehensive Pathway Review

**Stage Gate:** June 2020 Verification Go No-Go Decision Point
Management Plan: Collaboration

Collaboration across projects, consortia, and industry partners promotes integrated R&D

**Feedstock-Conversion Interface Consortium**
Establishing critical feedstock attributes and pre-processing strategies for FP and CFP

**ChemCatBio Enabling Projects**
Improving catalyst performance and durability with support from enabling projects

**Consortium for Computational Physics and Chemistry**
Informing process development and scale-up through atomistic, particle, and reactor-scale modeling

**ChemCatBio Industrial Advisory Board**
Guiding R&D towards commercially impactful outcomes

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

**Johnson-Matthey CRADA**
Advancing CFP catalyst and process development through collaborative R&D

**Feedstock Supply and Logistics**
Jordan Klinger Biomass Preprocessing

**Manganese and Coordination**
Trevor Smith BETO TM

**Hydrotreating**
Yuan Jiang Hydrotreating Analysis

**Catalyst Development and Characterization**
Susan Habas ACS Lead
Fred Baddour Engineering of Scale Up

**Hydroprocessing**
Huimin Wang Hydroprocessing 2.5.4.501

CRADA: Cooperative Research and Development Agreement

Streamlined communication enabled through the development of a multi-lab organizational structure
Early efforts within this project focused on benchmarking performance for several CFP catalysts and reactor configurations.

**In-Situ CFP Metal Oxide Catalysts**
- Circulating Catalyst
- Low capex requirements
- Harsh upgrading environment

**Ex-Situ Entrained Bed CFP Zeolite Catalysts**
- Controlled upgrading environment
- Higher capex required

**Ex-Situ Fixed Bed CFP Metal-Acid Catalysts**
- More diverse catalysts and chemistries possible
- Longer catalyst lifetimes required
A down-selection was informed by a first-of-its kind performance evaluation under a controlled set of conditions:

Due to the comparatively high CFP yields, low oil-oxygen content, and improved overall carbon efficiency, the ex-situ fixed bed CFP approach was down-selected as a leading pathway for the BETO 2022 Verification.

<table>
<thead>
<tr>
<th>Process</th>
<th>In-situ CFP</th>
<th>Ex-situ Riser CFP</th>
<th>Ex-situ Fixed-Bed CFP</th>
</tr>
</thead>
<tbody>
<tr>
<td>Catalyst (Conditions)</td>
<td>Red Mud (400°C)</td>
<td>ZSM-5 (550°C)</td>
<td>2 wt% Pt/TiO₂ (400°C, H₂ co-feed)</td>
</tr>
<tr>
<td>Reactor</td>
<td>Utah State’s Fluidized Bed Pyrolyzer</td>
<td>NREL’s 2” Fluidized Bed Pyrolyzer + Catalytic Upgrading System</td>
<td></td>
</tr>
<tr>
<td>CFP Carbon Efficiency* (%)</td>
<td>42</td>
<td>33</td>
<td>42</td>
</tr>
<tr>
<td>CFP O Content (wt%)</td>
<td>28</td>
<td>17</td>
<td>17</td>
</tr>
<tr>
<td>HT Carbon Efficiency* (%)</td>
<td>85</td>
<td>96</td>
<td>93</td>
</tr>
<tr>
<td>HT Oil O Content (wt%)</td>
<td>0.9</td>
<td>1.2</td>
<td>0.4</td>
</tr>
<tr>
<td>Overall Carbon Efficiency* (%)</td>
<td><strong>36</strong></td>
<td><strong>32</strong></td>
<td><strong>38</strong></td>
</tr>
</tbody>
</table>

*Normalized carbon efficiencies based on >500mL of CFP oil generated.
**Standard Conditions**

- **Feedstock:** Loblolly Pine
- **Catalyst:** 0.5 wt% Pt/TiO₂
- **Support:** 1.7 mm TiO₂ Pellets
- **Pyrolysis Temperature:** 500 °C
- **Upgrading Temperature:** 435 °C
- **Catalyst Mass:** 100 g
- **WHSV:** 1.4 g biomass/gcat*h
- **Near Atmospheric Pressure**
- **Hydrogen Concentration:** 83%
- **Biomass:Catalyst Ratio:** 3-13.2

*Griffin, M. et al., Energy Environ Sci, 2018*

**Approach: 2018 Baseline**

Integrated experiments demonstrate potential for high carbon yields using 0.5 wt% Pt/TiO₂

Data are consistent with literature benchmarks performed under hydrodeoxygenation conditions

*K. Wang, et al., Green Chem. 19 2017*

4x decrease in Pt loading achieved by using advanced synthesis techniques to maximize metal dispersion
With the potential benefits of the chemistry established, research in FY19-FY20 targeted technical objectives associated with reducing risks, diversifying feedstocks, and informing scale up:

- Reducing analytical uncertainty by improving material balances
- Assessing catalyst and process durability
- Increasing catalyst cycle length and regeneration efficiency
- Demonstrating compatibility with waste feedstocks (e.g., forest residues)
- Informing process scale up and supporting BETO Verification goals
Impact Section Will Follow Progress and Outcomes (Slides 20-21)
Progress and Outcomes: Improved Analytics

**Progress:** modifications to the system and methods resulted in improved carbon balance closure and reduced uncertainty in the product distribution.

<table>
<thead>
<tr>
<th></th>
<th>FY18</th>
<th>FY19</th>
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<tbody>
<tr>
<td>CFP Carbon Balance (%)</td>
<td>88</td>
<td>100</td>
</tr>
<tr>
<td>CFP Oil Carbon Yield</td>
<td>45</td>
<td>35</td>
</tr>
<tr>
<td>CFP Oil Oxygen (wt%, dry)</td>
<td>19</td>
<td>15</td>
</tr>
<tr>
<td>HT Carbon Yield (%)</td>
<td>89</td>
<td>95</td>
</tr>
<tr>
<td>CFP + HT Carbon Yield (%)</td>
<td>36</td>
<td>33</td>
</tr>
<tr>
<td>Co-Product Credit</td>
<td>-</td>
<td>$0.52</td>
</tr>
<tr>
<td>MFSP, $/GGE</td>
<td>3.50*</td>
<td>3.33</td>
</tr>
</tbody>
</table>

*This added level of analytical detail resulted in downward revisions to the 2018 normalized CFP oil carbon yield and increase in MFSP to $3.80

**Co-Product Opportunity:** high yields were observed for acetaldehyde, acetone, and 2-butanone.

Preliminary model compound data demonstrate acetone adsorption capacity. Ongoing research aims to confirm these results under realistic reaction conditions.

**Outcome:** Reduction in risk and analytical uncertainty, **$0.30/GGE** increase in MFSP, potential for ~**$0.50/GGE** reduction through valorization of acetone and 2-butanone.
Progress and Outcomes: Process Durability

Progress: integrated experiments performed for 100+ reaction cycles reveal minimal impact on yields, oil-quality, and product composition

- Standard Conditions
  - Feedstock: Loblolly Pine
  - Catalyst: 0.5 wt% Pt/TiO₂
  - Pyrolysis Temperature: 500 °C
  - Upgrading Temperature: 435 °C
  - Catalyst Mass: 100 g
  - WHSV: 1.4 g biomass/gcat*h
  - Near Atmospheric Pressure
  - Hydrogen Concentration: 83%
  - Biomass:Catalyst Ratio: 3

- Progress:
  - Integrated experiments performed for 100+ reaction cycles reveal minimal impact on yields, oil-quality, and product composition

- Outcome:
  - Improved confidence in catalyst and process durability, reduced risk for process model inputs, and support for technology transfer efforts

> 10 L of CFP oil was distributed to support research across BETO programs

*Data gaps are from experiments performed under non-standard conditions*
Progress and Outcomes: Increased Cycle Length

**Progress:** reducing the size of the catalyst support reveals potential for improved deoxygenation activity and increased cycle length

### Conditions
- **Feedstock:** Loblolly Pine
- **Catalyst:** 0.5 wt% Pt/TiO₂
- **Pyrolysis Temperature:** 500 °C
- **Upgrading Temperature:** 435 °C
- **Catalyst Mass:** 100 g
- **WHSV:** 1.4 g biomass/gcat*h
- **Near Atmospheric Pressure**
- **Hydrogen Concentration:** 83%
- **Biomass:Catalyst Ratio:** 3-12

**2018 SOT 1.7 mm Pellets**

- **FY18 SOT:** catalyst deactivates in 2-3 h

**2019 SOT 0.5 mm Spheres**

- **FY19 SOT:** catalyst can be run for 7+ hours

**Outcome:** 3.5 MM reduction in capital costs and improved process efficiency
Progress and Outcomes: Waste Feedstocks

**Standard Conditions**
- Feedstock: Loblolly Pine + Forest Residues
- Catalyst: 0.5 wt% Pt/TiO₂
- Pyrolysis Temperature: 500 °C
- Upgrading Temperature: 435 °C
- Catalyst Mass: 100 g
- WHSV: 1.4 g biomass/gcat*h
- Near Atmospheric Pressure
- Hydrogen Concentration: 83%
- Biomass:Catalyst Ratio: 3

**Progress:** reaction testing data demonstrates minimal impact of waste feedstocks on carbon yield or oil quality

**Clean Pine**
- Debarked stem-wood

**Forest Residues**
- Harvest waste including bark, needles, branches

**Outcome:** 20% reduction in feedstock costs, translating to a **$0.33/GGE** improvement in MFSP

**Ongoing Research:** establish critical feedstock attributes for CFP. FCIC: 1.2.2.804
Progress and Outcomes: Tracking Inorganic Deposition

**Progress:** catalyst characterization after reaction with forest residues tracks potassium deposition at the leading edge of the catalyst bed.

Experiments performed with a 50:50 wt% blend of clean pine and forest residues for a cumulative time on stream of 32 h. Dark field STEM images and EDS maps indicate well-dispersed K on the surface of the post-reaction samples from the top of the bed.

**Ongoing Research:**
- CDM: 2.5.4.501
- ACSC: 2.5.4.303/304/305

**ICP-OES**

- Potassium <100 ppm as prepared
- Phosphorous <50 ppm as prepared
- Iron 40 ppm as prepared
- Calcium 58 ppm as prepared

**XPS Spectra of K 2p Region** confirm K deposition.

**Outcome:** building foundational knowledge of critical deactivation mechanisms and mitigation strategies for biomass conversion pathways.
**Progress**: collaborative development of a new simulation frameworks for multiscale modeling to inform in-silico optimization and process scale up

**Outcome**: early identification of potential process disruption at the pilot scale. Ongoing efforts target improve heat transfer capabilities through catalyst development and reactor design: \textit{CCPC: 2.5.1.301}

Pecha, B.; et al. \textit{Reaction Chemistry and Engineering}, 2020
Adkins, B. D.; et.al, \textit{Reaction Chemistry and Engineering}, Submitted
**Progress and Outcomes: Verification Go/No Go**

**Progress:** data from this project informed a comprehensive pathway review performed with an independent engineering team to serve as a scale-up stage gate for the 2022 BETO Verification.

A detailed **block flow diagram** which clearly defines all inputs/outputs for pilot scale unit operations.

A **process indicator matrix** that provides a row-by-row comparison across scales.

An overarching **risk assessment** to identify research needs and inform forward looking decision making.

**Determination:** successfully meeting the verification goals by 2022 would require simultaneous technology development and scale-up. This exceeded risk tolerances and motivated a no-go decision for the pathway.

**Needed:** additional experimental data to meet $3/GGE cost target and de-risk process scale-up.

**Outcome:** early risk assessment and proactive project management to guide decision making for the BETO 2022 Verification.
Progress and Outcomes: Project Direction

Near term research addresses technical risks and data gaps through four targeted experimental campaigns:

**Feedstock Risks**
Establish critical material attributes for CFP feedstocks and identify pre-processing requirements
*FCIC: 1.2.2.805*

**Integration Risks**
Link CFP reaction conditions to bio-oil quality and downstream processing requirements
*PSUPE: 3.4.2.302*

**Catalyst Risks**
Tailor catalyst support morphology to increase cycle length and minimize pressure drop
*ACSC: 2.5.4.303/304/305
EOS: 3.2.2.701*

**Durability Risks**
Assess durability during prolonged exposure to reaction environments
*PSUPE: 3.4.2.302*

**Assessment of Co-Product Recovery and Separation**

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**Project Direction**

**FY21:** facilitate a constructive closeout of the fixed bed CFP + standalone hydrotreating pathway

**Outcome:** communicating advancements and R&D needs through a comprehensive closeout report

**Stand Alone Hydrotreating to Fuel Blendstocks**

**FY22+:** produce application specific CFP-oil for refinery integration

**Outcome:** adapting CFP to address emerging demand for biogenic refinery feedstocks

**Co-Processing with Fossil Streams**
Impact: Pathway to Market

Opportunity

>$2 billion invested to produce renewable diesel from fats, oils, and greases (FOG)

However, the supply of FOG is limited, and further growth in this sector will be inhibited by feedstock availability

CFP can help fill this gap by proving a stable biogenic liquid for refinery co-processing

Impact: establishing a **pathway to market** that allows refiners and chemical companies to diversify feedstock sources, leverage existing capital, and reduce the cost of regulatory compliance

**CFP Co-Processing Targets:**
- Increase biogenic carbon incorporation
- Reduce carbon intensity
- Mitigate potential for process disruption

**Integration Points**
- FCC w/VGO
- TH w/SRD

**Opportunity**
- Increase biogenic carbon incorporation
- Mitigate potential for process disruption

**Target:**
- Reduce carbon intensity

**FCC:** Fluid Catalytic Cracking
**VGO:** Vacuum Gas Oil
**HT:** Hydrotreating
**SRD:** Straight Run Diesel
### Impact: Science and Partnerships

#### Impact: Development of Industrial Partnerships

- **Johnson Matthey**
  - CRADA: Catalyst Development
  - **ExxonMobil**
  - CRADA: Biomass Pyrolysis

#### Impact: Spin-Off Projects (TCF, SBIR, DOE, USDA)

- **Aditya Birla Carbon**
  - Carbon Co-Products for Energy Storage Applications
- **OBIC**
- **Marrone Bio Innovations**
  - Chemical Co-Products for Bioinsecticide Applications
- **Ensyn**
- **Luna**
  - Chemical Co-Products for Biopolymer Applications

#### Impact: Generation of Scientific Knowledge

- **14 Peer Reviewed Publications Since 2019**
  - Average Journal Impact Factor of 7
  - See Supporting Slides 26-27
- **18+ External Presentations Since 2019**
  - Spanning CFP Catalyst and Process Development
  - See Supporting Slides 28-29
- **2 Issued Patents**
- **6 Pending Patent Applications**
  - Novel catalysts, processes, and co-products

**CRADA: Cooperative Research and Development Agreement**

**TCF: Technology Commercialization Fund**

**SBIR: Small Business Innovation Research**
Acknowledgements

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Zach Mills (ORNL)  
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Hao Cai (ANL)  
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**Feedstocks**
Jordan Klinger (INL)  
Danny Carpenter (NREL)

**Oil Analysis**
Jack Ferrell (NREL)  
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Renee Happs (NREL)  
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Earl Christensen (NREL)  
Lisa Fouts (NREL)

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Daniel (Miki) Santosa (PNNL)  
Suh-Jane Lee (PNNL)  
Igor Kutnyahov (PNNL)  
Douglas C. Elliott (PNNL)  
Kristiina Lisa (NREL)
Management

• **Clear management plan** with implementation strategy that advances foundational science and applied engineering
• **Established avenues for collaboration** including a well-defined multi-lab organizational structure to streamline communications
• **Active project management** through integration of risk identification and mitigation (comprehensive pathway review + go/no-go)

Approach

• **Advances the state-of-the-art** through innovative catalyst and process development
• **Builds on previous data with clear objectives** that reduce technical risk, diversify feedstock opportunities, and inform process scale-up
• **Supports BETO 2022 Verification goals** by evaluating pathways to meet cost and GHG reduction targets

Progress and Outcomes

• **Reduced analytical uncertainty** by closing carbon balances to 100 +/- 1%
• **Improved process efficiency** by achieving a 4x increase in catalyst cycle length
• **Demonstrated process durability** for 100+ reaction cycles (~275 h)
• **Demonstrated compatibility with waste feedstocks** (e.g., forest residues)
• **Identified risks and research needs for process scale up** to inform a proactive pivot for the 2022 verification

Impact

• **Generated broadly enabling scientific knowledge** (14 publications, 18+ presentations, 8 IP positions)
• **Considerable industry engagement** through partnerships across the value chain (e.g., CRADAs with Johnson Matthey and ExxonMobil)
• **Promising pathway to market** that addresses an emerging demand for biogenic refinery feedstocks
Supporting Information
**Project Quad Chart**

**Timeline**
- Project start date: October 1st, 2019
- Project end date: September 30th, 2021
- Percent complete: 44%

<table>
<thead>
<tr>
<th></th>
<th>FY20</th>
<th>Active Project</th>
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<tbody>
<tr>
<td>DOE Funding</td>
<td>3.4 MM</td>
<td>6.8 MM</td>
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**Project Goal**
Develop CFP as a versatile deconstruction technology that is compatible with biomass and waste carbon sources and enables the production of application specific bio-oils with properties that can be tailored to meet dynamic market needs.

**End of Project Milestone**
Develop refinery integration approaches and feasible co-products from catalytic fast pyrolysis pathways. Establish CFP-oil quality specifications and blend ratios for FCC and/or co-hydrotreating integration points to meet an overall minimum fuel selling price of $3/GGE in $2016 dollars.

**Project Partners**
- **Industry:** ExxonMobil, Johnson Matthey
- **Academia:** University of Southern California (FY20)

**Barriers addressed**
- **Ot-B: Cost of Production**
  - Reducing MFSP for CFP technology platform
- **Ct-F: Increasing the Yield from Catalytic Processes**
  - Developing catalysts and process operations to enhance carbon efficiency

**Funding Mechanism**
National Laboratory AOP Project


• Mukarakate, Calvin; Orton, Kellene; Kim, Yeonjoon; Dell’Orco, Stefano; Farberow, Carrie; Kim, Seonah; Watson, Michael; Baldwin, Robert; Magrini, Kimberly, “Isotopic Studies for Tracking Biogenic Carbon during Co-processing of Biomass and Vacuum Gas Oil”, *ACS Sustainable Chemistry and Engineering*, **2020**, 8(9), 2652-64.


Publications Since 2019 (2 of 2)


Presentations Since 2019 (1 of 2)

- Mike Griffin, Bruce Adkins, Brennan Pecha “Advancing Catalytic Fast Pyrolysis through Integrated Experimentation and Multi-Scale Computational Modeling” BETO ChemCatBio Webinar (virtual), January 2021


- Stefano Dell’Orco, Edoardo Miliotti, Nolan Wilson, Andrea Maria Rizzo, Kimberly A. Magrini and David Chiaramont, “Overcoming scale-up industrial barriers of hydrothermal liquefaction of lignin-rich streams: Carbon recovery from residual aqueous phase”, tcbiomassplus, October 2019, Chicago, IL.

- K. Magrini, Calvin Mukarakate, Kellene Orton, Yeonjoon Kim, Stefano Dell’Orco, Carrie A Farberow, Seonah Kim, Michael J Watson, Robert Baldwin, “Isotopic Studies for Tracking Biogenic Carbon during Co-processing of Biomass and VGO”, tcbiomassplus, October 2019, Chicago, IL.

- Braden Peterson, Chaiwat Engtrakul, Nolan Wilson, Stefano Dell’Orco, Jessica Olstad, Mike Sprague, Yves Parent, Kim Magrini, “Preconditioning Pyrolysis Vapors for Downstream Upgrading Processes via Coupled Catalytic Hot-Gas Filtration and Fractional Condensation”, tcbiomassplus, October 2019, Chicago, IL.

Presentations Since 2019 (2 of 2)


Ongoing collaborative research focuses on optimizing catalyst size and porosity using custom technical supports prepared at NREL (Engineering of Catalyst Scale Up: 3.2.2.701)

Utilizing smaller TiO₂ supports improves deoxygenation performance but increases pressure drop and necessitates the use of low L/D reactors with limited heat transfer capabilities.

Target outcome: achieve high catalyst activity while minimizing pressure drop to enable the use of reactor dimensions with improved heat transfer capabilities.
Improving hydrocarbon yields and increasing alkene selectivity using a Ga/ZSM-5 catalyst

A novel and scalable solution phase synthesis route to produce molybdenum carbide nanoparticles
Baddour, F., et al. JACS, 2020, 142, 2, 1010
Other Research: Developing Co-Product Pathways with Commercial Partners

Chemicals for Polymers

- Recyclable polymers: $1 - 3/kg
- Unsaturated polyvinyl alcohols: > $6/kg

High Value Carbon

- $5 - 10/kg

Bioinsecticides

- > $6/kg

Anodes for Lithium and Sodium Ion Batteries

- $5 - 10/kg
**Approach:** Develop modified zeolites with Johnson Matthey that target biomass conversion and are compatible with refinery fluidized catalytic cracking (FCC) catalysts; prepare CFP oils using a coupled pyrolyzer/FCC plant; evaluate catalyst impact on oil composition; assess FCC co-processability to biogenic carbon containing fuels.

**Feedstocks and Catalysts**
- Pine (baseline feed)
- Pine forest residues (FR)
- Miscanthus
- ZSM-5
- Ga/ZSM-5

**Feedstocks and ZSM-5**
FR and Miscanthus
- Increased oxygenates, reduced aromatics
Miscanthus: reduced phenolics (less lignin)

**Feedstocks and Ga/ZSM-5**
- Increased aromatics, phenols for all feedstocks
- Reduced furans and carbonyls from cellulose deoxygenation