2013 DOE Bioenergy Technologies Office (BETO) Project Peer Review

Catalytic Conversion to Liquid Hydrocarbons from Pyrolysis Oil and Syngas

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Bio-Oil Technology Area Review

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On many slides, the slide notes section has important additional information

Goal/Objective Statement

- MSU pyrolysis/syngas program goals:
  - Develop hydrocarbon and biodiesel fuels from raw bio-oil produced from southern pine and agricultural products and residues; develop auger reactor designs that can provide test quantities of raw bio-oil; test designs at pilot plant scale
  - Develop technologies for biomass gasification and catalytic conversion of cleaned syngas to hydrocarbon biofuels. This is accomplished through the design and optimization of syngas-to-hydrocarbons pilot plant, including biomass gasification, syngas cleanup, syngas upgrading as well as new catalyst material development.
- The MSU pyrolysis/syngas program goals support the development of technologies for conversion of forest resources into cost-competitive liquid fuels, such as renewable gasoline, jet fuel and diesel. Technologies to be tested at laboratory (and pilot scale in FY14).

Project Quad Chart Overview

Timeline

•Project start date: June 1, 2006

•Project end date: June 30, 2014

•Percent complete:

SERC phase I: 100%

SERC phase II: 100%

SERC phase III: 90%

SERC phase IV: 40%

Budget

- Total project funding
  - DOE share: $15,587,449
  - Contractor share: $4,414,254
- Funding received in FY 2011 (DOE & cost share): $4,597,536
- Funding in FY 2012 (DOE & cost share): $4,922,748
- ARRA Funding: $0
- Years the project has been funded & average annual funding: 8 years, $2,500,213 (DOE & cost share)

Barriers

- Barriers addressed
- Pyrolysis
  - Tt-A Feeding dry biomass
  - TT-G Fuel synthesis and upgrading
- Syngas
  - Tt.-F. Syngas Cleanup and Conditioning
  - Tt-G. Fuels Catalyst Development

Partners & Roles

Pyrolysis

•Piedmont Bio-Products: licensee

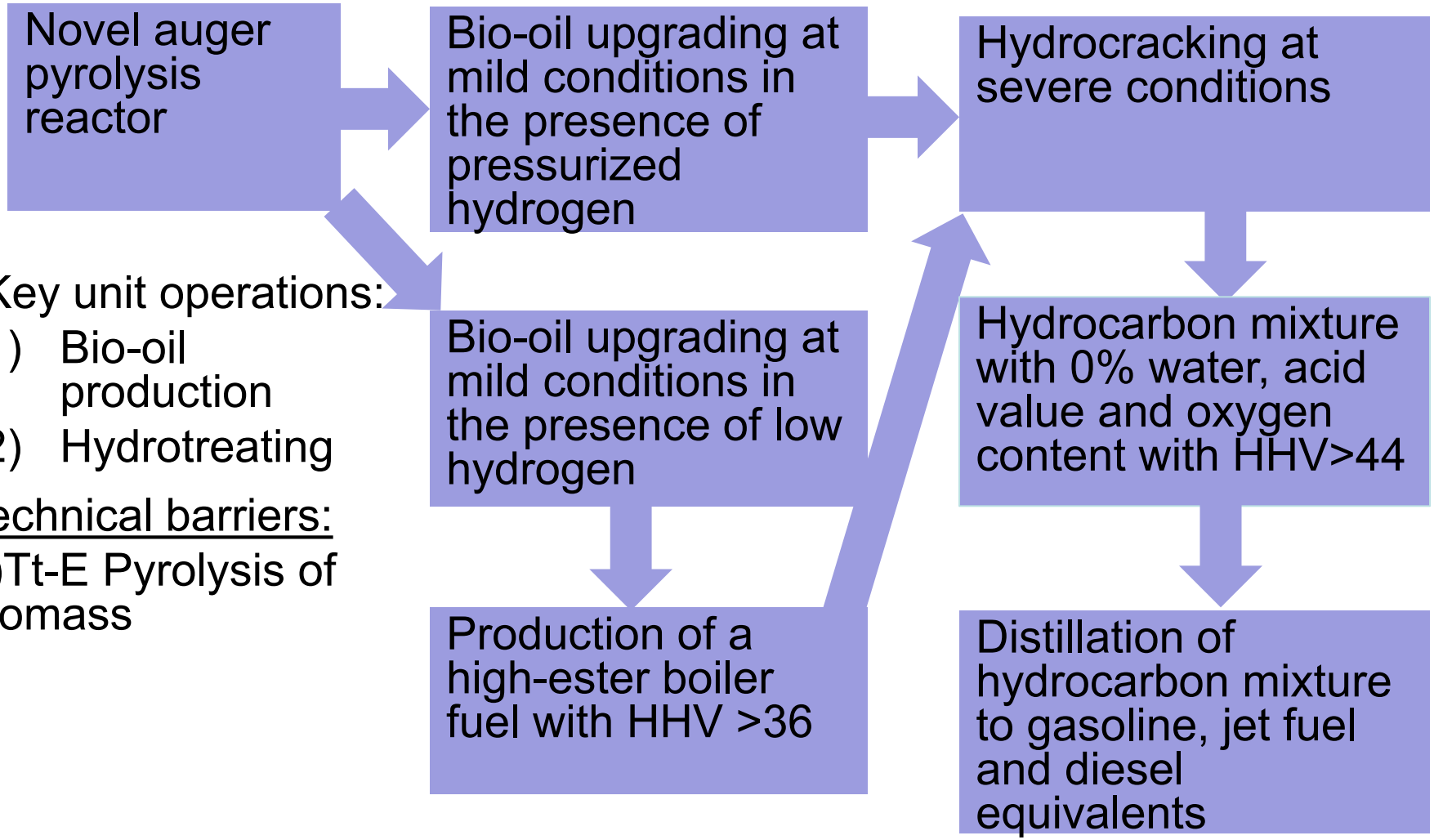
Syngas

•MS Choctaw Lignite LLC.

•Harrelson & Associates

Project Overview

Pyrolysis



Key unit operations:

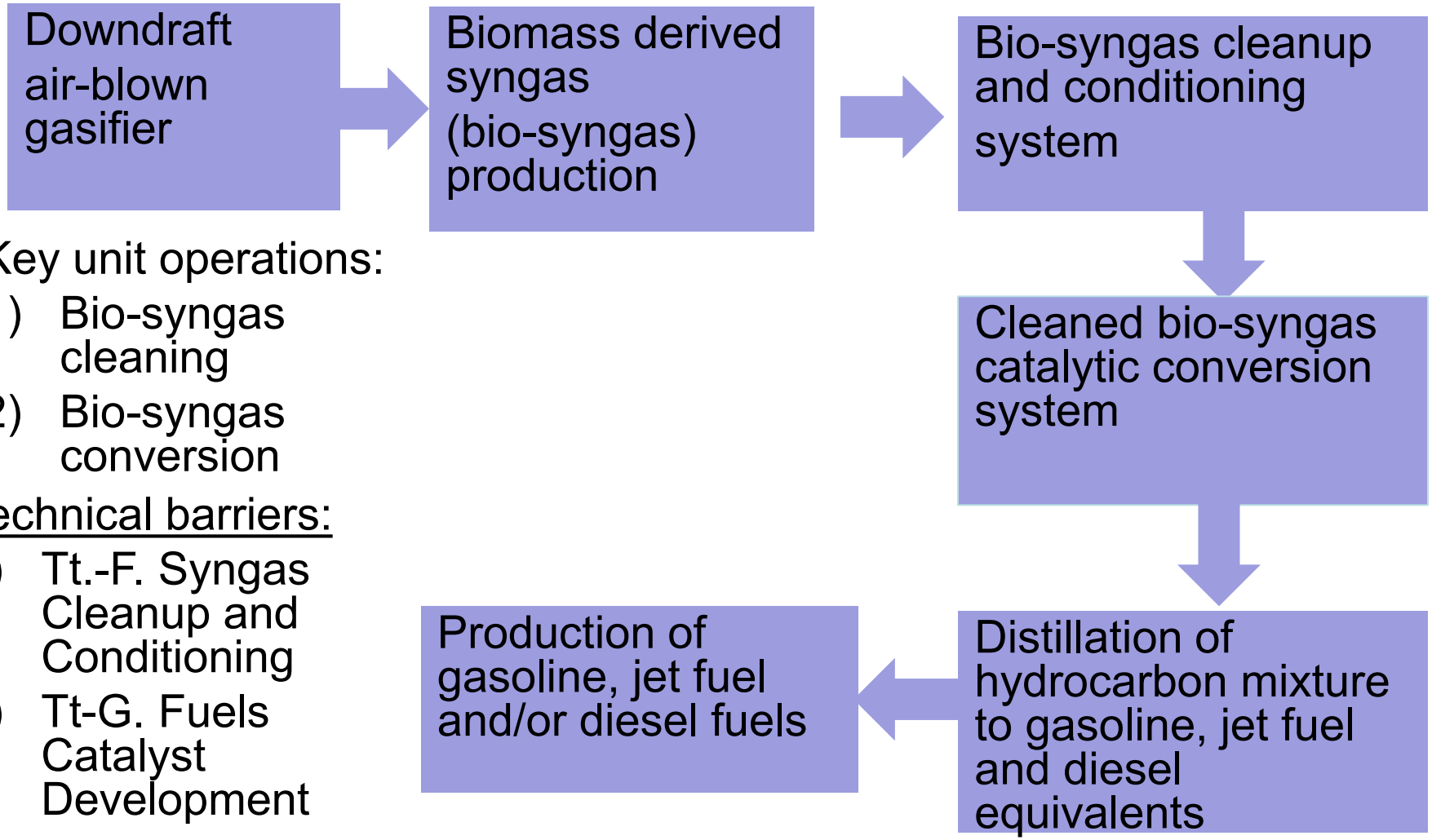
1) Bio-oil production
2) Hydrotreating

<u>Technical barriers:</u>

1)Tt-E Pyrolysis of biomass

Project Overview

Syngas



Key unit operations:

1) Bio-syngas cleaning
2) Bio-syngas conversion

<u>Technical barriers:</u>

1) Tt.-F. Syngas Cleanup and Conditioning
2) Tt-G. Fuels Catalyst Development

1 - Approach

Pyrolysis

The approach is to produce high quality bio-oil with a novel design auger reactor and test design at pilot plant scale; the bio-oil is dexoxygenated in a first stage with pressurized hydrogen ; this first stage can be utilized as a boiler fuel or hydrocracked to produce a hydrocarbon mixture comprised of (by simulated dist.) 55% gasoline, 30% jet fuel, 15% diesel. The research has also investigated hydrotreating with a low hydrogen gas which is estimated to conserve more than 60% of the hydrogen normally utilized during the deoxygenation stage.

1 - Approach

Syngas

The approach is to develop a pilot scale continuous process including catalyst preparation, gasification, syngas cleaning, and Fischer–Tropsch synthesis (FTS). Multi-functional catalysts are designed and tested for catalytic converting cleaned bio-syngas to biofuels. The activities, selectivity, stability, and life-time of the catalyst will be evaluated.

Pyrolysis and Syngas

This project has also utilized go/No-Go decision points numerous times as some projects were terminated for lack of results. We utilized technical metrics that ranged from in-depth to cursory. This depended on the stage and perceived future success of the project.

2 - Technical Accomplishments/ Progress/Results

Pyrolysis

- Design and fabrication of a 4-ton per day pyrolysis reactor (Pat. Pend.) with biomass processing equipment

2 - Technical Accomplishments/ Progress/Results

Pyrolysis

- Partial deoxygenation via low hydrogen consumption

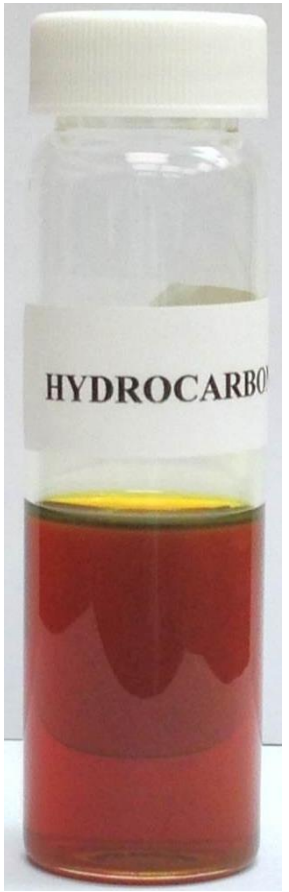
	Raw Bio-oil	Hydrogen	Low Hydrogen
Acid value	98.0	55.5	51.6
HHV, MJ/kg	16.1	34.7	36.5
Water, %	30.6	3.1	2.7
C,%	36.2	73.7	76.4
H,%	7.8	9.7	9.1
N,%	0.03	0	0
O,%	56.0	15.2	14.0

2 - Technical Accomplishments/ Progress/Results

Pyrolysis

- Full deoxygenation to produce mixed hydrocarbons with zero oxygen, acid value and water.

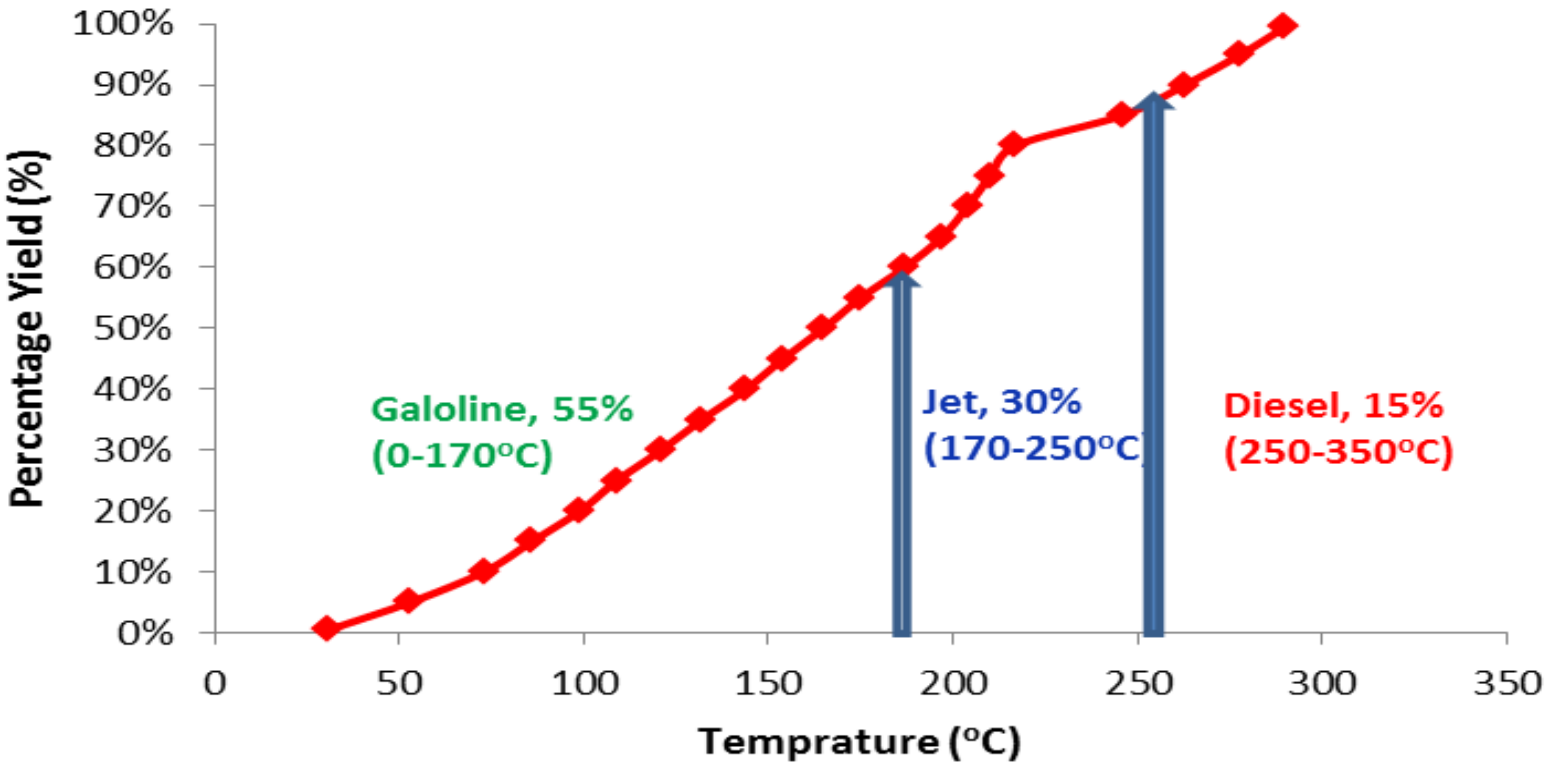
	Raw Bio-oil	HC bio-oil (H_2)	Diesel
Acid value	98.0	< 0.1	0
HHV, MJ/kg	16.1	43.0	45.8
Water, %	30.6	0.5	0
C,%	36.2	86.2	85.1
H,%	7.8	12.1	12.2
N,%	0.03	0.01	0
O,%	56.0	1.5	0



2 - Technical Accomplishments/ Progress/Results

Pyrolysis

- Simulated distillation showing the petroleum equivalent components of the mixed hydrocarbons produced by full deoxygenation.



2 - Technical Accomplishments/ Progress/Results (cont'd)

Pyrolysis

- Projects related to pilot plant production of bio-oil and fuels production:
  - Design and testing of a laboratory scale catalytic continuous packed bed and fluidized bed reactor for hydrotreating and hydrocracking
- Projects completed at this time:
  - Increase and utilization of anhydrosugars by spray method in the pyrolysis reactor. Project is complete, patent is pending.
  - Olefination/esterification of bio-oil to produce a high energy boiler fuel. Project complete and patent pending.
  - Produce hydroxymethyl furfural from maximized bio-oil anhydrosugars. Project complete.

2 - Technical Accomplishments/ Progress/Results

Syngas

- Development of both lab scale and pilot scale bio-syngas cleaning system to remove tar, oxygen, ammonia, and sulfur etc.

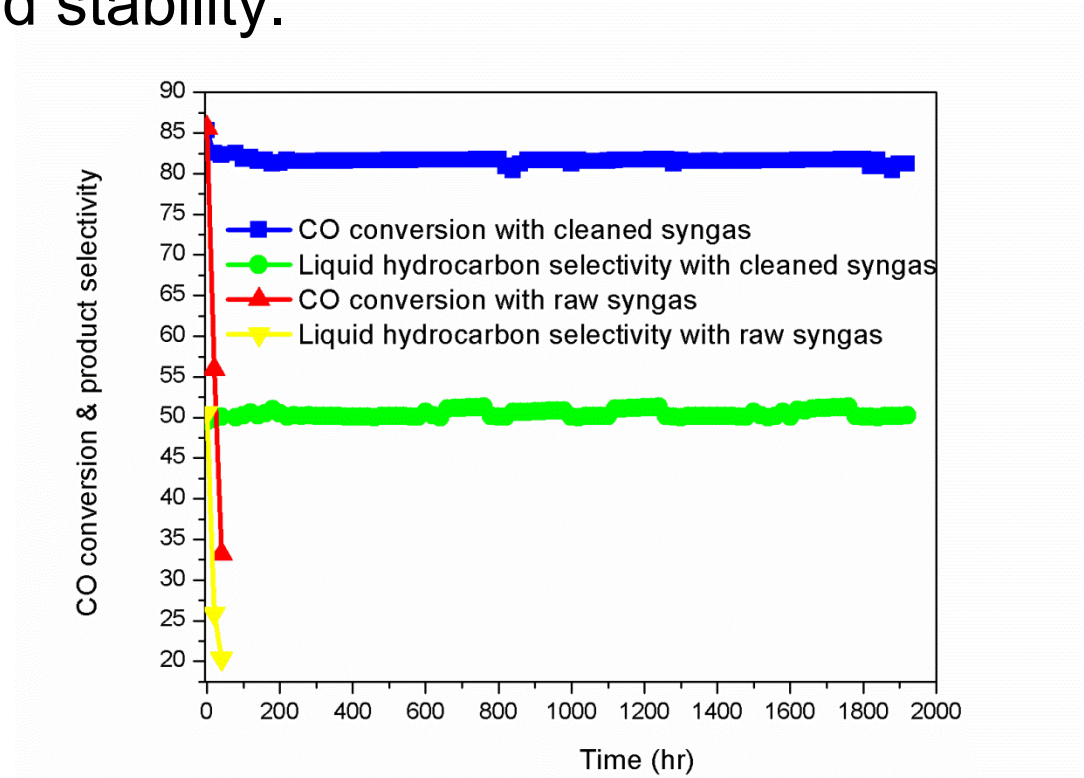
Impurities identified from bio-syngas before and after cleaning

Components	Tar	Oxygen	NH_3	H_2S + COS
Before Cleaning	500-3000 ppm	0.1~2%	200-1000	200-400 ppm
After Cleaning	<1ppm	<0.1ppm	<1ppm	<1ppb

2 - Technical Accomplishments/ Progress/Results

Syngas

- After bio-syngas cleaning, the selected catalyst showed better life time and stability.



Time on stream of CO conversion, light hydrocarbon and liquid product selectivity on the catalyst at 260°C, 1000 psig, 3000 h^{-1} with raw and cleaned bio-syngas.

2 - Technical Accomplishments/ Progress/Results

Syngas

- Design and fabrication of a 1-gallon liquid hydrocarbon per day pilot scale FT reactor.

2 - Technical Accomplishments/ Progress/Results

Syngas

- Development of a route to a Jet fuel sample with bio-syngas catalytic conversion.

Properties	Syncrude	Distilled fuel	Jet Fuel A
Average Molecular Weight	125.50	137.98	140.65
Relative Density	0.804	0.810	0.815
Reid Vapor Pressure @ 100°F(37.8 °C)	0.55	0.16	0.04
Percent Carbon	86.58	86.61	86.95
Percent Hydrogen	13.29	13.36	13.05
Bromine Number (Calc)	26.16	6.12	3.18
Total Oxygen Content (mass %)	0.12	0.025	0.0044
Freeze point (°C)	-28.5	-37	-40

2 - Technical Accomplishments/ Progress/Results

Syngas

- Development of a route to a wide-cut diesel sample with bio-syngas catalytic conversion.

Properties	Distilled fuel	No. 2 Diesel
Specific gravity at 15 °C (Kg/m^3)	840	845
Flash point (°C)	95	75
Average Molecular Weight	183.6	184.5
Total Oxygen Content (mass %)	0.015	-
Boiling point (°C)	177-355	188-345

2 - Technical Accomplishments/ Progress/Results (cont'd)

Syngas

- Projects related to pilot plant production of biofuels via syngas route:
  - Design and testing of bio-syngas purification system
  - Optimize the catalytic process conditions to account for the effects of impurities in the bio-syngas
  - Complete the process design and economic analysis
- Projects completed at this time:
  - Both pilot scale bio-syngas purification system and pilot scale catalytic conversion system were designed, fabricated and installed. Project complete.
  - Several multi-functional catalysts were screened and chosen for bio-syngas conversion into wide-cut diesel and jet fuel hydrocarbons in lab scale FT reactors. Project complete, provisional patent filed.

3 - Relevance

- The novel auger pyrolysis technology and the capability to reduce hydrogen costs during hydrotreating contributed to the platform goal of increasing the cost competiveness of bio-oil fuels.
- The novel bio-syngas based catalytic conversion technology to produce wide-cut diesel and jet fuel range hydrocarbons contributed to the platform goal of increasing the cost competiveness of bio-fuels.
- The project is highly cognizant of the need to commercialize less costly pathways to biofuels from bio-pyrolysis oil and syngas.

4 - Critical Success Factors

- Technical: Production of fungible hydrocarbons equivalent to petroleum fuels;
- Market/business: Biofuels must be produced at a cost that is competitive
- Challenges to pyrolysis route: 1) Optimize the low hydrogen approach to boiler fuel and fungible hydrocarbons production; 2) Minimize catalyst costs and maximize yield of the low hydrogen approach to assure commercial success.
- Significant reduction of hydrogen cost for hydroprocessing will advance the technology of deoxgenation and greatly reduce the cost of production of fuels from bio-oil.
- Challenges to syngas route: 1) Improve the lifetime and stability of the catalysts from bio-syngas; 2) Demonstrate high conversion yields and high selectivity at pilot scale; and 3) Reduce costs through process integration.

5. Future Work

Pyrolysis

- MSU pyrolysis pilot plant will be completed; low hydrogen approach will be optimized and a low-cost catalyst will be developed.

Complete pilot plant	Quarter 1	Quarter 2	Quarter 3	Quarter 4
Optimize pyrolyzer bio-oil production	→			
Complete biomass processing system	→	→		
Produce bio-fuels in 4.5 gal stirred reactor	→	→	→	
Testing and analysis of fuels for fungibility	→	→	→	→

5. Future Work

Syngas

- MSU syngas pilot plant will be completed and tested; the economic analysis will be performed using the data from the pilot-scale process.

Complete pilot plant	Quarter 1	Quarter 2	Quarter 3	Quarter 4
Complete pilot plant via syngas route				
Test selected catalysts in the pilot bio-syngas cleaning reactor				
Test selected catalysts in the pilot FT reactor				
Perform the economic analysis				

Summary

- **Relevance:** Addresses barrier Tt-E to produce high quality bio-oil and produce biofuels at competitive cost. Addresses barrier Tt-F and Tt-G to produce biofuels from biosyngas.
- **Approach:** Laboratory scale and pilot scale production of bio-oil, syngas and biofuels.
- **Technical accomplishments:** Four projects completed; 4-ton per day pyrolysis reactor being optimized for bio-oil production; low hydrogen bio-oil successfully produces boiler fuel and hydrocarbons; both pilot scale bio-syngas purification system and pilot scale catalytic conversion system were installed. Several multi-functional catalysts were screened and chosen for bio-syngas conversion into biofuels.
- **Future work:** Completion of pilot plant; production of biofuels at pilot plant scale.
- **Success factors and challenges:** Bio-oil and biofuels produced at commercially viable cost.
- **TT:** Patents and numerous papers have presented this work to a wide audience.

Additional Slides

Responses to Previous Reviewers' Comments

- Reviewer: The scope and approach of the project is quite wide. Response: Researchers have responded by limiting their projects to only those relevant to the pilot plant activity of producing lower cost bio-oil and biofuels.
- Reviewer: Lack description of their technical approach.

  Response: The lack of description was related to our patenting program. DOE instructions are not to mention patentable technology approaches.
- Reviewer: MSU appears to be doing quality research but I would like to see better coordination with the TC platform.

  Response: MSU is attempting to solve the barriers defined in TC platform Tt-E: Improvement in bio-oil quality and catalytic production of fuels from bio-oil. MSU has collaborated with PNNL in catalyst design and with INL in feedstock production.

Publications and Presentations

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