Fractional Multistage Hydrothermal Liquefaction of Biomass and Catalytic Conversion into Hydrocarbons
Goal Statement

Project Goal – Develop a novel Multistage Hydrothermal Liquefaction (HTL) of biomass and integrate with Virent’s Catalytic BioForming® Process to efficiently produce cost effective “drop-in” fuels from woody biomass and corn stover, with particular focus in maximizing jet fuel and diesel yields.

- Developing commercially Viable Bioenergy Technology
  - Improve pretreatment strategies
  - Improve fuel yields

- Reduction of Greenhouse Gas Emission
  - Non-Food Feedstock – Woody Biomass, Corn Stover
  - Improve fuel yields

- Process Generates “Direct Replacement” Hydrocarbons compatible with today’s transportation infrastructure
  - Distillate Range Products for use as either jet fuel or diesel fuel
  - “Advantaged” Jet and Diesel Fuels

- Relevance and Tangible Outcomes for the United States
  - Promotes National Security
  - Growing a Sustainable future
  - Generating green jobs
Quad Chart Overview

**Timeline**
- Project Start: October 2013
- Project End: September 2016
- Percent complete: ~50%

**Barriers**
- Tt-B: Feeding Wet Biomass
- Tt-D: Biomass Pretreatment
- Tt-F: Deconstruction of Biomass to Form Bio-Oil Intermediates
- Tt-J: Catalytic Upgrading of Bio-Oil Intermediates to Fuels and Chemicals

**Budget**

<table>
<thead>
<tr>
<th></th>
<th>FY10–FY12 Costs</th>
<th>FY13 Costs</th>
<th>FY14 Costs</th>
<th>FY15-End Costs</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>DOE Funded</strong></td>
<td>$0</td>
<td>$0</td>
<td>$451,831</td>
<td>$2,948,169</td>
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<tr>
<td><strong>Virent Cost Share</strong></td>
<td>$0</td>
<td>$0</td>
<td>$158,751</td>
<td>$1,035,843</td>
</tr>
</tbody>
</table>

**Partners**
- Idaho National Laboratory
  - Feedstock Supply
  - Biomass Pretreatment
1 - Project Overview

- Convert lignocellulosic feedstocks to drop-in liquid fuels
- Multistage Hydrothermal Liquefaction
  - Develop multistage HTL utilizing appropriate solvent and process conditions to convert woody biomass and corn stover to liquid intermediates that can be catalytically upgraded to “direct replacement” hydrocarbons
  - Progress from Applied Research to Preliminary Investigation
  - Focus on integration with Virent BioForming platform
- Pilot Build
  - Design, build, and operate a continuous pilot for multistage HTL technology
- Catalytic Upgrading
  - Integrate HTL technology with Virent BioForming catalytic upgrading to produce “direct-replacement” hydrocarbon liquid fuels
  - Focus on distillate fuels – jet fuel and diesel
- TEA and LCA
  - Complete Techno-Economic Analysis and Life Cycle Analysis
2 – Approach (Technical)

- Overall Technical Approach

- Critical Success Factors – Feedstock, Biomass Pretreatment, Deconstruction, Hydrolysate Conditioning, Catalytic Conversion, Product Certification, Overall Process Economics

- Potential Challenges – Biomass to usable carbon, HTL product integration into catalytic upgrading, Intensify process by eliminating processing steps

- Each zone will target solubilizing a specific biomass fraction (hemicellulose, cellulose, or lignin) with an appropriate solvent

- Solvents may be internally generated or economically obtained
2 – Approach (Technical)

- Critical Success Factors
  - Ash Removal
    - Necessary to Reduce Potential Catalyst Poisons
  - Carbon Recovery
    - Maximize carbon recovery from hemicellulose and cellulose
    - Maximize the solubilization and conversion of lignin components
    - Maximize carbon conversion in the catalytic conversion to desired liquid hydrocarbon
  - Process Economics – Reduction in capital and operating cost of biomass to jet fuel distillate

- Potential Challenges
  - Improve yields of “convertible carbon” intermediate streams through HTL process optimization
  - Removal of ash components in preconversion and processing steps.
  - Process Intensification through the potential elimination of HTL and/or catalytic processing steps
INL Tasks

• Supply formatted loblolly pine to Virent upon request (up to three shipments)
  – Characterize with proximate/ultimate, ash composition and calorific analyses

• Perform chemical preconversions on corn stover using the Chemical Preconversion System and subsequent bench top extractions/washes
  – Characterize ash and nitrogen removal and changes to ash composition
    • SiO₂, alkali metals, alkaline earth metals and nitrogen
  – Supply samples to Virent for testing

• Optimize chemical preconversion process using sequential alkali/acid treatments and extractions/washes based on Virent’s input for desired results
Why Is Ash A Quality Issue?

- Biomass contains both introduced soil ash and endogenous ash
- Endogenous ash is comprised of structural and non-structural physiological ash
- Ash is comprised of metals and heteroatoms that may be
  - Inert
  - Destructive to conversion products
  - Fouling agents for conversion catalysts
  - Sources of pollutants
  - Damaging to equipment
- This increases processing costs and/or reduces product yields
- Knowing the chemical form, function and plant tissue location of specific ash components aids in identifying effective reduction methods
2 – Approach (Management)

- **Critical Market and Business Success factors**
  - Establish Cost and Technical Targets for Catalytic derived hydrocarbon fuels based on TEA
  - Market Size and Opportunity

- **Potential Market and Business Challenges**
  - Biomass Cost, Crude Oil Prices, Financing of Plant, Government Policy

- **Management approach**
  - Gantt Chart for 3 year project
  - Key Milestones
    - TRL-2 Milestones (September 2014)
    - TRL-3 Milestones (September 2015)
    - Stage Gate Review (September 2015)
    - TRL-4 Milestones (September 2016)
### 3 – Technical Accomplishments/Progress/Results

<table>
<thead>
<tr>
<th>WBS</th>
<th>Milestone Title</th>
<th>Description</th>
<th>Deliverable</th>
<th>Complete?</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.2.2</td>
<td>Develop Initial Kinetic Model</td>
<td>Develop an initial kinetic model based on preliminary investigation</td>
<td>Report initial Kinetic Model results in the quarterly report</td>
<td>✔️</td>
</tr>
<tr>
<td>1.2.1</td>
<td>TRL-2: Applied Research</td>
<td>Demonstrate carbon efficiency of &gt;60% for HTL from biomass to soluble carbon in batch tube experiments.</td>
<td>Report carbon efficiency in the quarterly report</td>
<td>✔️</td>
</tr>
<tr>
<td>1.2.3</td>
<td>TRL-2: Applied Research Catalytic Upgrading</td>
<td>Demonstrate &gt;65% yield to liquid fuel products with model feeds</td>
<td>Report carbon efficiency in the quarterly report</td>
<td>✔️</td>
</tr>
</tbody>
</table>
3 – Technical Results – Yield

- Milestone of >60% C to liquid products from real feedstock demonstrated
- Hemicellulose, cellulose, and lignin liquefied

- Milestone of >65% C to liquid fuels from model HTL intermediates demonstrated
- Finished fuel meets jet fuel and diesel specifications
3 – Technical Results – Kinetic Model

- Initial kinetic milestone met
- Use of flow through system to accurately capture kinetics to apply to pilot design
- Model will be verified, expanded, and refined with pilot
3 – Technical Results – Pilot Build

- Initial design of continuous HTL pilot complete
  - Feed System, HTL Reactor, Solid/Liquid Separation, Product Collection
- All major equipment identified, utilizing scalable and economical process design
- Build to be completed mid-2015
4 – Relevance

- **Contributions to meeting the platform goals and objectives of the BETO Multi-Year Program Plan**
  - If successful, project would address the strategic goal to develop commercially viable technologies for converting biomass into energy-dense, fungible finished liquid fuels, such as renewable jet and diesel.
  - This project utilizes analysis interface through the use of TEA and LCA to inform feed collection methods and processing steps.
  - Working with INL will investigate feedstock supply, preconversion technologies, and logistics interfaces.
  - Biofuels distribution infrastructure interface will be addressed through ASTM certification of resulting jet fuel, and diesel product qualification.
  - Addresses barriers such as
    - Tt-B Feeding Wet Biomass
    - Tt-C Biomass Pretreatment
    - Tt-F Deconstruction of Biomass to Form Bio-Oil Intermediates
    - Tt-J Catalytic Upgrading of Bio-Oil Intermediates to Fuels and Chemicals
    - Tt-K Product Finishing
    - Tt-L Knowledge Gaps in Chemical Processes

- **Applications of the expected outputs in the emerging bioenergy industry**
  - Results from this project provide technical viability of combining a biochemical conversion technology frontend with a chemical (catalytic) conversion technology to generate “direct replacement” hydrocarbons from a lignocellulosic feedstock.
  - Project has resulted in 1 US Patent application
## 5 – Future Work

<table>
<thead>
<tr>
<th>WBS</th>
<th>Milestone Title</th>
<th>Description</th>
<th>Deliverable</th>
<th>Estimated Completion</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.2.3</td>
<td>Refine Kinetic Model</td>
<td>Refine kinetic model using liquefaction data from the prototype unit</td>
<td>Report the refined Kinetic Model in the quarterly report</td>
<td>8/4/15</td>
</tr>
<tr>
<td>2.3</td>
<td>TRL-3: Proof of Concept</td>
<td>Demonstrate &gt;50% yield to liquid fuel products from biomass- 70% recovery</td>
<td>Report the results in this the quarterly report</td>
<td>9/30/15</td>
</tr>
<tr>
<td></td>
<td>Demonstrated Yield in HTL and 70% in catalytic step</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2.4</td>
<td>TEA and LCA</td>
<td>Complete intermediate TEA and LCA on the process configuration. Demonstrate &gt;60% GHG reduction in LCA</td>
<td>Stage Gate TEA and LCA Report</td>
<td>9/30/15</td>
</tr>
<tr>
<td>4.2</td>
<td>Stage Gate Review</td>
<td>Hold Stage Gate review to assess TRL-3 status and determine if the project should proceed to TRL-4</td>
<td>Go/No-Go Decision</td>
<td>9/30/15</td>
</tr>
<tr>
<td>3.3</td>
<td>TRL-4: Preliminary Integration</td>
<td>Demonstrate &gt;60% yield to liquid fuel products from biomass, with 75% recovery in catalytic step</td>
<td>Report the results in this the quarterly report</td>
<td>9/30/16</td>
</tr>
<tr>
<td></td>
<td>Demonstrated Yield recovery in catalytic step</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>3.4</td>
<td>Final TEA and LCA</td>
<td>Complete TEA and LCA on the final process configuration.</td>
<td>Final TEA and LCA Report</td>
<td>9/30/16</td>
</tr>
</tbody>
</table>
Summary

1. Overview
   - Convert lignocellulosic feedstocks to drop-in liquid fuels
   - Develop novel Multistage Liquefaction and integrate with Virent’s BioForming Catalytic Upgrading

2. Approach
   - Progress from TRL-2 Applied Research to TRL-4 Preliminary Integration

3. Technical Results
   - TRL-2 Applied Research completed: yield milestones met, initial kinetic model developed, pilot design completed

4. Relevance
   - Demonstrate technical and economic viability of process

5. Future Work
   - Complete pilot build, meet TRL-3 Proof-of-Concept and TRL-4 Preliminary Integration milestones
Additional Slides
Responses to Previous Reviewers’ Comments

- This project has not been previously presented for Peer Review
Publications, Patents, Presentations, Awards, and Commercialization

- Patent Application
- Multi-Stage Biomass Liquefaction
- Nelson et al.
- US 62/009,500
- 6/9/2014
- Status – Pending
2. Approach (Technical)

Current State of Technologies

- Currently the Nighthawk project is mid Stage 2 which correlates to TRL-3.
- Completion of this project would put the project at the end of Stage 3 and ready to advance to TRL-5.
Large Addressable Market Sizes

The ultimate market for any biofuel is the national and global refined product markets, which collectively represents over a trillion dollar business. In these markets, the US consumes significant amounts of liquid transportation fuels with a large portion derived from imported crude oil.

<table>
<thead>
<tr>
<th>Fuel Type</th>
<th>Current Consumption in 2012 (MMGPY)¹</th>
<th>Projected Consumption in 2040 (MMGPY)</th>
<th>CAGR (%) 2012 – 2040</th>
</tr>
</thead>
<tbody>
<tr>
<td>Motor Gasoline</td>
<td>133,378</td>
<td>101,373</td>
<td>(1.00%)</td>
</tr>
<tr>
<td>Jet Fuel</td>
<td>21,845</td>
<td>24,369</td>
<td>0.50%</td>
</tr>
<tr>
<td>Distillate Fuel Oil</td>
<td>59,772</td>
<td>70,836</td>
<td>0.80%</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>DOD Fuel Requirements²</th>
<th>FY 2012 (MMGPY)</th>
<th>Product Cost ($MM)</th>
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</thead>
<tbody>
<tr>
<td>Jet Fuel</td>
<td>3,521</td>
<td>11,842</td>
</tr>
<tr>
<td>Distillates and Diesel</td>
<td>865</td>
<td>2,764</td>
</tr>
<tr>
<td>Totals</td>
<td>4,386</td>
<td>14,606</td>
</tr>
</tbody>
</table>

1) EIA Annual Energy Outlook 2014 Early Release Reference Case
2) DLA Fiscal Year 2012 Energy Facts. [http://www.energy.dla.mil/library/Pages/Publications.aspx](http://www.energy.dla.mil/library/Pages/Publications.aspx)
## 2. Approach (Management) (Market/Business Challenges)

<table>
<thead>
<tr>
<th>Business Risk</th>
<th>Mitigation Strategy</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Biomass Price Escalation and Sourcing</strong></td>
<td>Continue to work with groups like INL to determine costs of harvesting biomass. Continue to work on improving yields to lower effect of biomass price volatility on final fuel product.</td>
</tr>
<tr>
<td><strong>Prolonged Depression of Crude Oil Prices</strong></td>
<td>Strive to be the low cost producer of cellulosic hydrocarbon biofuels. Optimize byproduct streams for use as chemical/petrochemical to increase co-product value.</td>
</tr>
<tr>
<td><strong>Financing</strong></td>
<td>Financing will be determined by projected cost of production, profitability, capital risk, and optimal site location.</td>
</tr>
<tr>
<td><strong>Policy Uncertainty</strong></td>
<td>Continue promoting efforts and participation in groups like Advanced Biofuels Association (ABFA) and others.</td>
</tr>
<tr>
<td><strong>Emerging Competitive Technologies</strong></td>
<td>Monitor competitive landscape and continue to expand IP portfolio.</td>
</tr>
</tbody>
</table>
### Approach – Management

**Project Gantt Chart with Milestones**

<table>
<thead>
<tr>
<th>Task Name</th>
<th>Duration</th>
<th>Start</th>
<th>Finish</th>
</tr>
</thead>
<tbody>
<tr>
<td>DE-EE0006286</td>
<td>767 days</td>
<td>Tue 10/1/13</td>
<td>Thu 10/13/16</td>
</tr>
<tr>
<td>TRL-2 Applied Research</td>
<td>482 days</td>
<td>Tue 10/1/13</td>
<td>Thu 8/27/15</td>
</tr>
<tr>
<td>▪ TRL-2 Preconversion</td>
<td>482 days</td>
<td>Tue 10/1/13</td>
<td>Thu 8/27/15</td>
</tr>
<tr>
<td>▪ TRL-2 Liquefaction</td>
<td>430 days</td>
<td>Thu 10/31/13</td>
<td>Thu 7/16/15</td>
</tr>
<tr>
<td>▪ Develop Initial Kinetic Model</td>
<td>0 days</td>
<td>Thu 8/28/14</td>
<td>Thu 8/29/14</td>
</tr>
<tr>
<td>▪ TRL-2 Demonstrated Carbon Efficiency</td>
<td>0 days</td>
<td>Tue 9/30/14</td>
<td>Tue 9/30/14</td>
</tr>
<tr>
<td>▪ TRL-2 Catalytic Upgrading</td>
<td>415 days</td>
<td>Mon 12/2/13</td>
<td>Thu 7/23/15</td>
</tr>
<tr>
<td>▪ TRL-2 Catalytic Upgrading Demonstrated Yield</td>
<td>0 days</td>
<td>Tue 9/30/14</td>
<td>Tue 9/30/14</td>
</tr>
<tr>
<td>▪ TRL-3 Proof Of Concept</td>
<td>323 days</td>
<td>Mon 6/23/14</td>
<td>Wed 8/30/15</td>
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<tr>
<td>▪ TRL-3 Preconversion</td>
<td>268 days</td>
<td>Wed 9/10/14</td>
<td>Wed 9/30/15</td>
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<tr>
<td>▪ TRL-3 Liquefaction</td>
<td>285 days</td>
<td>Mon 6/23/14</td>
<td>Thu 8/8/15</td>
</tr>
<tr>
<td>▪ Complete Liquefaction Prototype Skid</td>
<td>0 days</td>
<td>Mon 1/5/15</td>
<td>Mon 1/5/15</td>
</tr>
<tr>
<td>▪ Refine Kinetic Model</td>
<td>0 days</td>
<td>Tue 8/4/15</td>
<td>Tue 8/4/15</td>
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<tr>
<td>▪ TRL-3 Demonstrated Carbon Efficiency</td>
<td>0 days</td>
<td>Wed 9/30/15</td>
<td>Wed 9/30/15</td>
</tr>
<tr>
<td>▪ TRL-3 Catalytic Upgrading</td>
<td>100 days</td>
<td>Thu 4/2/15</td>
<td>Thu 8/20/15</td>
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<tr>
<td>▪ TRL-3 Catalytic Upgrading Demonstrated Yield</td>
<td>0 days</td>
<td>Wed 9/30/15</td>
<td>Wed 9/30/15</td>
</tr>
<tr>
<td>▪ TEA / LCA</td>
<td>295 days</td>
<td>Mon 6/23/14</td>
<td>Thu 8/20/15</td>
</tr>
<tr>
<td>▪ TEA / LCA Completed</td>
<td>0 days</td>
<td>Wed 9/30/15</td>
<td>Wed 9/30/15</td>
</tr>
<tr>
<td>▪ TRL-4 Preliminary Integration</td>
<td>252 days</td>
<td>Thu 10/1/15</td>
<td>Fri 9/30/16</td>
</tr>
<tr>
<td>▪ TRL-4 Preconversion</td>
<td>240 days</td>
<td>Thu 10/1/15</td>
<td>Tue 9/13/16</td>
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<tr>
<td>▪ TRL-4 Liquefaction</td>
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<td>Mon 7/25/16</td>
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<td>0 days</td>
<td>Fri 9/30/16</td>
<td>Fri 9/30/16</td>
</tr>
<tr>
<td>▪ TRL-4 Catalytic Upgrading</td>
<td>150 days</td>
<td>Mon 12/21/15</td>
<td>Mon 7/25/16</td>
</tr>
<tr>
<td>▪ TRL-4 Catalytic Upgrading Demonstrated Yield</td>
<td>0 days</td>
<td>Fri 9/30/16</td>
<td>Fri 9/30/16</td>
</tr>
<tr>
<td>▪ TEA / LCA</td>
<td>50 days</td>
<td>Fri 7/8/16</td>
<td>Fri 9/16/16</td>
</tr>
<tr>
<td>▪ Final TEA / LCA</td>
<td>0 days</td>
<td>Fri 9/30/16</td>
<td>Fri 9/30/16</td>
</tr>
<tr>
<td>▪ Project Management</td>
<td>767 days</td>
<td>Tue 10/4/13</td>
<td>Thu 10/13/16</td>
</tr>
<tr>
<td>▪ Reporting</td>
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<tr>
<td>▪ Stage Gate</td>
<td>3 days</td>
<td>Mon 9/28/15</td>
<td>Wed 9/30/15</td>
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