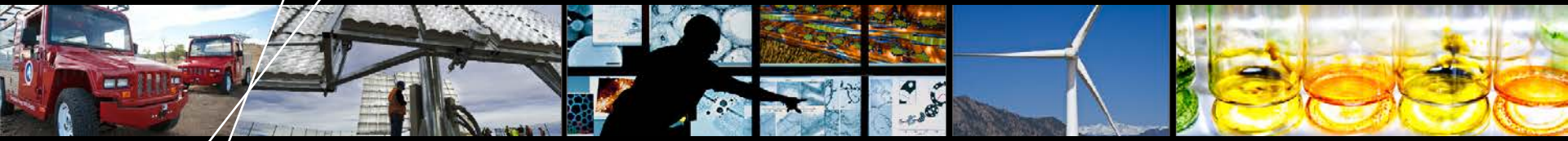
DOE Bioenergy Technologies Office (BETO) 2015 Project Peer Review

Catalytic Upgrading of Pyrolysis Products



March 24th, 2015
Thermochemical Conversion

Josh Schaidle
NREL

This presentation does not contain any proprietary, confidential, or otherwise restricted information

Goal Statement

The **goal** of this project is to ***design and develop scalable and cost-effective next generation catalysts*** for ex-situ catalytic fast pyrolysis (CFP) to improve the fuel quality and stability of the resulting bio-oil by ***reducing oxygen content, increasing hydrogen content, and increasing carbon number*** to a range suitable for gasoline, diesel, or jet fuel. These catalysts must achieve the following intermediate technical targets for upgraded oils at the large-bench scale (~200g catalyst) in 2017:

- H/C molar ratio of 1.3
- Carbon efficiency of 34%
- Oxygen content of 12.5wt%

Near-term Impact:

- Ex-situ CFP catalysts that outperform SOT materials
- In-house data collection and validation for ex-situ CFP pathway

Long-term Impact:

Catalysts developed within this project, when integrated with upstream and downstream processing, can be ***scaled to commercial size and yield a drop-in hydrocarbon fuel at a minimum selling price of $3.30/GGE by 2022.***

Quad Chart Overview

Timeline

- **Project start date:** 10/1/2012
- **Project end date:** 9/30/2017
- **Percent complete:** 50%

Barriers addressed

- Tt-E. Liquefaction of Biomass and Bio-Oil Stabilization
- Tt-G. Fuel Synthesis and Upgrading
- Conversion enabling technologies through *development of next generation catalysts*

Budget

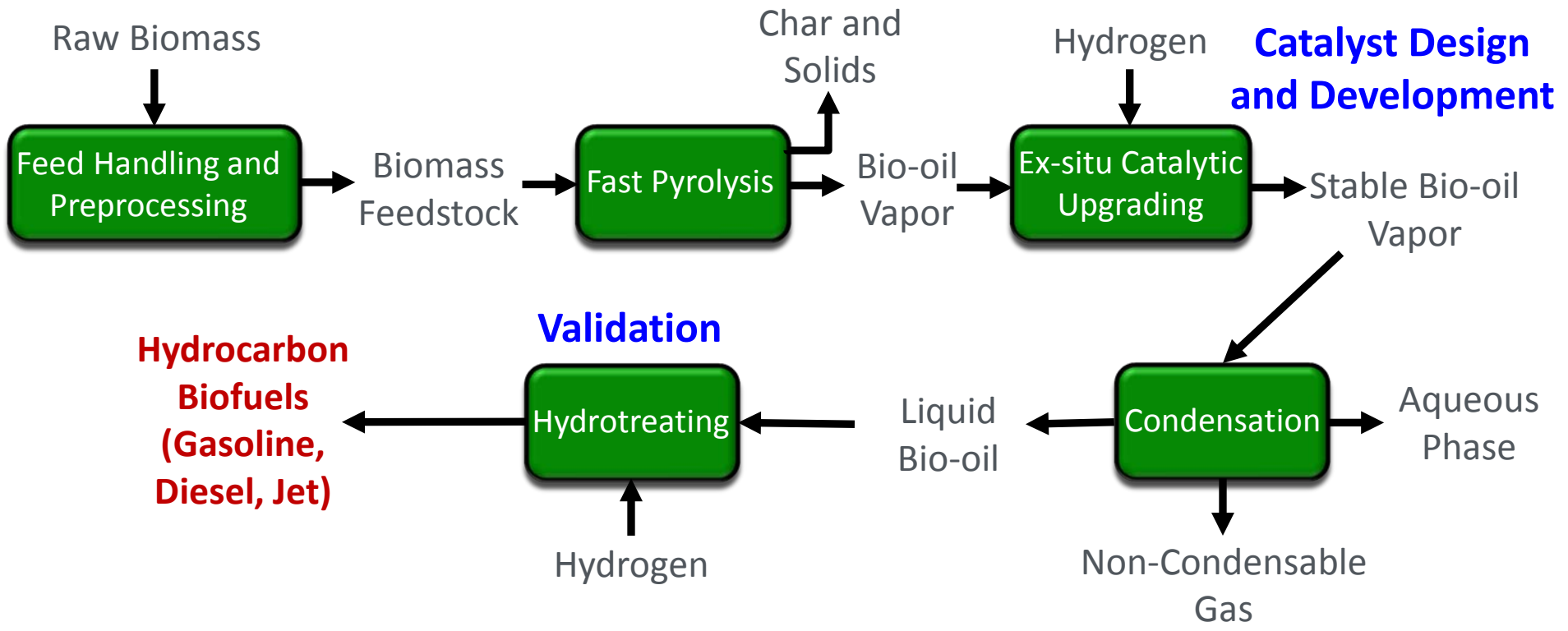
	Total Costs FY10 –FY12	FY13 Costs	FY14 Costs	Total Planned Funding (FY15-Project End Date)
DOE Funded	$0	$1.52M	$2.0M	$7M

Partners

Key Collaborators

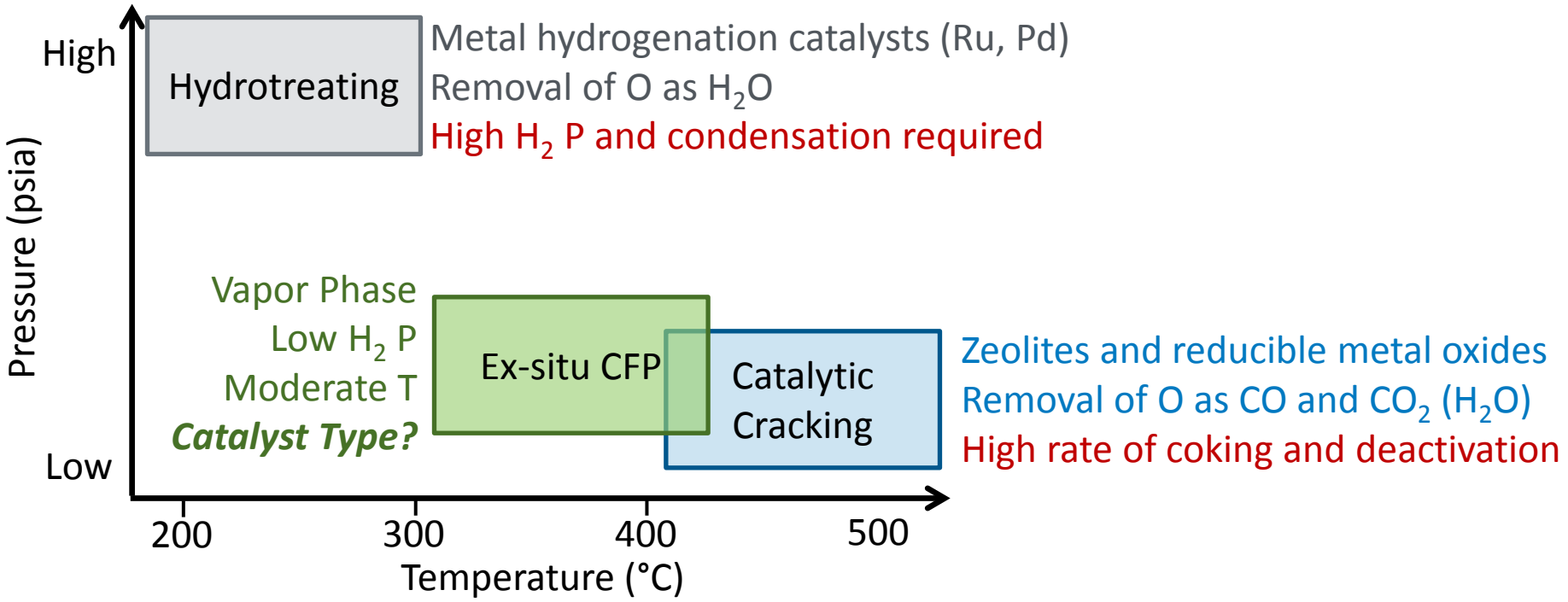
- **Internal**
  - Computational Pyrolysis Consortium (2.5.1.302)
  - Catalytic Pyrolysis Science (2.3.1.313)
- **External**
  - Dr. Levi Thompson, University of Michigan (SUB)
  - Dr. Will Medlin, University of Colorado-Boulder (SUB)
  - Dr. Richard Brutchey and Dr. Noah Malmstadt, University of Southern California

Project Overview: FY13-FY15

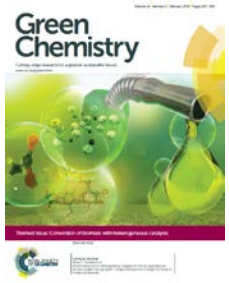


Design and development of *ex-situ* CFP catalysts using a "Bottom Up" approach combined with validation of improvement in downstream processing

Project Overview: Ex-situ CFP Conditions



In FY13, we evaluated the SOT and identified promising catalysts for ex-situ CFP through a critical lit review:



D. Ruddy, J. Schaidle, J. Ferrell, J. Wang, L. Moens, J. Hensley, *Green Chemistry* 2014, 16, 454-490.

- Bifunctionality is *essential*
  - Hydrogenation (metallic) and dehydration (acid or oxygen-vacancies)
  - Bifunctional active phase or active phase/support combination
- Metals and metal phosphides/carbides

Project Overview: Platform Integration

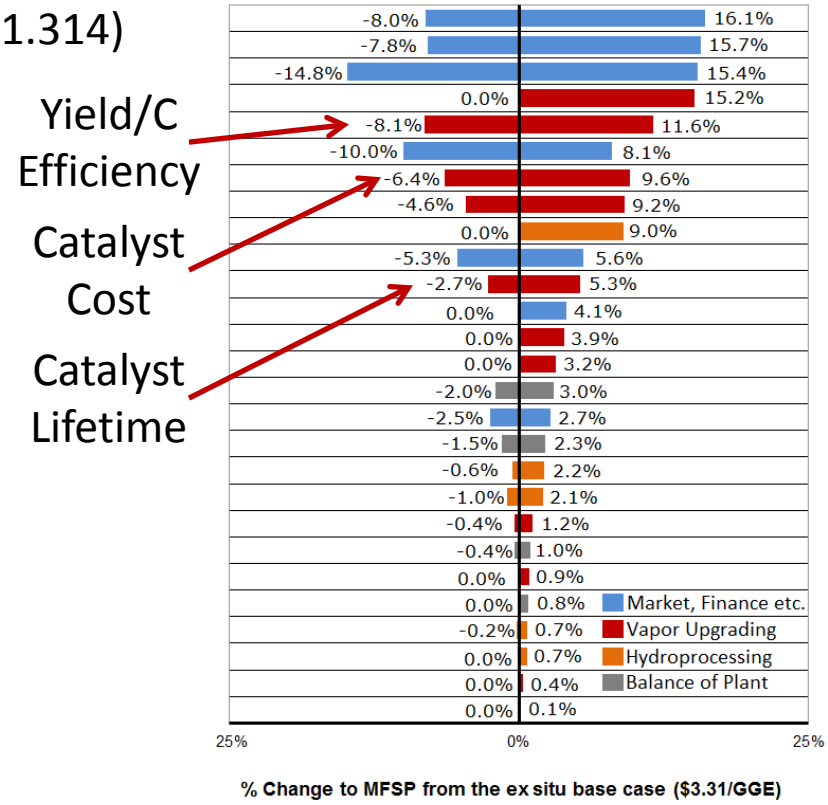
WBS: 2.3.1.313	2.3.1.314	2.3.1.315	2.4.1.301	2.1.0.302
Catalytic Pyrolysis Science Pyrolysis science fundamentals, catalyst modeling Lab scale	Catalytic Upgrading of Pyrolysis Products *Ex-situ, in-situ* Catalyst development/evaluation Lab scale	Biomass Deconstruction: Catalyst Development/Testing *Ex-situ* catalyst evaluation/ characterization Lab-small pilot	Engineering Integration and Scale-up *Ex-, in-situ* TCPDU pilot scale demonstration Pilot scale	Thermochemical Platform Analysis: TEA *Ex-, in-situ*, product/process costs at Pilot scale

mg-g → g-kg → 100's kg

Near-term emphasis: Zeolites and modified zeolites (2.3.1.313 and 2.3.1.315)

Long-term emphasis: Next-generation catalysts (2.3.1.314)

Process Parameter	2017 Target	2022 Target / Design Case
Vapor Products		
Non-Condensable Gases	30	23
Aqueous Phase (% C Loss)	26 (2.3)	30 (1.3)
Solids (Char + Coke)	12 + 10.2	12 + 8.0
Organic Phase	22.0	27.2
H/C Molar Ratio	1.3	1.6
Carbon Efficiency (%)	34	44
Oxygen Content (% of organic)	12.5	6.4
Diesel-Range Product (% GGE basis)	14	55
Minimum Fuel Selling Price ($ / GGE)	$4.58	$3.31



Project Overview: Objectives

The primary objectives of this project are:

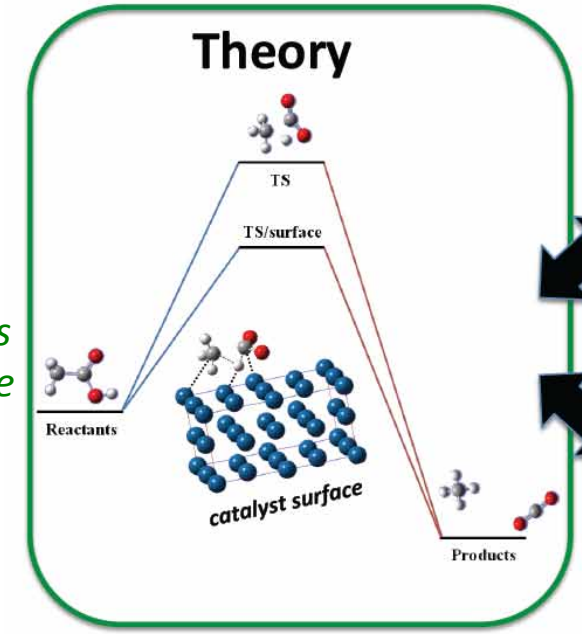
- Design and develop scalable and cost-effective next generation ex-situ CFP catalysts which outperform commercial and SOT materials for ***hydrogen incorporation, deoxygenation, and C-C coupling***
- Provide ***in-house data*** for technoeconomic analysis to ***guide the selection of the optimal process design*** for producing liquids fuels at ~$3/GGE via biomass pyrolysis
- Validate that ex-situ CFP ***reduces the severity, cost, and/or unit operations*** for down-stream hydroprocessing
- Demonstrate catalyst performance at the large-bench scale (~200g cat) with ***real pyrolysis vapors*** in 2017

Technical Approach

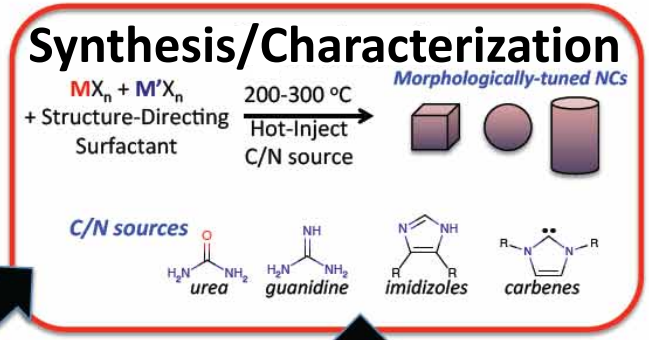
Combine ***experimental*** and ***computational*** efforts to drive catalyst discovery and development through theoretical modeling, advanced synthetic techniques, rigorous catalyst characterization, and reaction testing with model compounds and real pyrolysis vapors

Computational Pyrolysis Consortium (2.5.1.302)

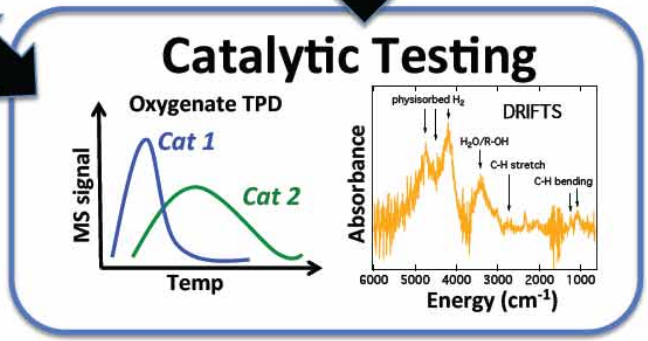
- *Mechanisms*
- *Performance Descriptors*
- *Predictive Capability*



Precise control of size, shape, composition, and morphology of active phase



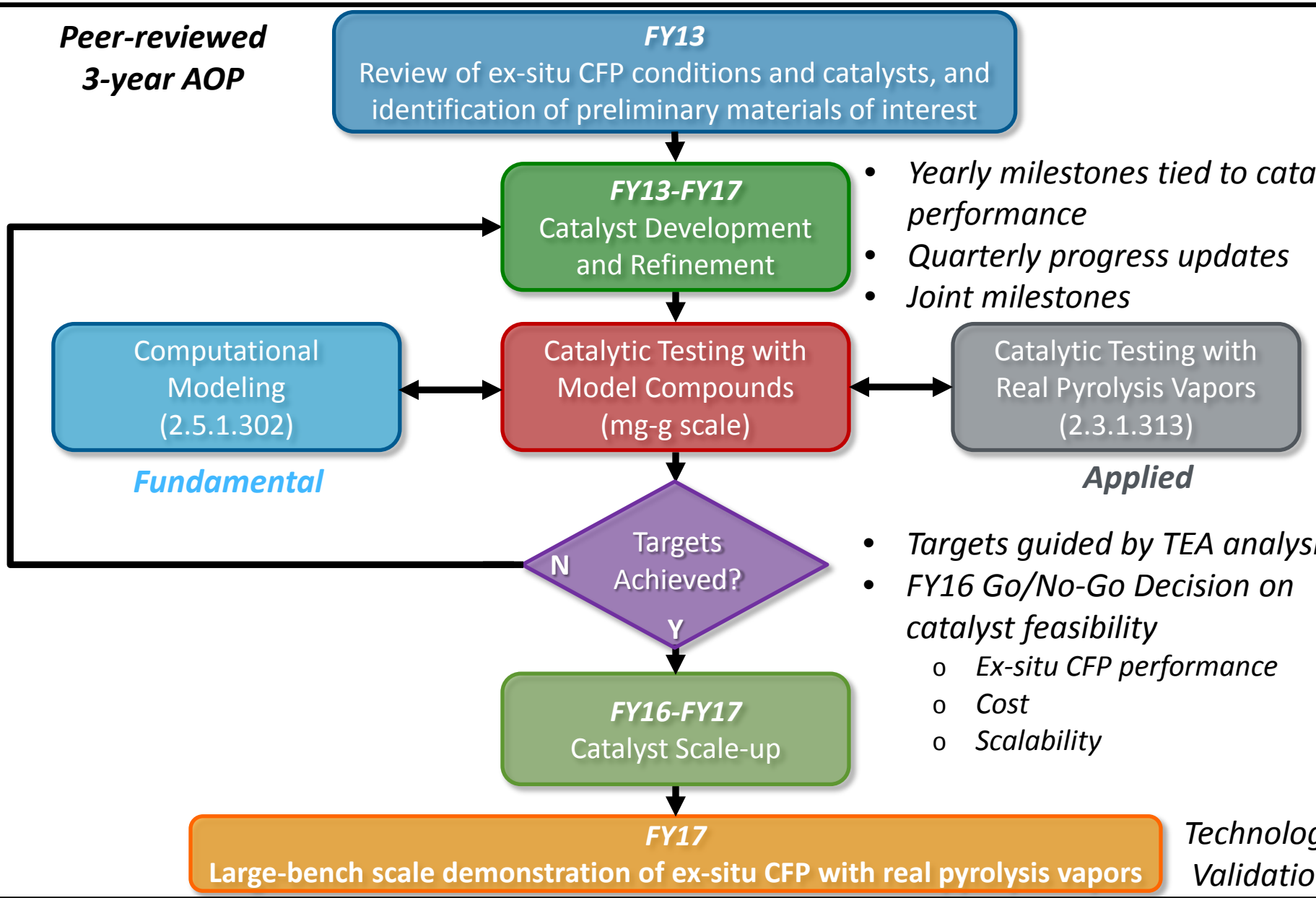
- *Colloidal solution-phase synthesis*
- *Support independent*
- *Metrics:*
  - Cost
  - Scalability



- *Catalyst down-selection*
- *Metrics:*
  - Extent of deoxygenation
  - Extent of H_2 incorporation
  - C efficiency
  - Stability

Testing with model compounds and real pyrolysis vapors

Management Approach



Challenges and Success Factors

Grand Challenges

- ***Activation and incorporation of H_2*** at moderate T (300-500°C) and low H_2 P to achieve ***selective hydrogenation*** of specific functional groups
- ***Removal of O via H_2O*** instead of CO/CO_2 (C-O vs. C-C bond cleavage)
- ***Increase product carbon number*** (C_{8+}) via C-C coupling while minimizing deactivation due to carbon deposition
- Achieve this functionality with ***cost-effective, scalable materials***

Critical Success Factors

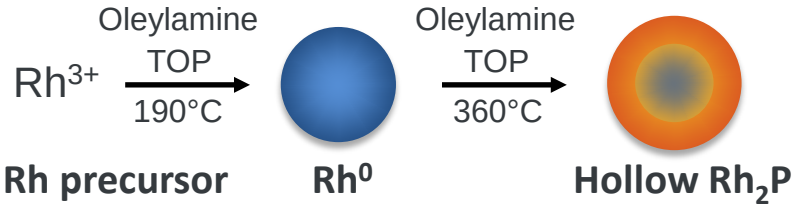
- Achieve 2017 targets for ex-situ CFP at large-bench scale: H/C molar ratio = 1.3, C efficiency = 34%, and O content = 12.5wt%
- Minimize catalyst cost by using low-cost metals and precursors and achieving high dispersion of the active phase
  - Target: scaled catalyst cost < $50/lb
- Demonstrate that ex-situ CFP reduces cost of downstream hydrotreating

Go/No-Go Decision Point - March 2016

- Commercial feasibility assessment of colloidal nanoparticle catalysts
- Metrics: Cost, scalability, and ex-situ CFP performance

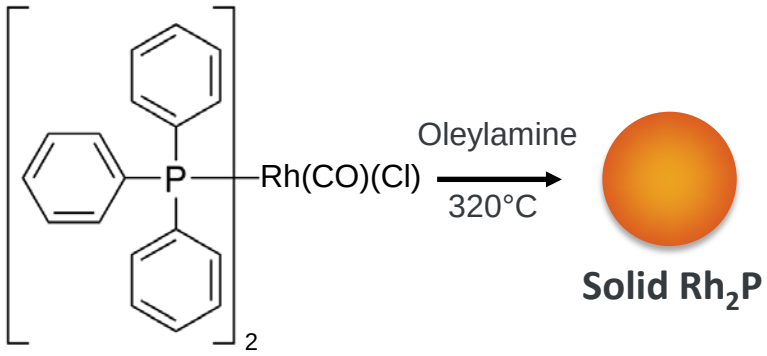
Research Progress: NP Synthesis-Phosphides

Standard phosphidation method

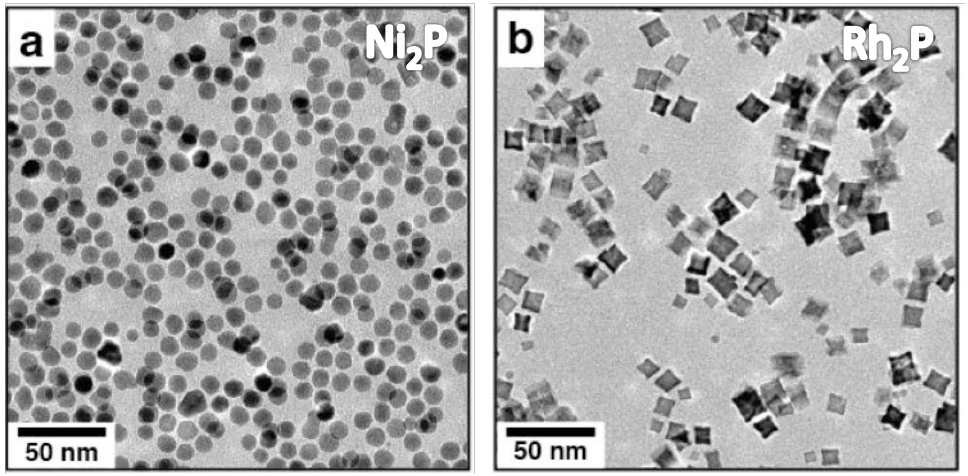


- **Catalytically relevant phosphides**
  - Ni-P, Rh-P, Pd-P, Fe-P, Mo-P
- **Phase and composition control**
  - Ni_2P, $Ni_{12}P_5$, Fe_2P, FeP, amorphous MoP
- **Shape control**
  - Cubic Rh_2P, FeP nanorods

Single-source precursor route



J. Wang, D. Ruddy, S. Habas, F. Baddour, NREL PROV/13-62, 2015.

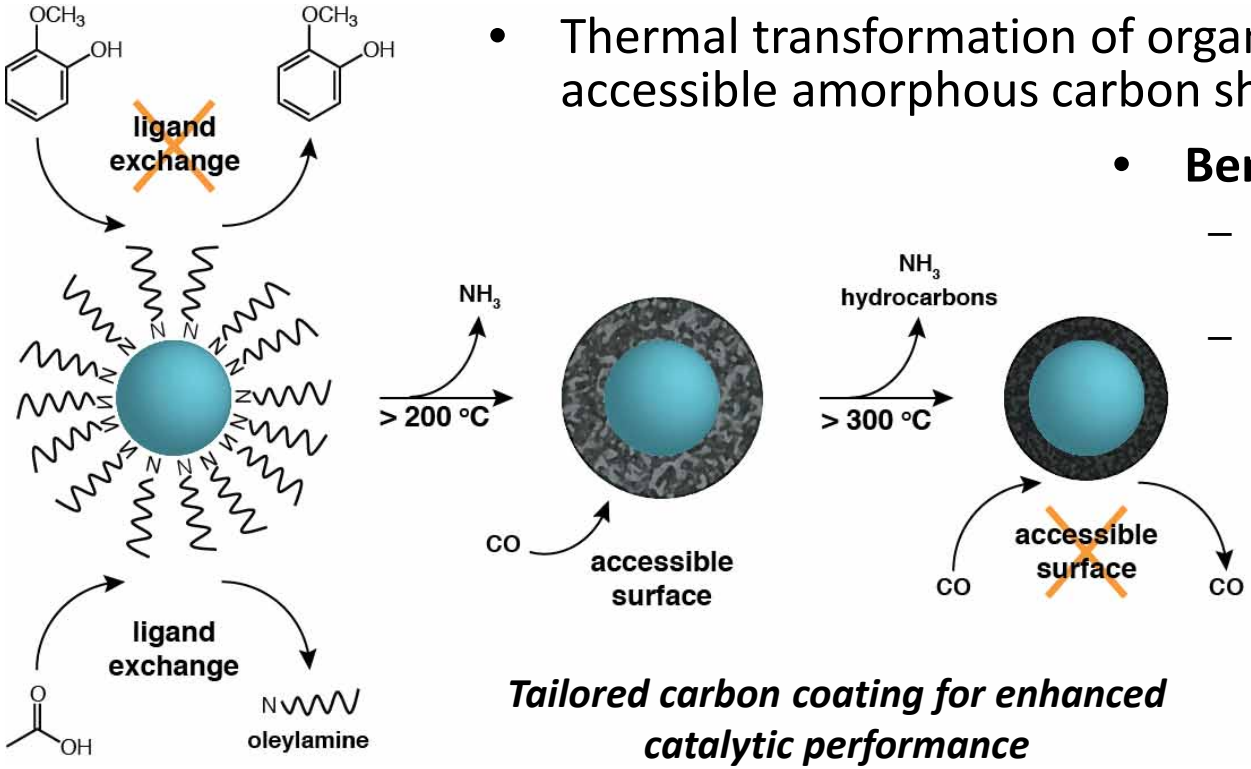


Versatile route to well-defined metal phosphide nanoparticles

Support-independent synthesis with precise control over the size, shape, and composition of the active phase accelerates catalyst development and facilitates direct linkages to theory

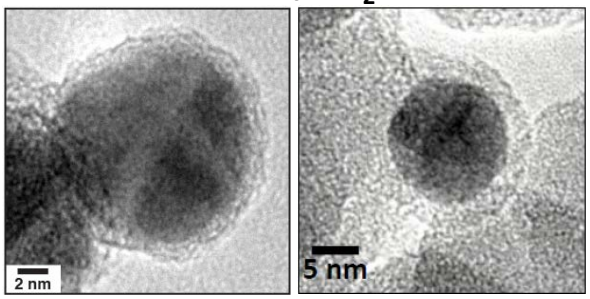
Research Progress: NP Surface Chemistry-Ligands

- **Organic ligands (e.g., alkylamines) stabilize NPs and control features (size, shape, composition)**
- **Investigated the effect of ligands on NP surface chemistry and reactivity**
  - As-prepared NPs are resistant to ligand exchange with phenolic but not acidic compounds
  - Thermal transformation of organic ligands leads to an accessible amorphous carbon shell
  - **Beneficial catalytic effects**
    - Active site geometric/electronic structure
    - Active phase-support interface



Tailored carbon coating for enhanced catalytic performance

NP-Ni/SiO_2

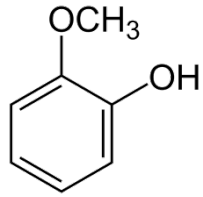


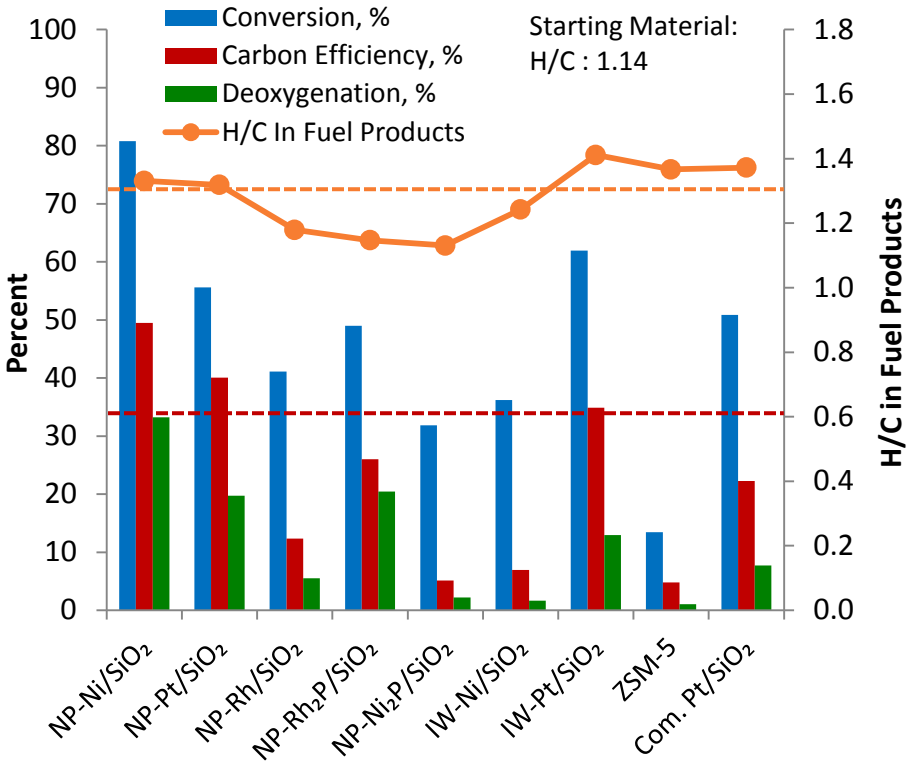
350 °C in H_2 | **Spent, 8h, Guaiacol + H_2, 350°C**

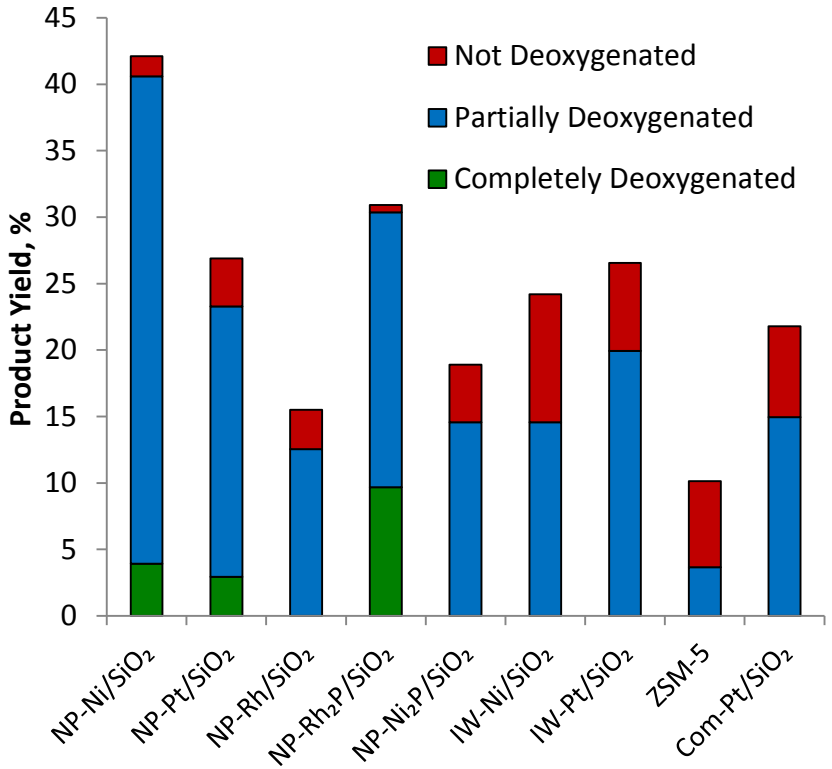
Understanding ligand effect on reactivity will help us design improved catalysts

Research Progress: Model Compound Upgrading

Catalytic upgrading of guaiacol was used to evaluate and down-select active phase materials





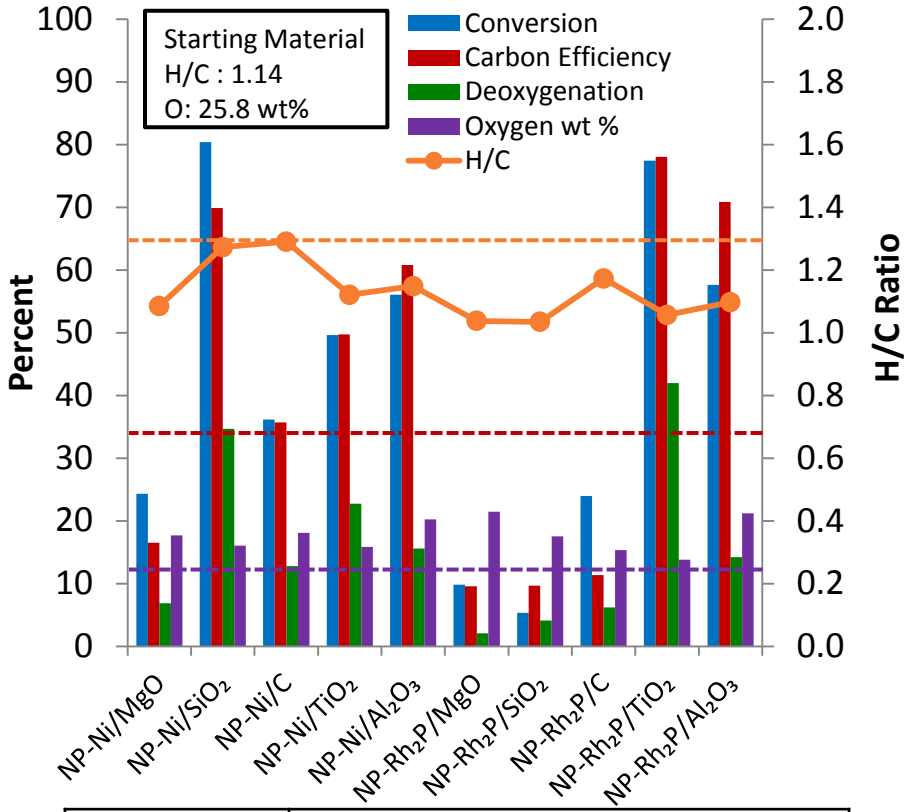


- Colloidal NP catalysts outperformed incipient wetness materials
- Catalysts down-selected for further study: NP-Ni/SiO_2, NP-Pt/SiO_2, and NP-Rh_2P/SiO_2

350°C, 0.5MPa, WHSV 5 h^{-1}, 12:1 H_2:guaiacol

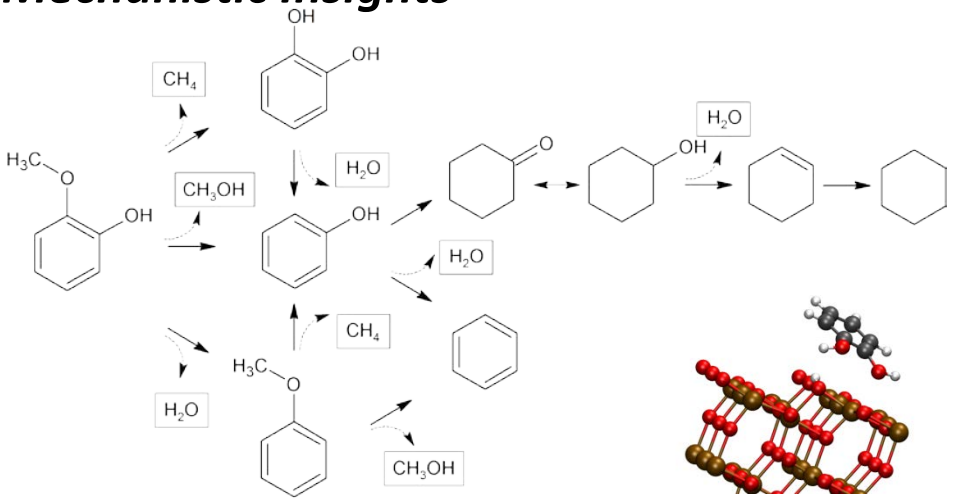
Research Progress: Model Compound Upgrading

Catalytic upgrading of guaiacol over NP-Ni and NP-Rh_2P dispersed on selected supports was used to evaluate and down-select active phase-support materials



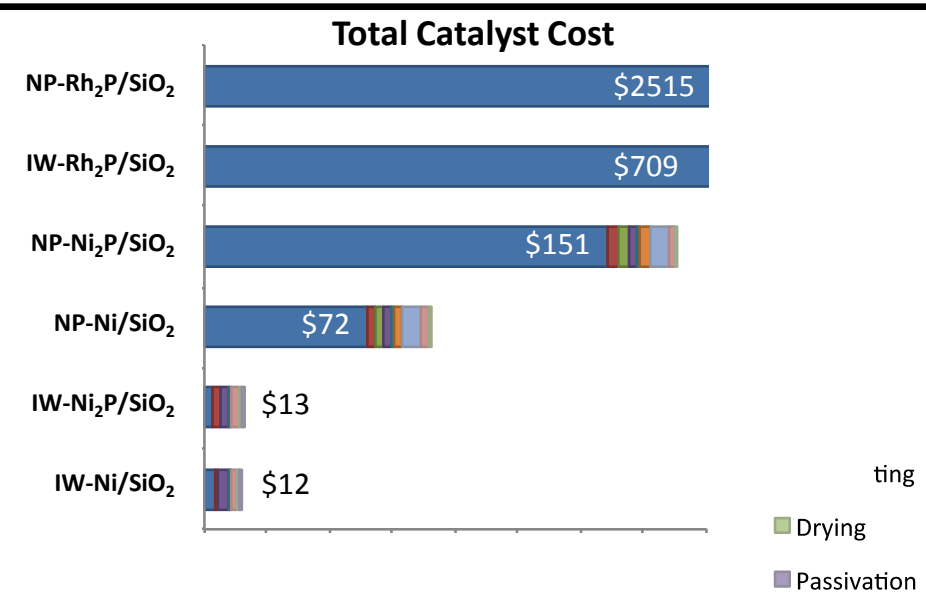
Catalyst	Deactivation Rate Constant (min^{-1})
NP-Ni/Al_2O_3	0.0007
NP-Rh_2P/TiO_2	0.0007
NP-Rh_2P/Al_2O_3	0.0008
NP-Ni/SiO_2	0.0011
NP-Ni/TiO_2	0.0014

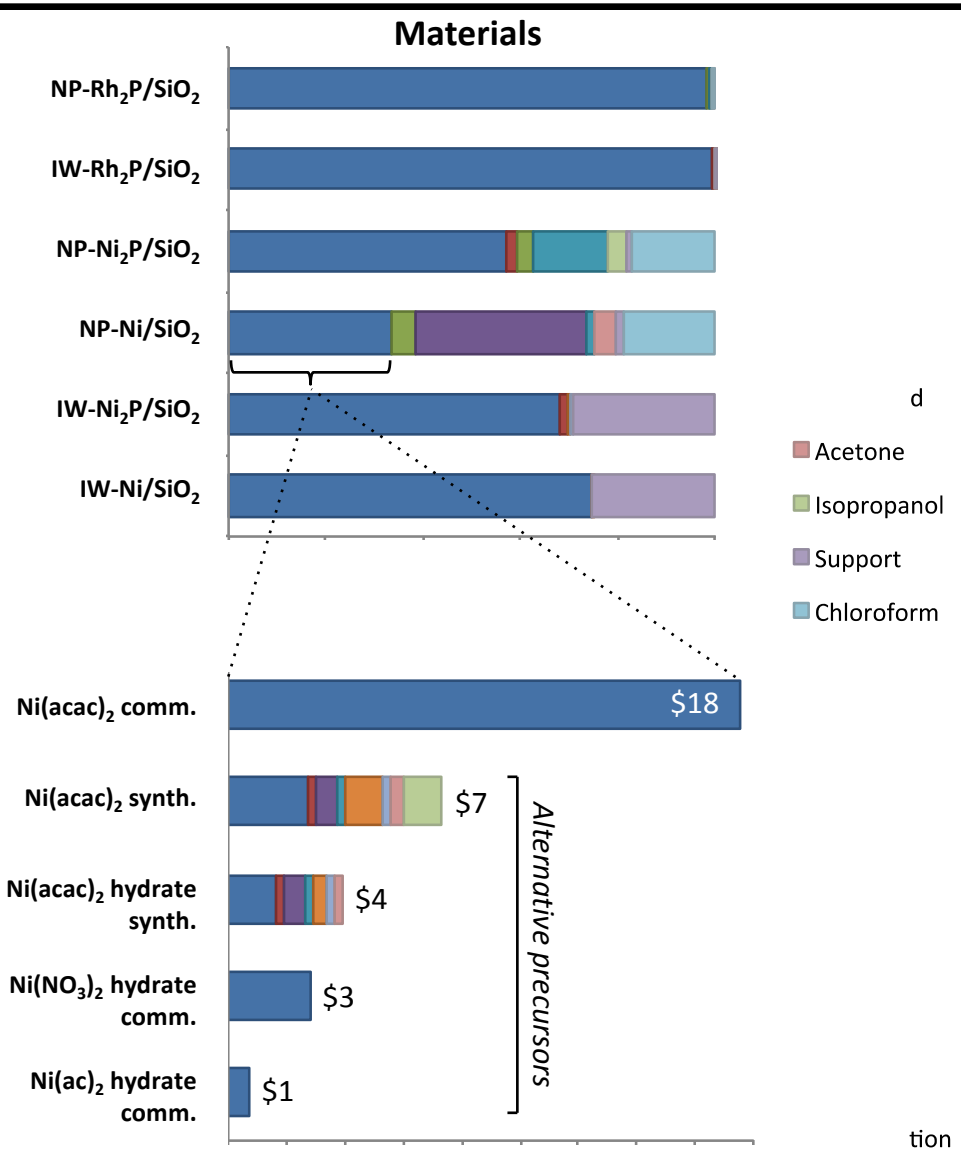
Mechanistic Insights



- Best performing materials: NP-Rh_2P/TiO_2, NP-Ni/SiO_2, NP-Ni/TiO_2
- Combining upgrading results with computational efforts to determine dominant reaction mechanism under ex-situ CFP conditions:
  - Ring hydrogenation
  - Tautomerization
  - Direct Deoxygenation

Research Progress: Preliminary Catalyst Cost

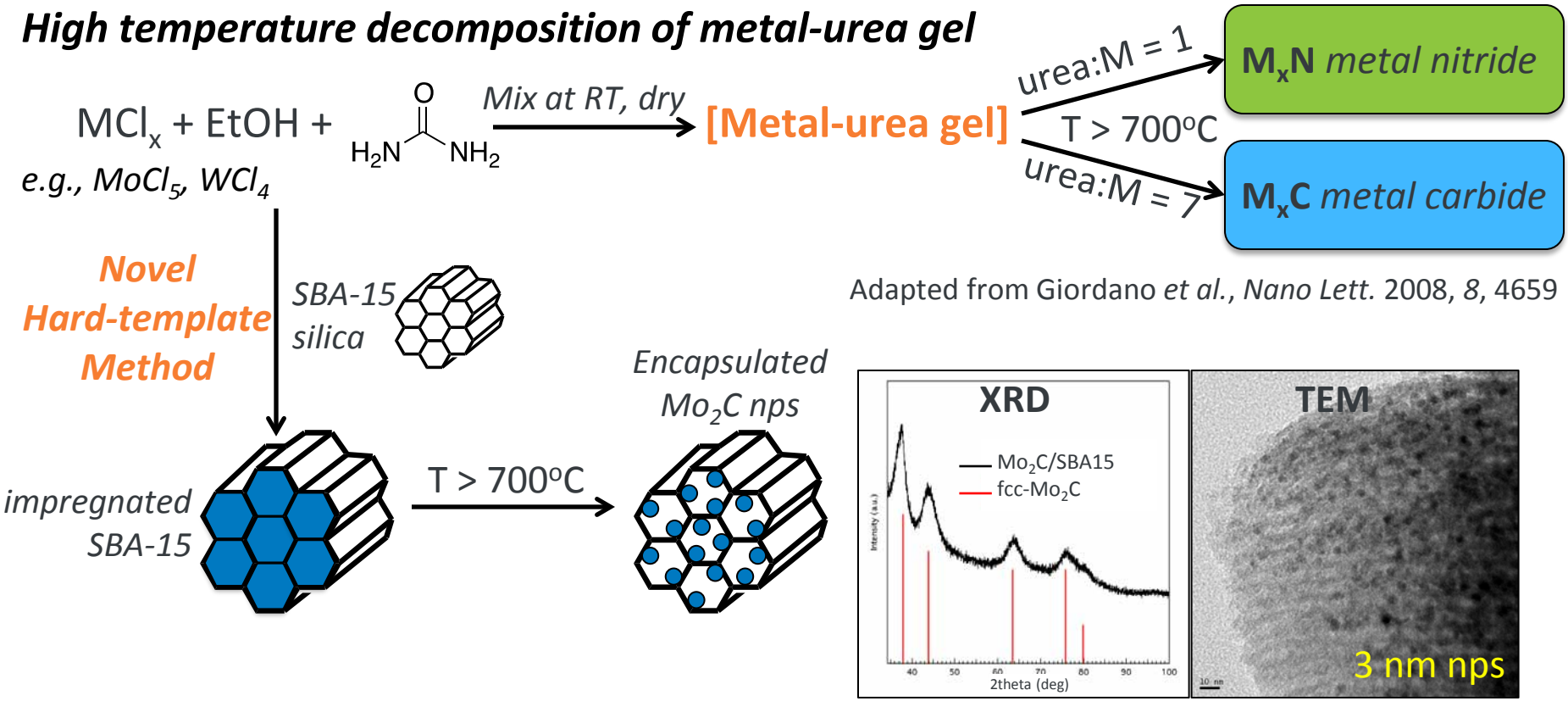




- Processing costs for metals and metal phosphides are similar
- Materials for NP catalysts are the greatest contributor to cost
- Opportunities to reduce cost exist through:
  - Alternative metal precursors
  - Elimination of TOP

Research Progress: NP Synthesis-Carbides/Nitrides

High temperature decomposition of metal-urea gel

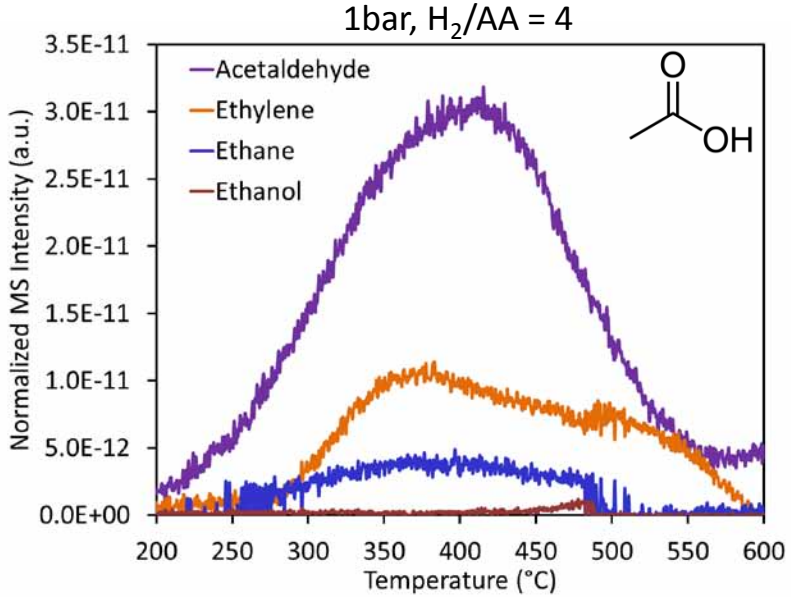


Adapted from Giordano *et al.*, *Nano Lett.* 2008, *8*, 4659

- Successfully synthesized nanoparticles of metal carbides (Mo_2C, W_2C, VC) and metal nitrides (Mo_2N, W_2N, VN, Mo_xV_yN)
- Developed a hard-templating method

Research Progress: Mo_2C-Acetic Acid Upgrading

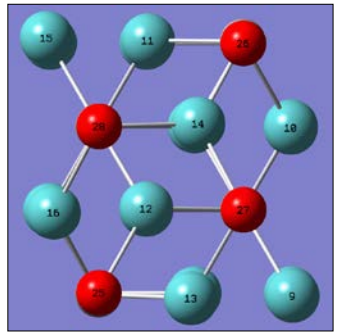
Experimental Results



- At T<400°C, Mo_2C favors hydrogenation-dehydration products (C-O bond cleavage)
- Mo_2C surface and sub-surface contains oxygen even after pretreatment in H_2
- Good agreement between experiment and theory

Modeling Results

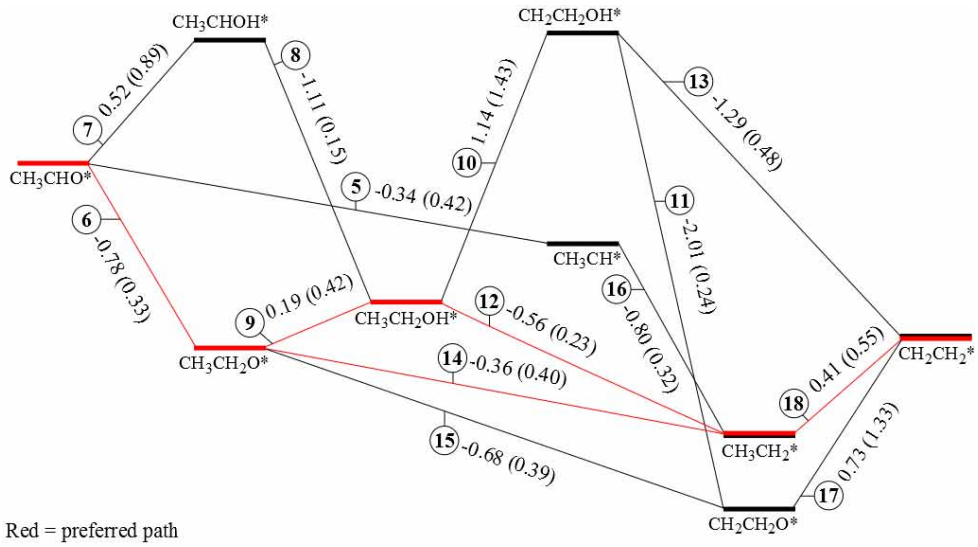
(O)-Mo-β-Mo_2C



XPS Elemental Estimates

	Exp.	Theor.
XPS depth, Å	50	
O/Mo	0.62	0.52

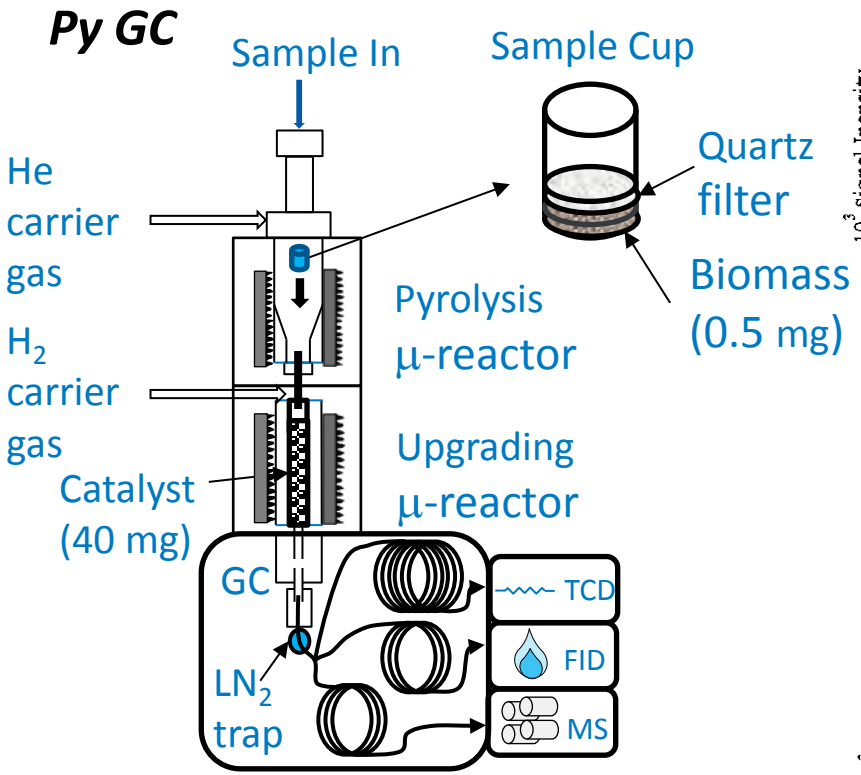
Reaction pathways and energetics

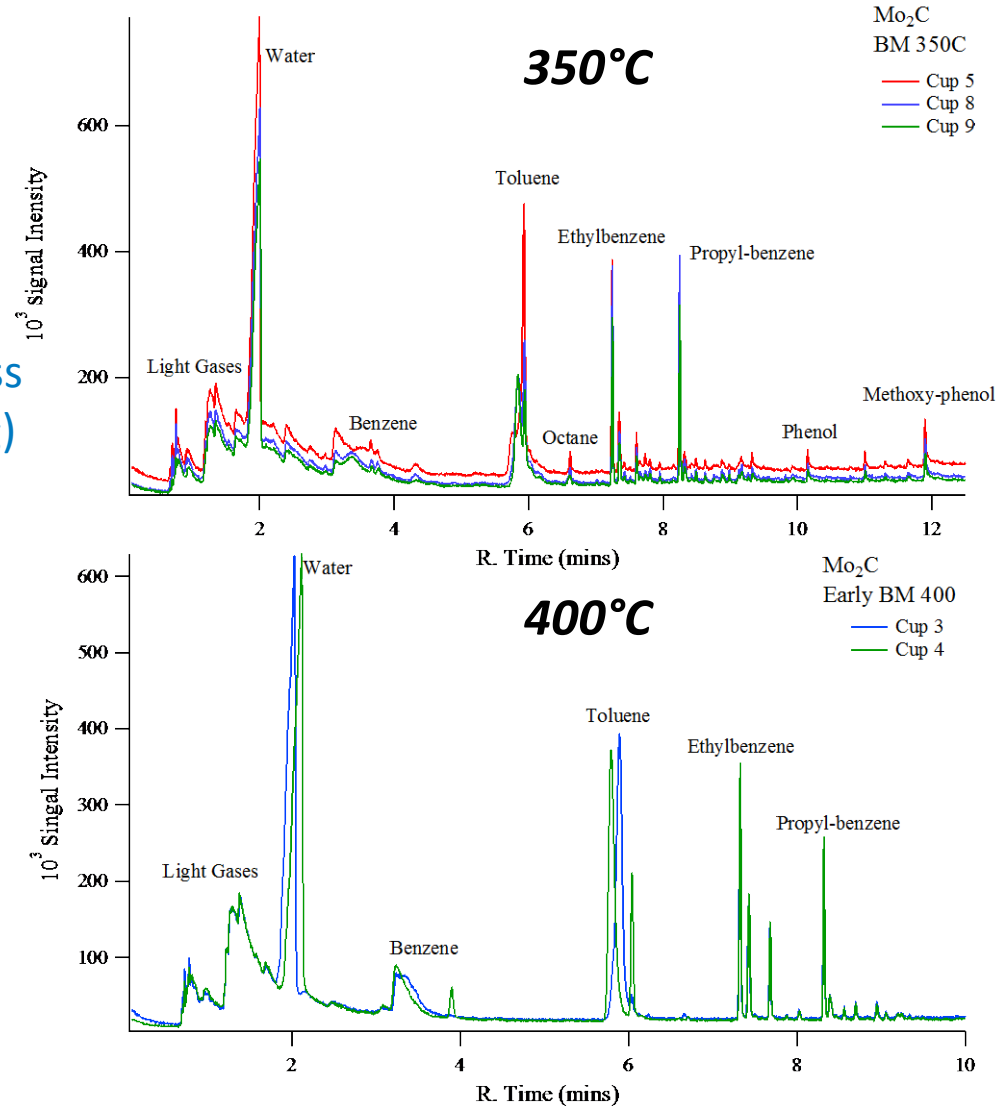


Joint with Computational Pyrolysis Consortium (2.5.1.302)

Research Progress: Mo_2C-Biomass Upgrading

Tested the ex-situ CFP performance of Mo_2C with real biomass pyrolysis vapors using a py-GC system



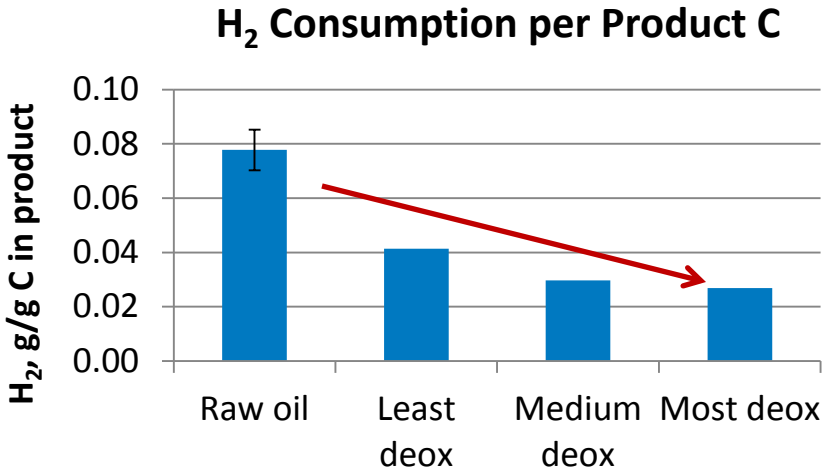


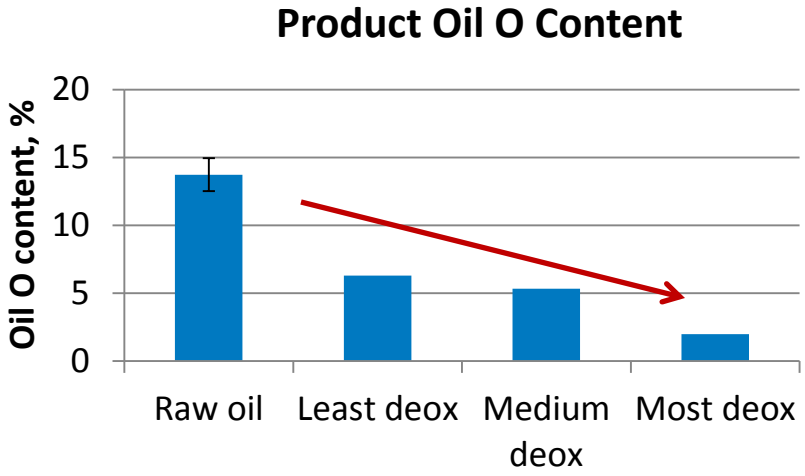
Preliminary results indicate that Mo_2C is a promising material for ex-situ CFP

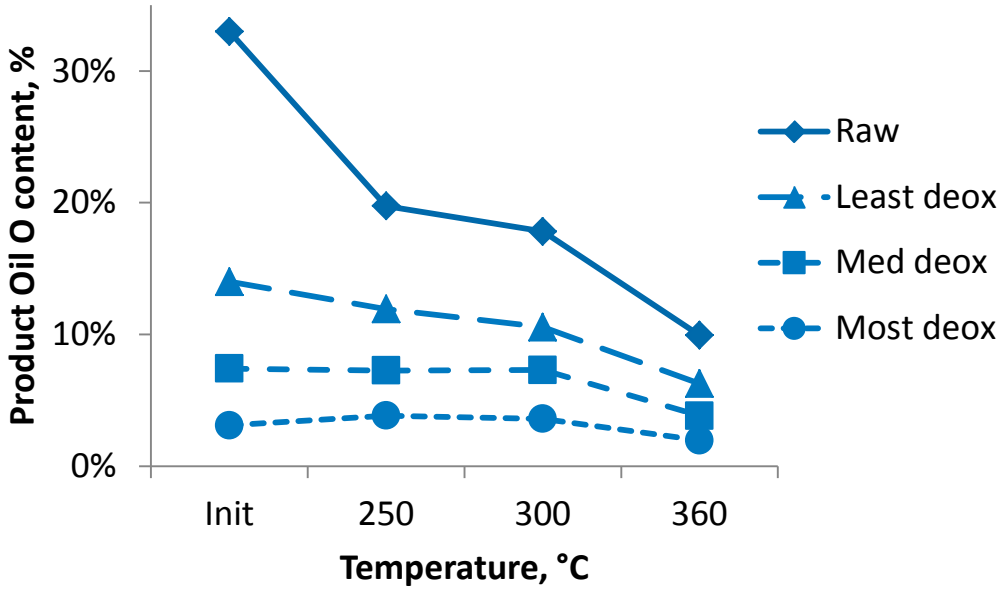
Joint with Catalytic Pyrolysis Science (2.3.1.313)

Research Progress: Hydrotreating Validation

Compared the hydrotreatability of surrogate mixtures representing catalytically upgraded and raw pyrolysis oils over Pd/C at 250-360°C





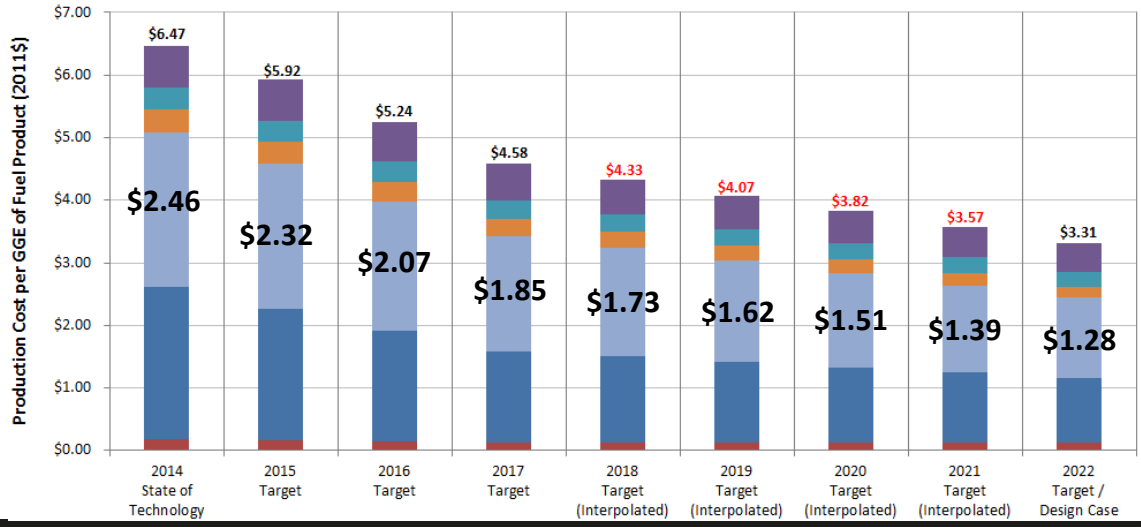


- Catalytically upgraded oils result in lower oxygen content in the final product and reduced H_2 consumption
- Oxygenates remaining in catalytic pyrolysis oil are recalcitrant and require temperatures in excess of 350°C for deoxygenation

Relevance

Decreasing Biomass Conversion Costs through Catalyst Development

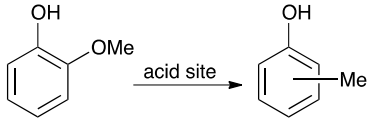
- Directly supports BETO's mission:
  "Develop and transform our renewable biomass resources into commercially viable, high performance biofuels"
- Addresses BETO's 2017 target for Bio-Oils Pathways R&D of a conversion cost of $1.83 per gallon of total blendstock
  - Project fulfills a critical need for Conversion Enabling Technologies:
    "The ***need to develop the next generation of catalysts*** for conversion of biomass and conditioning of bio-oils is ***critical in the advancement of biomass processing technologies.***"
- Project metrics and technical targets are driven by TEA
- Reduction in conversion costs through improvements in:
  - H_2 incorporation
  - Deoxygenation
  - C efficiency/yield
  - C-C coupling



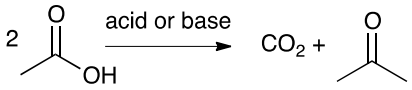
Future Work

- Continue ***catalyst development***
  - ***Increase extent of hydrogenation and deoxygenation by 15%*** in 2016 compared to FY15 guaiacol upgrading results
  - Evaluate catalyst performance with ***model compound mixtures of increasing complexity***
  - Leverage mechanistic insights from computational modeling to design next-generation catalysts
    - Modification of Mo_2C to enhance H_2 incorporation
    - Tune active phase-support interface to drive chemistry towards specific reaction pathways
- ***Demonstrate C-C coupling*** with model compounds

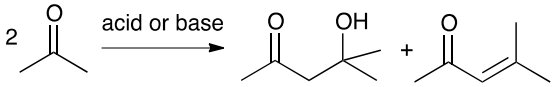
Methyl/Alkyl Transfer



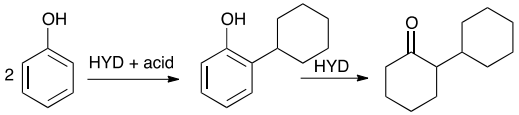
Ketonization



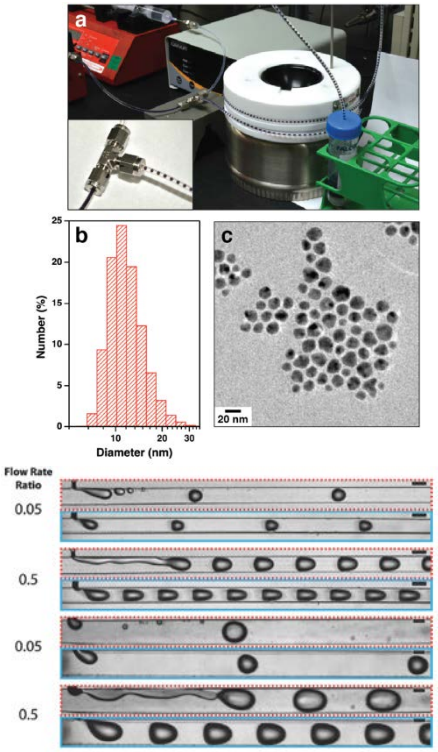
Aldol Condensation



Hydroalkylation



- ***Validate*** reduction in H_2 consumption and oxygen content during hydrotreating with real upgraded oils
- ***Demonstrate scale-up*** of colloidal NP catalysts from 5g to 200g per batch using micro-fluidic flow reactors



Riche, Zhang, Gupta, and Malmstadt. *Lab on a Chip* 14:1834-1841. 2014.

Summary

- Ex-situ CFP conditions (vapor phase, low H_2 P, moderate T) lie outside of those typically explored for hydrotreating and catalytic cracking
  - ***Multi-functional catalytic materials*** need to be developed specifically for these conditions
- This project focuses on ***developing next generation catalysts for ex-situ CFP through a combined experimental-computational approach*** which leverages theoretical modeling, advanced synthetic techniques, rigorous catalyst characterization, and reaction testing with model compounds and real pyrolysis vapors
  - Catalysts are designed and evaluated based on ***metrics derived from TEA***
  - ***Collaborative*** research across multiple projects
  - ***Commercial viability*** is assessed through catalyst cost calculations and scale-up of synthesis methods
- Results suggest that ***NP-Ni and Mo_2C are promising catalysts*** for ex-situ CFP
- ***Further catalyst advancements*** will be driven by mechanistic insights and will reduce conversion costs to ***achieve our 2017 targets at the large-bench scale***

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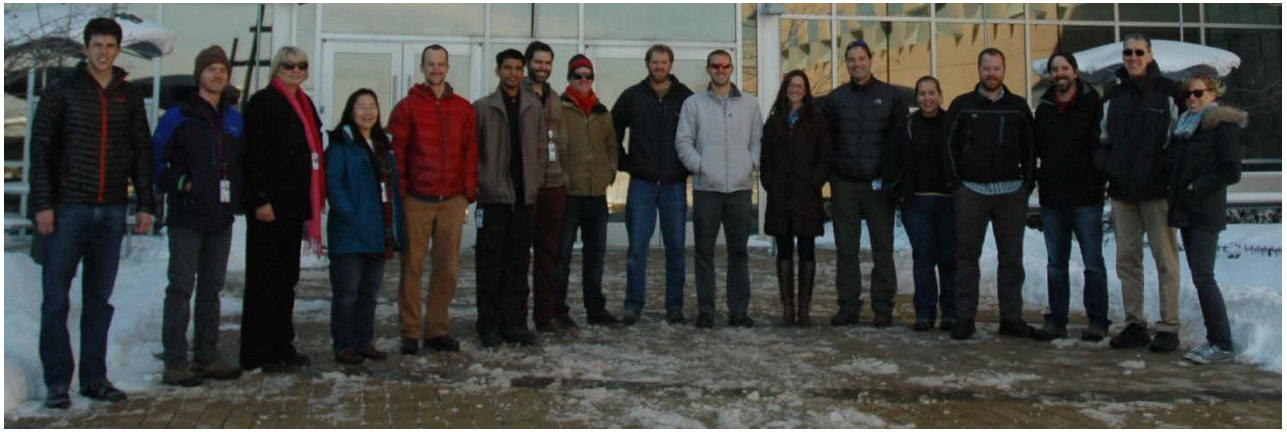
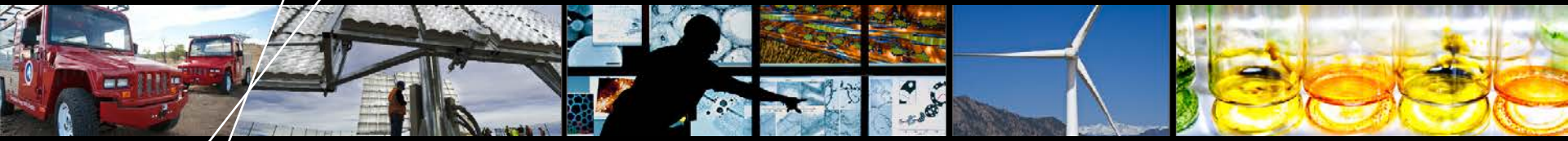


DOE Bioenergy Technologies Office (BETO) 2015 Project Peer Review

Catalytic Upgrading of Pyrolysis Products



March 24th, 2015
Thermochemical Conversion

Josh Schaidle
NREL

This presentation does not contain any proprietary, confidential, or otherwise restricted information

Nomenclature

AA: Acetic Acid
CFP: Catalytic Fast Pyrolysis
CU: University of Colorado
GGE: Gasoline Gallon Equivalent
IW: Incipient Wetness
MFSP: Minimum Fuel Selling Price
NP: Nanoparticle
SOT: State of Technology
SUB: Subcontract
TCPDU: Thermochemical Process Development Unit
TEA: Technoeconomic Analysis
TOP: Trioctylphosphine
UM: University of Michigan
USC: University of Southern California

Presentations

Presentations

- J. Schaidle, S. Habas, D. Ruddy, M. Yung, J. Hensley, Upgrading of Model Pyrolysis Compounds using ZSM-5 and Hydrogen Donors, Western States Catalysis Club Annual Symposium, Provo, UT, April 19, 2013.
- R. French, Refinery feedstock from partially hydrogenated biomass pyrolysis oil, 2013 AIChE Spring Meeting, San Antonio, TX, April 28-May 3, 2013.
- R. French, Hydrotreating Pyrolytic Lignin to Produce a Refinery Feedstock, TCBiomass 2013, Chicago, IL, September 3-6, 2013.
- J. Schaidle, J. Clark, D. Ruddy, J. Wang, J. Blackburn, D. Robichaud, J. Hensley, Development of Metal Carbide, Nitride, and Phosphide Catalysts for Bio-oil Deoxygenation: Acetic Acid Hydrogenation, AIChE Annual Meeting, San Francisco, CA, November 4, 2013.
- S. Habas, J. Wang, D. Ruddy, J. Schaidle, M. Griffin, J. Hensley, Exploring Structural and Compositional Changes of Metal Phosphide Nanoparticle Catalysts under Pyrolysis Oil Upgrading Conditions, AIChE Annual Meeting, San Francisco, CA, November 6, 2013.
- D. Ruddy, J. Wang, J. Schaidle, J. Blackburn, J. Hensley, Synthesis and Characterization of Metal Carbide and Nitride Nanoparticle Catalysts for Bio-oil Deoxygenation, AIChE Annual Meeting, San Francisco, CA, November 8, 2013.
- M. Griffin, G. Ferguson, S. Habas, J. Hensley, D. Ruddy, J. Schaidle, An investigation into the vapor phase deoxygenation of a bio-oil model compound over monometallic, bimetallic, and metal phosphide catalysts. Gordon Conference for Catalysis. New London, NH, June 22-27, 2014.
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- G. Ferguson, M. Griffin, D. Ruddy, S. Habas, J. Schaidle, M. Biddy, G. Beckham, Catalytic Upgrading of Biomass: Aromatic Hydrodeoxygenation over Metal Phosphide Surfaces, ACS Annual Meeting, San Francisco, CA, August 12, 2014.
- G. Ferguson, M. Griffin, S. Habas, J. Schaidle, D. Ruddy, M. Biddy, G. Beckham, Understanding the Mechanism of Guaiacol Deoxygenation using DFT Calculations, ACS National Meeting, San Francisco, CA, August 13, 2014.
- L. Moens, R. French, K. Iisa, Hydrotreating of biomass pyrolysis oils in the presence of solvents, TCS 2014, Denver, CO, September 2-4, 2014.
- F. Baddour, S. Habas, D. Ruddy, M. Pan, C. Nash, J. Wang, J. Hensley, J. Schaidle, Single-source molecular precursor route to metal phosphide nanoparticles and their evaluation as deoxygenation catalysts, ACS National Meeting, Denver, CO, March 22-26, 2015.
- S. Habas, F. Baddour, D. Ruddy, C. Nash, M. Pan, J. Wang, J. Hensley, J. Schaidle, Nanostructured metal phosphide catalysts for conversion of biomass to liquid fuels, Invited talk, ACS National Meeting, Denver, CO, March 22-26, 2015.

Publications, Patents, and Awards

Publications

- J. Schaidle, A. Lausche, N. Schweitzer, L. T. Thompson, in *Comprehensive Inorganic Chemistry II (Second Edition)*, Editors-in-Chief: J. Reedijk and K. Poeppelmeier, Elsevier, Amsterdam, 2013, pp. 371-404.
- D. Ruddy, J. Schaidle, J. Ferrell, J. Wang, L. Moens, J. Hensley, Recent Advances in Heterogeneous Catalysts for Bio-oil Upgrading via "*Ex Situ* Catalytic Fast Pyrolysis": Catalyst Development through the Study of Model Compounds, *Green Chemistry* 2014, 16, 454-490.
- R. French, J. Stunkel, S. Black, M. Myers, M. Yung, and K. Iisa, Evaluate Impact of Catalyst Type on Oil Yield and Hydrogen Consumption from Mild Hydrotreating, *Energy Fuels* 2014, 28, 3086−3095.
- R. French, L. Moens, J. Stunkel, K. Iisa, Hydrotreating of Biomass Pyrolysis Oils in The Presence of Solvents, *Preprints, 248th ACS National Meeting,* San Francisco, CA, August 10-14, 2014. *Prepr. Pap.-Am. Chem. Soc., Div. Energy Fuels*. *2014*, 59(2), 54-55.
- S. Habas, F. Baddour, D. Ruddy, M. Pan, C. Nash, J. Wang, J. Hensley, J. Schaidle, A Facile Single-Source Molecular Precursor Route to Metal Phosphide Nanoparticles and their Evaluation as Hydrogenation Catalysts, *Chemistry of Materials*, submitted.
- F. Baddour, D. Ruddy, J. Schaidle, Synthesis of Molybdenum Carbide Nanoparticles Employing an SBA-15 Hard Templating Method, *manuscript in preparation.*
- J. Schaidle, J. Blackburn, J. Clark, C. Nash, K. Steirer, J. Wang, D. Ruddy, D. Robichaud, Experimental and Computational Investigation of the Acetic Acid Deoxygenation Mechanism over Molybdenum Carbide, *manuscript in preparation*.
- M. Griffin, F. Baddour, S. Habas, J. Hensley, D. Ruddy, J. Schaidle, An Investigation into the vapor phase deoxygenation of guaiacol over metal and metal phosphide catalysts, *Topics in Catalysis*, *manuscript in preparation*.

Patents/ROIs

- D. Ruddy, J. Schaidle, J. Blackburn, Synthetic routes for shape- and composition-controlled metal carbide and metal nitride nanocrystal catalysts, NREL ROI-12-00034.
- D. Ruddy, S. Habas, J. Schaidle, M. Griffin, Novel pretreatment procedure for the activation of supported colloidal nanoparticle catalysts, NREL ROI-14-11.
- J. Wang, D. Ruddy, S. Habas, A General Method for the Synthesis of Colloidal Single Crystalline Metal Phosphide Nanoparticles, NREL ROI-13-00061.
- J. Wang, D. Ruddy, S. Habas, F. Baddour, Metal Phosphide Catalysts and Methods for Making the Same and Uses Thereof, NREL PROV/13-62, 2015.

Awards

- J. Schaidle, NREL Chairman's Award for Exceptional Performance, September 2014.

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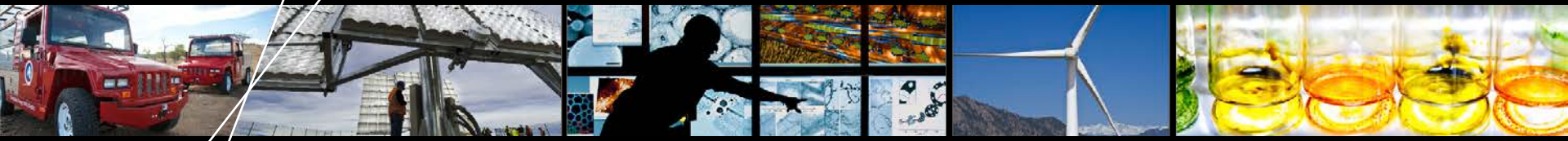
Milestones



Milestones-FY15

Level	Performance Measure	Planned Completion Date
Deliverable	Characterization and Evaluation of Colloidal Ligand-capped Nanoparticles: Determine the key physical and/or chemical attributes of colloidal ligand-capped nanoparticles that provide their superior upgrading performance compared to standard incipient wetness, bulk catalysts. This study will determine these attributes using two (2) probe tests: transformation of the ligands under pre-treatment and reaction conditions and external ligand exchange. These tests will be combined with CO or H_2 chemisorption, DRIFTS, XPS, *in-situ* NMR, and/or thermogravimetric analysis. Results from this milestone will be used to guide catalyst selection and activation procedures for the FY15 Q2 milestone.	12/31/2014
Regular	Catalyst Screening for *Ex-situ* CFP: Evaluate a series of at least ten (10) active phase-support combinations for high temperature (300-500°C), low pressure (<1MPa) *ex-situ* CFP of at least one (1) model pyrolysis compound. The active phase materials will be selected from our FY14 Q2 milestone and from materials predicted by the Computational Pyrolysis Consortium (WBS 2.5.1.302) to have high performance. Support materials will be selected from our FY14 Q3 milestone. Active phase-support combinations will be ranked for performance in terms of i) their abilities to increase the ratio of H:C and decrease the ratio of O:C from reactants to products, ii) higher selectivities to larger carbon number products, and iii) relative deactivation rates. Catalyst rankings will inform the down-selection of two (2) to three (3) catalysts for further study. These down-selected materials will be identified as the state-of-the-art materials in 2015 and will be given to the Catalytic Pyrolysis Science project (WBS 2.3.1.313) for evaluation under real pyrolysis vapors in FY15 Q3.	3/31/2015
Deliverable	Demonstrate C-C Coupling Capability: Demonstrate the coupling of at least two (2) model *ex-situ* CFP intermediates over at least two (2) commercial or in-house catalysts under conditions relevant to *ex-situ* CFP. The model *ex-situ* CFP intermediates will be selected based on the results from the FY15 Q2 milestone.	6/30/2015
Deliverable	Hydrodeoxygenation of Catalytically Upgraded Bio-oils: Compare the hydrotreating severity required to deoxygenate three (3) upgraded whole bio-oils collected from *ex-situ* CFP over the catalysts down-selected in the FY15 Q2 milestone or over HZSM-5. The results for the upgraded oils will be compared with those of representative whole non-upgraded pyrolysis oil. The evaluation metrics will be temperature and H_2 consumption.	9/30/2015

Milestones-FY16

Level	Performance Measure	Planned Completion Date
Deliverable	Demonstrate Scale-up of the Colloidal Nanoparticle Synthesis Method: Demonstrate the scale-up of our colloidal nanoparticle synthesis method from 5g of supported catalyst (5wt% active phase) per batch to 200g per batch while maintaining similar physical properties (i.e., particle size and shape, phase purity, crystallinity). Provide an estimate of synthesis method costs and potential pathways to reduce cost.	12/31/2015
Regular	Advanced Catalyst Testing for *Ex-situ* CFP: Evaluate a series of at least six (6) advanced catalysts for *ex-situ* CFP of at least two (2) model pyrolysis compounds. These catalyst formulations will be guided by results from the FY15 Q2 and Q3 milestones, the Computational Pyrolysis Consortium (WBS 2.5.1.302), and selective hydrogenation studies at the University of Michigan. The target for this milestone is to improve the extent of hydrogenation and deoxygenation by 15% and the extent of C-C coupling by 30% compared to the state-of-the-art catalysts down-selected in FY15.	3/31/2016
Go/No-Go	Feasibility Assessment of Colloidal Nanoparticle Catalysts: Based on results from the FY16 Q1 and Q2 milestones, determine the feasibility of using colloidal nanoparticle catalysts in a commercial-scale process. The metrics for evaluating feasibility will be cost ($/kg-catalyst), scalability (compared to common bulk synthesis methods), and *ex-situ* CFP performance (compared to similar materials synthesized using bulk methods). Provide cost estimates to TC Analysis team (WBS 2.1.0.302).	3/31/2016
Deliverable	Evaluation of Bench-Scale *Ex-Situ* CFP Performance with Complex Model Compound Feed Mixtures and Real Biomass Pyrolysis Vapors: Down-selected catalysts from the FY16 Q2 milestone will be tested at the bench-scale (~1g) for *ex-situ* CFP with (i) a mixture containing at least two (2) pyrolysis model compounds and steam and (ii) real biomass pyrolysis vapors. The effect of steam and additional compounds with increased functionality will be established, and the relationship between model compound experiments and real pyrolysis vapor experiments will be elucidated.	9/30/2016

Milestones-FY17

Level	Performance Measure	Planned Completion Date
Deliverable	Catalyst Scale-Up for Large Bench-Scale Experiments: Produce ~200g of each of the catalysts down-selected in the FY16 Q2 milestone. These materials will be used for tests in FY17 Q2 and Q4 milestones.	12/3/2016
Regular	Generation of *Ex-situ* CFP kinetic data for Process Models: Generate a set of kinetic expressions (e.g., power laws) for *ex-situ* CFP of model compound mixtures over the down-selected catalysts in FY16 that capture deactivation and the effects of temperature, pressure, steam concentration, and hydrogen concentration. Provide these kinetic models to the TC Analysis team (WBS 2.1.0.302).	3/31/2017
Deliverable	Evaluation of Reactor Configuration and Design: In conjunction with the TC Analysis team (WBS 2.1.0.302), evaluate reactor configurations (e.g., fixed bed, fluidized bed, circulating bed) based on reactor cost and properties of the down-selected catalysts (e.g., attrition resistance, surface area, deactivation rate), and identify two (2) configurations to be further investigated.	6/30/2017
Regular	*Ex-Situ* CFP of Real Pyrolysis Vapors at the Large-Bench Scale: Demonstrate *ex-situ* CFP of real biomass (pine) pyrolysis vapors at the large-bench scale (~200g of catalyst) over one (1) of the advanced catalysts developed during FY14-FY17 for 24 hours on stream while achieving our 2017 intermediate technical targets of 34% carbon efficiency to the organic phase and 12.5wt% oxygen content in the oil.	9/29/2017