

# **2013 DOE Bioenergy Technologies Office (BETO) Project Peer Review**

## **Mild Biomass Liquefaction Process for Economic production of Stabilized Refinery- Ready Bio-Oils**

May 20-24, 2013  
Bio-Oil Technology Area Review

Santosh Gangwal, Kevin McCabe, Andrew Campos and  
Demps Pettway

Southern Research Institute

# Goal/Objective Statement

## Project Goal

- Demonstrate a mild thermochemical liquefaction process to convert woody biomass to stabilized refinery ready bio-oils.

## Project Objectives

- Demonstrate process at laboratory scale.
- Partner with a refinery to move the technology towards commercialization.
- Move proposed technology from TRL-2 to TRL-4.

## FOA: Bio-oil Stabilization and Commoditization (DOE-FOA-0000686)

- Conduct R&D on conversion of high-impact biomass to bio-oil using thermochemical liquefaction technologies.
- Goal: Utilize bio-oil within a petroleum refinery to leverage its existing capital for further processing to final fuels.

## Project goals/objectives support:

- The solvent/hydrothermal liquefaction pathway.
- BETO's objectives to accelerate the production of transportation fuels from high impact biomass.
- Industry's capacity and need to meet the renewable fuel standard (RFS) volume requirements.

# Project Quad Chart Overview

## Timeline

- Project start date: Jan. 1, 2013
- Project end date: Dec. 31, 2013
- Percent complete: 20%

## Budget

Total project funding: \$924,641  
- DOE/BETO :\$654,329  
- Southern Research :\$260,312  
Funding in FY 2011: None  
Funding in FY 2012: None  
Funding for FY 2013: \$924,641  
ARRA Funding: None  
12 month project

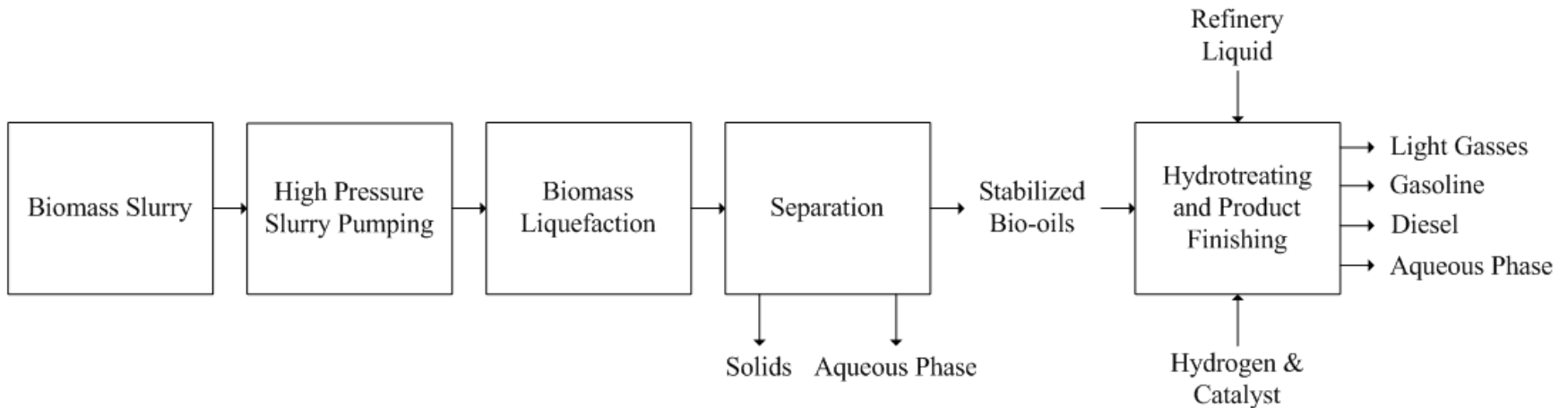
## Barriers

- Barriers addressed
  - High yield of stabilized bio-oil
  - Reduction in oxygen content

## Partners & Roles

Southern Research – Project Management  
& Laboratory-Scale Process  
Development  
Princeton University – TEA/LCA Support  
Refinery Partner (TBD) – Evaluation of  
Bio-oils & Specification of Upgrading  
Requirements

# Project Overview



## Unit Operation Being Improved

- Biomass liquefaction
- Bio-oil separation

## Barrier Addressed

- Bio-oil stability
- Low oxygen content in bio-oil

# 1 – Approach

Activity / Milestones	Approach and Outputs
Laboratory Scale Liquefaction and Upgrading Studies : demonstrate high yield of stabilized refinery-ready bio-oils	Install batch and continuous reactors, calibrate & test analytical equipment. Run planned tests and determine yield, mass balance, and characterize bio-oils and aqueous phases
Technical and Economic Analysis & Life Cycle Assessment (LCA); demonstrate greater than 80 % GHG reduction	Preliminary technical and economic analysis (TEA) and life cycle assessment (LCA). Continuous upgrading of the TEA and LCA as data is generated during the project.
Establish Refinery Partnership	Meet with and present data to several oil companies to identify and select a refinery partner with interest in co-developing and commercializing Southern Research's process technology. Define bio-oil quality specifications that meet requirements for direct insertion at various points in the petroleum refining process. Submit samples to partner for evaluation.
Project Management and Reporting	Project management, required progress and financial reports, meetings, final report

## 2 - Technical Accomplishments/ Progress/Results

- High pressure batch reactor was commissioned (600 ml Parr reactor)
- 23 statistically designed experiments (Minitab 1/4 factorial) were completed with white pine wood.
- Micro-GC, TAN auto-titrator, HPLC (RI, Fluorescence detectors), GC-MS, GC-FID, TGA, CHN, Viscometer, and Karl-Fischer titrator equipment have been setup with analytical methods developed for bio-oil and aqueous phase analysis.
- Design/engineering/procurement of continuous liquefaction and hydrotreating reactors is 35 % complete
- Cloud point/pour point tester, distillation column, flash point tester, and direct oxygen analyzer have been ordered.
- Catalyst partners have been established for supply of suitable catalysts for bio-oil upgrading tests.
- Potential refinery partners have been short listed and discussions initiated
- Preliminary mass and energy balance of process has been developed.
- TEA/LCA has been initiated with Princeton University.

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

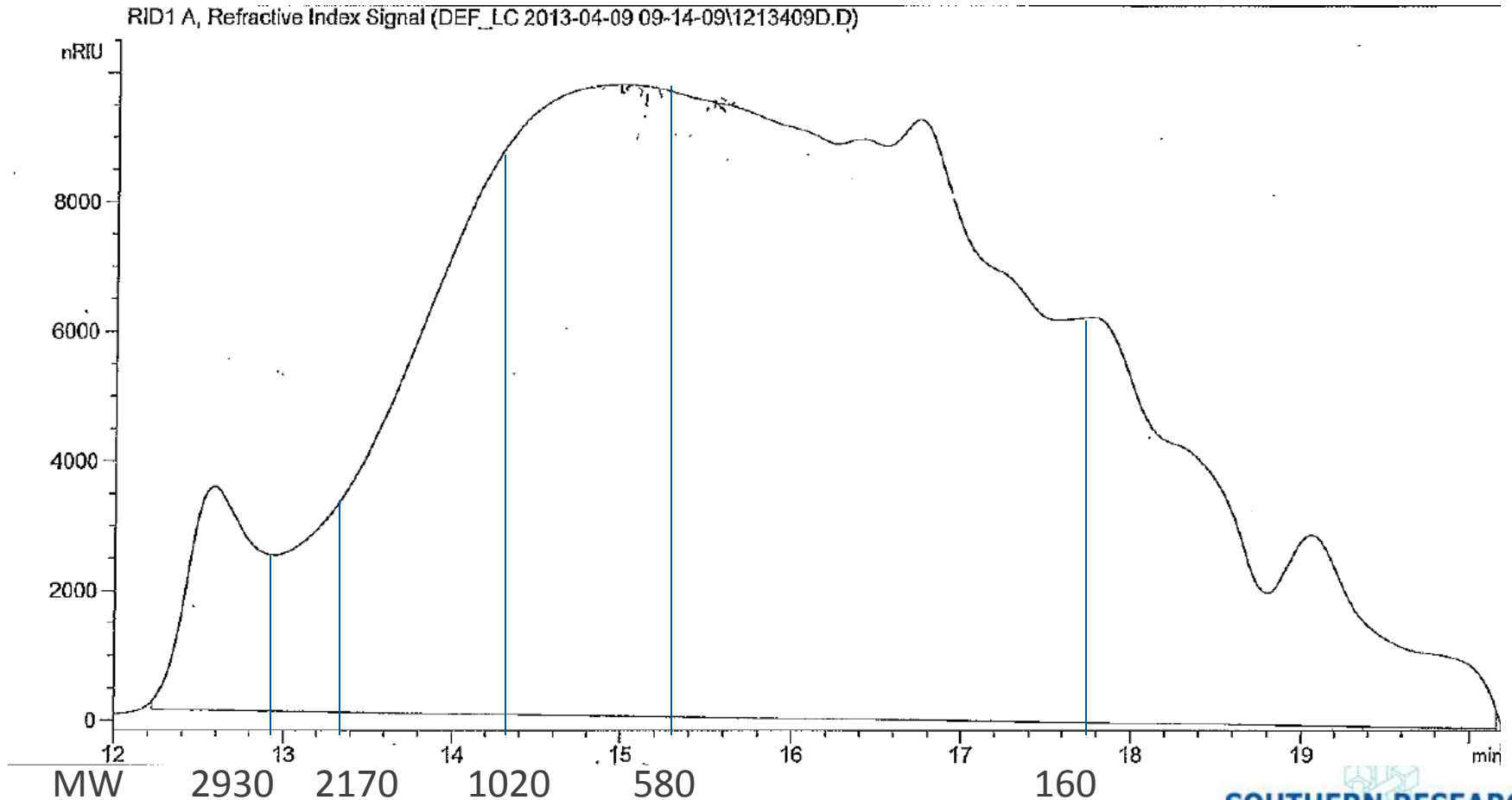
- Reaction conditions and separation techniques withheld pending patent status/IP clearance.
- Results of statistically designed experiments have identified potential optimum conditions.
- Batch reactor results demonstrate high impact of reaction conditions, for example, MAF biomass conversion ranges from 45-98%.
- Results at near optimum reaction/separation conditions:
  - MAF biomass conversion: 94-98%
  - Bio-oil yield: 53-60%
  - Oxygen content (by difference): 25-27%
  - Moisture content <1%
  - TAN <0.8 mg KOH/g

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

- Bio-oil average molecular weight is a strong function of reaction conditions.
- At near optimum conditions, molecular weights range from
  - RI detector: 160-3000 g/mol,
    - >55% in the 370-1000 range
  - Fluorescence detector: 160-1600 g/mol
    - >55% in the 370-580 range.
- Gas produced is less than 6% by weight of input MAF biomass and >90% of gas is CO<sub>2</sub>
- Aqueous phase characterization is currently being performed (pH; extraction followed by capillary GC/GC-MS).

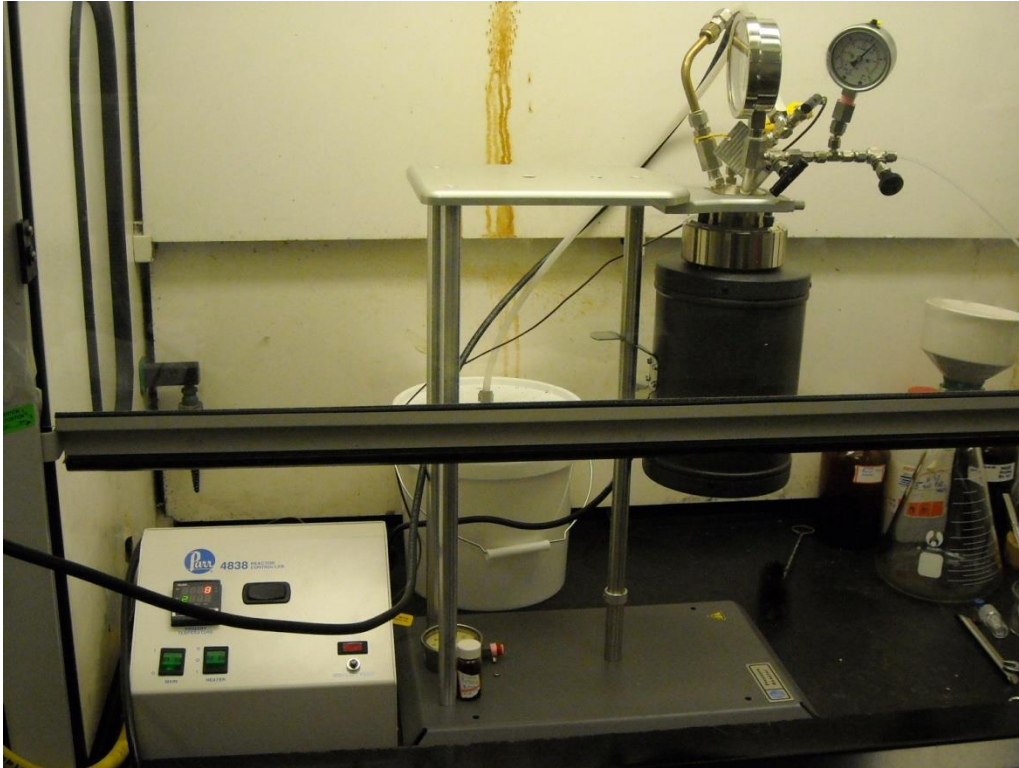


## 2 - Technical Accomplishments/ Progress/Results (cont'd)

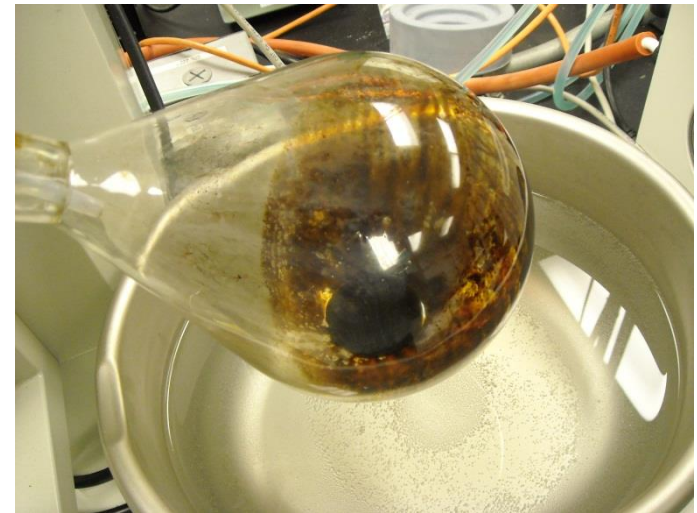


Molecular weight distribution of bio-oil sample

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



Batch Parr reactor setup



Bio-oil produced

# 3 - Relevance

Project addresses several conversion goals, cross-cutting goals, and demonstration & deployment goals in Biomass MYPP

- **Conversion Goals:**
  - Lower cost process
    - High conversion and yield
    - Mild conditions and relatively simple technology = lower capital cost
    - Stabilized and higher quality bio-oil reduces upgrading costs
  - Optimizes reactor performance
  - High performance separation technology
    - Novel bio-oil extraction methods considered in analytical techniques
- **Demonstration & Deployment Goals:**
  - Testing of bio-oil/refinery oil blend -- supports advanced biofuels compatibility testing
- **Cross-cutting Goals:**
  - Strategic Analysis
    - Defines and validates technology performance targets
  - Strategic Communications
    - Presentations to stakeholders and refinery partners
    - Present papers at reputable thermochemical biomass conferences and national meetings

## 3 – Relevance (cont'd)

- Relevance to bio-energy industry
  - Laboratory scale data and TEA will generate basis for a pilot scale reactor design.
  - Refinery partnership will foster relationships that will enable commercialization.
  - Deployment of technology will be rapid due to simplicity of design.
  - Enable industry to meet RFS2 goals and comply with EISA 2007.

# 4 - Critical Success Factors

- Success Factors:
  - Production of stabilized bio-oil with >55% yield.
  - Demonstration of bio-oil compatibility with refinery upgrading operations.
  - Greater than 80% greenhouse gas (GHG) reduction.
- Challenges:
  - Validate batch reactor performance in continuous reactor.
  - Establish refinery partnership.
  - Development of effectively integrated commercial embodiment to minimize GHG emissions.
- Successful project will:
  - Advance the state of technology by generating stabilized bio-oils with high yields.
  - Enable rapid commercialization of bio-fuels with the refinery partner.

# 5. Future Work

Task No. and Description	CY 2013														
	J	F	M	A	M	J	J	A	S	O	N	D			
A. Batch Reactor Studies	a			b											
B. Continuous Reactor Studies															
C. Techno-economic Analysis and Life Cycle assessment												c			
D. Refinery Collaboration	d														
E. Project Management and Reporting	e										f				

## Legend

- a Project planning meeting
- b Technical Decision Point
- c LCA verification of >80 % GHG reduction
- d Establish at least one refinery partner
- e Present paper at DOE biomass conference
- f Final report

# Summary

- Mild liquefaction process being developed to produce stabilized refinery-ready bio-oils
- 23 statistically designed batch reactor experiments completed. Conditions significantly affect bio-oil yield
- Biomass conversion up to 98 % and bio-oil yield up to 60 % of input biomass was achieved at optimum conditions
- Total acid number (TAN)  $<0.8$  and water  $<1\%$  was achieved in the bio-oil. GC and HPLC shows that bio-oils produced are stable.
- Stability is further being confirmed by viscosity measurements
- Preliminary mass and energy balance of a commercial embodiment of the process was completed
- Significant future work will include:
  - Construction/commissioning of continuous liquefaction and hydrotreating/hydrocracking reactors
  - Development of a partnership agreement with a refinery and verification of suitability of the bio-oils for refinery insertion