Upgrading of C1 Building Blocks

WBS# 2.3.1.305

Daniel Ruddy
National Renewable Energy Lab
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 Goal

Develop the centerpiece technology for a market-responsive, integrated biorefinery concept based on the conversion of renewable C1 intermediates to produce a suite of fuels with improved carbon efficiency, reduced capital expense, and control of the product distribution to meet market demand.
Project Outcome

- Develop the catalyst and process for direct conversion of CO₂-rich syngas (15-20% CO₂) to high-octane gasoline and jet fuels
- Exceed the carbon efficiency of benchmark Mobil Olefins-to-Gasoline-and-Distillates (MOGD) process with lower capital expense

Key Differentiators

- Address known drawbacks for traditional syngas-to-fuels processes at smaller production scales – high capital cost, limited product quality – by focusing on mild process conditions, high yield and carbon efficiency, high-quality (high-value) fuel products
Traditional syngas to hydrocarbon fuels have known drawbacks

- Fischer-Tropsch (FT): Costly catalytic upgrading to produce quality fuels
- Methanol-to-Gasoline (MTG): Capital intensive, high aromatics content
- Mobil Olefins-to-Gasoline-and-Distillate (MOGD): Capital intensive, high number of process steps

**MFSP from biomass (2016 $)**
- FT = $3.17/GGE
- MOGD = $4.23/GGE


**Advanced upgrading technologies can reduce MFSP through reduced process complexity, reduced separations duty, higher quality fuel products**
NREL has been developing the High-Octane Gasoline (HOG) Pathway

**Key Differentiators of HOG versus MTG**
- HOG pathway yields branched alkanes, not aromatics
- Higher octane (102 vs 87), higher value fuel product
  - Alkylate versus regular-grade gasoline
- Lower severity conditions for HOG vs MTG
  - Higher yield (18% relative), higher C-efficiency
- Modeled costs for MeOH-to-HOG of $0.49–0.66/GGE compare favorably against other alcohol conv tech.
  - EtOH-to-Jet $0.89–1.19/GGE
    (L. Tao et al., *Green Chem.* 2017, 19, 1082)
1. Management

Task management integrated with CCB enabling technologies and analysis team, other BETO projects and consortia, and technology advancement opportunities

<table>
<thead>
<tr>
<th>ACSC</th>
<th>CCPC</th>
<th>CCM</th>
<th>CDM</th>
<th>TEA</th>
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Task 1 (D. Ruddy) DME/Syngas to High-Octane Gasoline and Jet Fuel

Task 2 (A. To) CO₂ Conversion to Jet Fuel

- “Constant contact” between PIs and Enabling Technology points-of-contact
- Research tasks are cooperative and synergistic
  - Focus on specific challenges for syngas versus CO₂ conversion
  - Lessons learned from each task inform the common goal of CO₂-rich syngas conversion
- TEA-informed metrified milestones and Go/No-Go to relate catalyst improvements to costs
- Utilized the TEA-informed FY19 results to inform FY20 syngas conversion research path
2. Approach – Dual Research Cycle

- **Hypothesis-driven catalyst and process development** coupled with:
  - Sophisticated catalyst characterization (*with Adv. Cat. Synthesis. & Characterization*)
  - Reactor design for cascade chemistry (*with Cons. Comp. Physics & Chemistry*)
- **TEA-informed research targets**, experimental data informs process models and TEA
2. Approach – Pathways Explored in this Project

Pathway Objectives

- Catalyst and process development for **cost reduction in the 3-step process** for dimethyl ether (DME) to high-octane gasoline (HOG) over NREL’s Cu/BEA zeolite

- Utilize **Process Intensification** to evaluate catalytic pathways that will **improve the overall carbon efficiency with reduced capital expense – 5-10% decrease in CapEx**

- **Leverages research team’s experience** with syngas, DME, and Cu/BEA catalyst

- Compare against benchmark MOGD and recent reports for syngas-to-olefins (STO) processes (e.g., OX-ZEO process)
2. Approach – Opportunities and Challenges

Similar process conditions offer the opportunity for process intensification to enable direct syngas-to-fuels through methanol in a single reactor:

- Utilize commercial, inexpensive Cu-based MeOH synthesis catalyst
- Opportunity to co-convert CO₂ with CO during MeOH synthesis
- Go/No-Go Decision in FY21 for this new, direct syngas-to-fuels pathway

Research Challenges and Critical Success Factors:
- Activate CO₂ in the presence of CO, convert to products for increased C-efficiency
- Balance multiple reactions under low severity conditions
- Maximize yield and carbon efficiency with multi-functional catalyst system that performs cascade chemistry
- Advance technology with bioenergy industry partners, TCF funding
3. Impact – High-Octane Gasoline Product

HOG product targets **growing premium gasoline fuel demand and value**

- Unlike ethanol, **gasoline product has no blend limit**

**HOG technology awarded a Technology Commercialization Fund**

$740k investment by DOE + $750k cost-share from Enerkem in 2018-2019

- Demonstrated HOG production at the **pilot scale** (20-kg\textsubscript{cat}) with **MSW-derived methanol** **for 500 h time-on-stream**
- Produced **20 L** of high-octane gasoline
- Sent to **refinery industry partners**

**Composition of High-Octane Product**

- **73.9% Triptane** (2,2,3-trimethylbutane)
- RON = 108  MON = 97

- **This TCF project was critical to generate liquid product for industry analysis**
- **Research license to commercialize NREL’s HOG technology with large energy company executed in 2020**
3. Impact – Jet-range Product

Developed a mild-condition route for olefin coupling to jet-range hydrocarbons

- Process model and TEA indicates only a minor fuel synthesis cost increase to generate this additional product (+ 3¢/GGE)

Product meets 5 key ASTM Int’l jet fuel property specifications

<table>
<thead>
<tr>
<th>Fuel Property</th>
<th>ASTM D1655 Limits</th>
<th>Coupled Olefin - Jet Values</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density (kg/m³)</td>
<td>775–840</td>
<td>783</td>
</tr>
<tr>
<td>Freze point (°C)</td>
<td>–40 max</td>
<td>–81</td>
</tr>
<tr>
<td>Viscosity (mm²/s)</td>
<td>8.0 max</td>
<td>7.6</td>
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<tr>
<td>Net Heat of Combustion (MJ/kg)</td>
<td>42.8 min</td>
<td>43.8</td>
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<tr>
<td>T10 (°C)</td>
<td>205 max</td>
<td>178</td>
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<tr>
<td>T50</td>
<td>Report</td>
<td>188</td>
</tr>
<tr>
<td>T90</td>
<td>Report</td>
<td>239</td>
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<tr>
<td>FBP</td>
<td>300 max</td>
<td>281</td>
</tr>
</tbody>
</table>

C₄-C₈ Branched OLEFINS → C₈-C₂₀ Branched JET

Diagram: Weight % vs Carbon # Range
## 4. Progress & Outcomes – Baseline in FY20

<table>
<thead>
<tr>
<th></th>
<th>MOGD Benchmark</th>
<th>“3-step” DME-to-HOG</th>
<th>“Direct” Syngas-to-HOG</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Catalyst</strong></td>
<td>ZSM-5</td>
<td>Cu/BEA</td>
<td>CZA</td>
</tr>
<tr>
<td><strong>Severity of Process Conditions</strong></td>
<td>350–400 °C 20 atm Frequent regen.</td>
<td>200–225 °C 1–3 atm Stable &gt;100h</td>
<td>200–240 °C 1–10 atm unknown</td>
</tr>
<tr>
<td><strong>Start of FY20 Metrics</strong></td>
<td>–</td>
<td>44% DME conv. 0.070 g/gcat/h</td>
<td>New Effort in FY20</td>
</tr>
<tr>
<td><em><em>Fuel Yield</em> and MFSP</em>*</td>
<td>G= 34 GGE/ton D= 27 GGE/ton $4.23/GGE</td>
<td>G= 49 GGE/ton $3.53/GGE</td>
<td>Baseline to be set in FY20</td>
</tr>
</tbody>
</table>

- FY19 TEA data **sets the stage for catalyst and process development**
  - TEA-directed research goals to target most impactful metrics
  - Comparison against benchmark MOGD process
4. Progress & Outcomes – Advancing DME-to-HOG

Overview of the pathway

<table>
<thead>
<tr>
<th>Traditional 3-step Process</th>
<th>200-225 °C</th>
<th>1-3 atm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Syngas → MeOH → Dimethyl Ether</td>
<td>Cu/BEA</td>
<td>C₄-C₈ Branched HOG</td>
</tr>
</tbody>
</table>

Differentiators versus MTG

- Catalyst – BEA vs MFI
- Product composition
  - Alkylate vs regular-gasoline
- Higher product RON – ≥95 vs 92
- Lower coking rate
- Higher yield from biomass – +18%

- Cu/BEA catalyst reduces MFSP versus commercial BEA by 20% with C₄ by-product recycle and reactivation
  - MeOH-to-HOG cost reduced by 30–35% (FY18-19)

- Coupled process models and TEA with fundamental and applied catalysis research to quantify the impact of catalyst development on process economics in the production of HOG and jet fuel from DME

**Goal:** Increase C₄ recycle efficiency to increase yield, C-efficiency and reduce MFSP *(with TC Platform Analysis)*

- Explored C₄H₁₀ recycle at higher C₄H₁₀/DME ratios that simulate **continuous recycle** in a fixed-bed reactor

- Recycle efficiency increases with C₄/DME ratio
- Shift in product C-selectivity with higher C₄/DME ratio
  - Decrease in C₄
  - Increase in C₅, C₇, C₈
  - C₄ converted to C₅+

- Modeled yield increased from 49 to 55 GGE/ton, C-efficiency from 24.8 to 26.1%
- MFSP reduced from $3.53/GGE to $3.45/GGE (34% decrease vs BEA)
  - Continuing progress towards FY22 Target of $3.30/GGE
  - MeOH-to-HOG cost reduced from $0.54/GGE (FY19) to $0.45/GGE (FY20)
4. Progress & Outcomes – Advancing DME-to-HOG

**Goal:** Determine differences in carbon speciation for BEA vs Cu/BEA leading to catalyst deactivation, develop regeneration protocol *(with Adv. Cat. Synth. & Char.)*

- Cu/BEA prevented polycyclic aromatic formation that was observed on BEA
- **Cu promoted carbon removal at 200 °C lower temperature** versus BEA
- Activity of Cu/BEA was **fully recovered using the regen procedure**

### Overview of the pathway

**Process Intensification:**

**Direct Syngas-to-Hydrocarbons**
- Co-convert CO₂, use 1 reactor

**CO₂-rich Syngas**
\[ \text{1 CO} + 0.8 \text{ CO}_2 + 2 \text{ H}_2 \]

**CZA** (CuZnO/Al₂O₃) (MeOH/DME synthesis)  
**Cu/BEA** (HOG synthesis)

**200-240 °C**  
**1-10 atm**

**C₄-C₈ Branched HOG**

#### Differentiators versus OX-ZEO
- **Catalysts**
  - CuZnO/Al₂O₃ vs ZnCrOₓ
  - BEA vs AEL
- **Intermediate**
  - MeOH vs Ketene
- **Product composition**
  - Alkylate vs Olefins

### Progress & Outcomes – Direct Syngas-to-HOG

- **CuZnO/Al₂O₃** vs **ZnCrOₓ**
- **BEA** vs **AEL**
- **MeOH** vs **Ketene**
- **Alkylate** vs **Olefins**


- **Product selectivity similar to DME-to-HOG**
- **High-octane, high-value product direct from syngas**
- **Stacked-bed outperformed mixed-bed**
- **Cu/BEA outperformed BEA**

**High-Octane Gasoline**

### Carbon Selectivity (%)

- **Direct Syngas to HOG**
- **DME to HOG reference**

**Carbon Number**

- C₁, C₂, C₃, C₄, C₅, C₆, C₇, C₈, C₉⁺
**Goals:** Achieve high C-yield to C\textsubscript{4+} products from syngas and syngas+CO\textsubscript{2}, follow C from CO\textsubscript{2} using \textsuperscript{13}CO\textsubscript{2} and mass-spectroscopy of hydrocarbon products

### Key Results from FY20

<table>
<thead>
<tr>
<th>CO\textsubscript{2} co-feed</th>
<th>No</th>
<th>Yes</th>
</tr>
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<tbody>
<tr>
<td>CO+CO\textsubscript{2} conversion (%)</td>
<td>77.3</td>
<td>27.0</td>
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<tr>
<td>C\textsubscript{4+} HC yield (%)</td>
<td>44.9</td>
<td>23.8</td>
</tr>
<tr>
<td>CO\textsubscript{2} C-selectivity (%)</td>
<td>38.8</td>
<td>28.4</td>
</tr>
<tr>
<td>Total HC Prod. ( (\text{g}<em>{\text{HC}} \cdot \text{g}</em>{\text{Cu/BEA}}^{-1} \cdot \text{h}^{-1}) )</td>
<td>0.10</td>
<td>0.054</td>
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</table>

- Exceeds initial 10% target
- Greatly exceeds 2-12% in STO literature
- Decrease suggests activation
- Comparable to DME feed

**Mass spectra of major hydrocarbon products**

- High single-pass C\textsubscript{4+} yield achieved with co-fed CO\textsubscript{2}
- Mass spectra peaks at \( m/z+1 \) with \textsuperscript{13}CO\textsubscript{2} provide critical data for CO\textsubscript{2} conversion into desired products

### 4. Progress & Outcomes – Summary

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| Severity of Process Conditions | 350–400 °C 20 atm
Frequent regen. | 200–225 °C 1–3 atm
Stable >100h, Multiple Regens demonstrated | 200–240 °C 1–10 atm
Stable >50 h |
| Current Metrics | –                   | 44% DME conv.
0.094 g/gcat/h | High-yield to C4+ products, CO2 incorporation |
| Fuel Yield* and MFSP | G= 34 GGE/ton
D= 27 GGE/ton
$4.23/GGE | G= 55 GGE/ton
$3.45/GGE | Baseline set in FY20, Compared at FY21 Go/No-Go |

- Continued process advancement and cost reduction of the 3-step process achieved
- Catalyst performance in direct process exceeded initial targets
- FY21 Go/No-Go (June 2021) will compare processes to inform future research
4. Progress & Outcomes – CO$_2$-to-HCs (Task 2)

**Goals:** Separately explore approaches for conversion of CO$_2$ to hydrocarbons

1. Stacked-bed Approach – informs and supports analogous direct syngas-to-HOG pathway

- Product selectivity favors C$_{4-5}$ hydrocarbons
- Great building blocks for C$_{8-20}$ jet fuel

2. New Materials Approach – opportunity for new pathway to olefins for jet fuel

- New synthetic method to prepare colloidally-stable $\alpha$-MoC$_{1-x}$ nanoparticles developed with ACSC Project
- 2X greater per-site activity in CO$_2$-to-Hydrocarbons AND 2X greater selectivity to C$_{2+}$ hydrocarbons vs bulk $\alpha$-MoC$_{1-x}$

Determine the effect of CO$_2$/CO ratio on catalyst performance and product selectivity in the direct syngas-to-HOG reaction
- Reduce CO$_2$ selectivity
- Quantify gasoline versus olefins for jet product yield

Utilize in situ spectroscopy to compare surface carbon speciation from syngas versus DME
- Develop an efficient catalyst regeneration protocol for the CZA+Cu/BEA catalyst system

**FY21 Go/No-Go:** Use comparative TEA for DME-to-HOG versus direct syngas-to-HOG to determine the process model for State-of-Technology (SOT) reports going forward
- Complete the FY21 SOT based on Go/No-Go process choice

**FY22:** Demonstrate 200 h time-on-stream for direct conversion of CO$_2$-rich syngas to HOG in a single reactor
- Use experimental data in process models to target 31.8% C-efficiency
## Timeline
- 10/01/2019
- 09/30/2022

<table>
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<th>FY20</th>
<th>Active Project</th>
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<tr>
<td>$1.6 M</td>
<td>$4.8 M</td>
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## DOE Funding

### Project Goal
Develop the centerpiece technology for a market-responsive, integrated biorefinery concept based on the conversion of renewable C1 intermediates to produce a suite of fuels with improved carbon efficiency, reduced capital expense, and control of the product distribution to meet market demand.

### End of Project Milestone
Demonstrate 200 h time-on-stream with 14% relative improvement in C-efficiency for direct conversion of CO$_2$-rich syngas to high-octane gasoline in a single reactor using the developed catalyst and process technology.

### Project Partners
- Univ. of Minnesota, Prof. Aditya Bhan ($99k)

### Barriers addressed

**Ct-F: Increasing the Yield from Catalytic Processes**
- Developing catalysts that enable processes with higher carbon efficiency and yield

**Ct-G: Decreasing the Time and Cost to Develop Novel Industrially Relevant Catalysts**
- Developing catalysts that meet cost and performance targets assessed on a year-by-year basis

### Funding Mechanism
ChemCatBio Consortium
AOP Lab Call 2019
Project Goal

Develop new, low-severity catalytic upgrading technologies for renewable C1 building blocks to high-value fuels that address the shifting gasoline/distillate demand.

Approach

- Process intensification with multi-functional catalysts to perform selective, cascade reactions, leading to low operating costs, high C-yields, and high C-efficiency.
- Interdisciplinary, collaborative approach within ChemCatBio leveraging enabling technologies.

Impact

- Demonstrated technology transfer with the bioenergy industry (e.g., TCF with Enerkem, research license) to reduce risk of commercialization.
- Patented intellectual property, and published results in top-tier peer-reviewed journals.

Research Progress & Outcomes

- Significant increase in yield and reduction in MFSP demonstrated in DME-to-HOG technology with Cu/BEA catalyst.
- Direct conversion of CO2-rich syngas-to-HOG demonstrated with high single-pass C4+ yield.
- Evidence for CO2 incorporation into hydrocarbon products using 13CO2.
Acknowledgements

NREL Catalyst Development Team
Anh To               Connor Nash
Jesse Hensley       Carrie Farberow
Joshua Schaidle     Daniel Dupuis
Fred Baddour        Susan Habas
Nicole LiBretto     Claire Nimlos
Qiyuan Wu           Martha Arellano-Trevino
Matthew Yung        Andy Young
Seth Noone          Jason Thibodeaux
Jacob Miller        Rianna Martinez

TEA Team
Abhijit Dutta (NREL)
Eric Tan (NREL)
Kylee Harris (NREL)

Collaborators
Ted Krause (ANL)
Kinga Unocic (ORNL)
Jeffrey Miller (Purdue)
Earl Christensen (NREL)
Bruce Adkins (ORNL)
Aditya Bhan (Minn.)
The path to catalyst deployment is slow and difficult.

ChemCatBio is accelerating the catalyst and process development cycle.

dan.ruddy@nrel.gov

ChemCatBio
Chemical Catalysis for Bioenergy

NREL/PR-5100-79306
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 end-of-year goal, where we will explore deactivation and regeneration in all three of our oxygenate conversion pathways.
- This project is making great progress already by delivering a promising pathway with several others to follow. This is the type of technology platform pipelining that BETO needs to continue funding in order to remain an innovative leader in bioenergy technology for the future. These types of processes that are rooted in alcohol conversion over modified zeolite chemistry will accelerate the progress toward the 2022 target. The information received from TEA and the CCPC make this project approach very robust. The team should stay vigilant and not trivialize the oligomerization chemistry and related unit operations required to drive the carbon-carbon bond formation to distillate-range material.
  - Although our focus is on the catalyst development for oxygenate conversion to versatile hydrocarbon intermediates, the reviewer is correct that the oligomerization cannot be trivialized. Due to time limitations, these results were not presented. However, this is an active area of research in the project, and based on these comments, it will remain as such.
- Scaling is an important consideration on this project. It would be beneficial to evaluate modular processes as well and evaluate how synthetic catalysts work on large-scale processes.
  - In addition to our on-going efforts around catalyst reusability, the reviewer presents a useful suggestion to consider varying scales and modular processes. Our initial assessment of smaller scales suggested less favorable process economics, as typically associated with small-scale gasification technologies. However, we acknowledge that the opportunity to utilize renewable carbon sources may someday favor modular systems, and we will consider how our technology scales.
Publications


Patents


• “High-octane synthetic fuels” US Patent Application 17/098,785, November 16, 2020
Presentations

- **Invited:** Ruddy, D.A. “Catalysis to enable high-octane gasoline within a market-responsive bio-refinery concept”. Presented at the 28th International Materials Research Congress, Cancun, Mexico, August 19, 2019.


Commercialization

- Research license to commercialize NREL high-octane gasoline technology, executed Jan 2020.
Methanol to HOG within a Market-Responsive Biorefinery Concept Enabled by Catalysis

D. Ruddy, J. Hensley, C. Nash, E. Tan, E. Christensen, C. Farberow, F. Baddour, K. Van Allsburg, J. Schaidle


- Coupled process models and TEA with fundamental and applied catalysis research
- Quantified the impact of catalyst development on process economics for NREL’s Cu/BEA catalyst in the production of HOG and jet fuel
• New synthetic method to prepare colloidally-stable $\alpha$-MoC$_{1-x}$ nanoparticles from a commercially-available, air-stable precursor at 300-320 °C
  • Previous approaches require reactive gases (CH$_4$, H$_2$) and thermal treatments >550 °C
• Translated the synthesis to a continuous millifluidic flow reactor, enabling 99% yield and production of up to 18.6 g of NP-MoC$_{1-x}$ product per 24 h, 450 g of 4 wt% catalyst
• NP-MoC$_{1-x}$ catalyst exhibited a 2X increase in per-site activity and 2X increase in C$_2+$ hydrocarbon selectivity versus bulk $\alpha$-MoC$_{1-x}$ in CO$_2$ reduction reaction
Spectroscopic insight into carbon speciation and removal on a Cu/BEA catalyst during renewable high-octane hydrocarbon synthesis

Q. Wu, A.T. To, C.P. Nash, D.P. Dupuis, F.G. Baddour, S.E. Habas, D.A. Ruddy


- Surface carbon species were identified and compared for BEA and Cu/BEA catalysts
- Cu/BEA had lower polycyclic aromatic content and more defective graphitic carbon
- Presence of Cu promoted carbon removal at lower temperature by activating O₂
- In situ spectroscopy informed a regeneration procedure for Cu/BEA
- Activity of Cu/BEA for DME-to-HOG was fully recovered following developed procedure