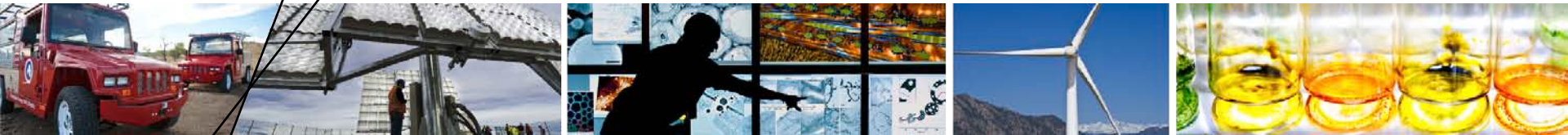


# **DOE Bioenergy Technologies Office (BETO) 2015 Project Peer Review**

## **Separations Development and Application**



**March 25, 2015**

**Technology Area Review: Biochemical Platform**

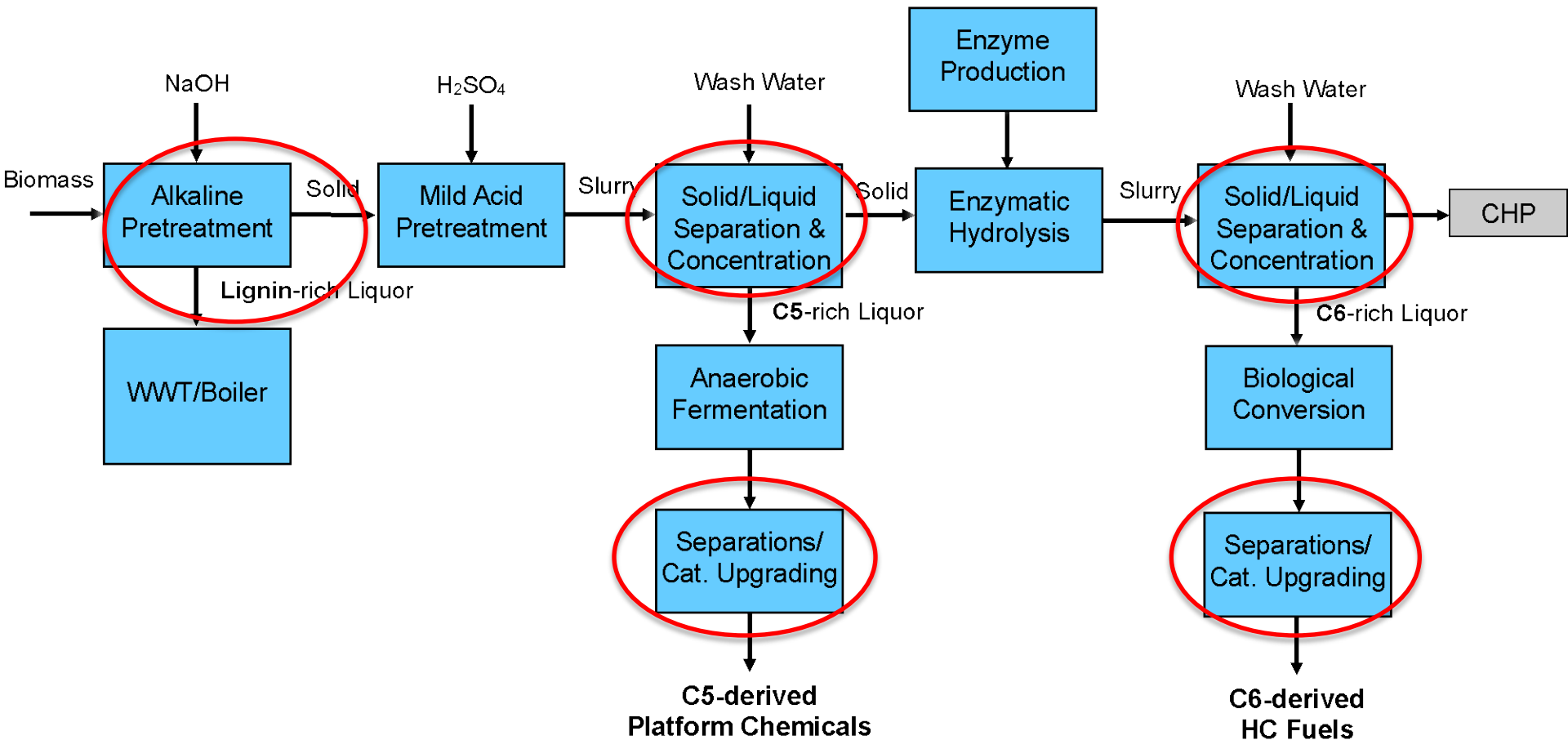
**Principal Investigator: James D. McMillan**

**Organization: NREL**

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

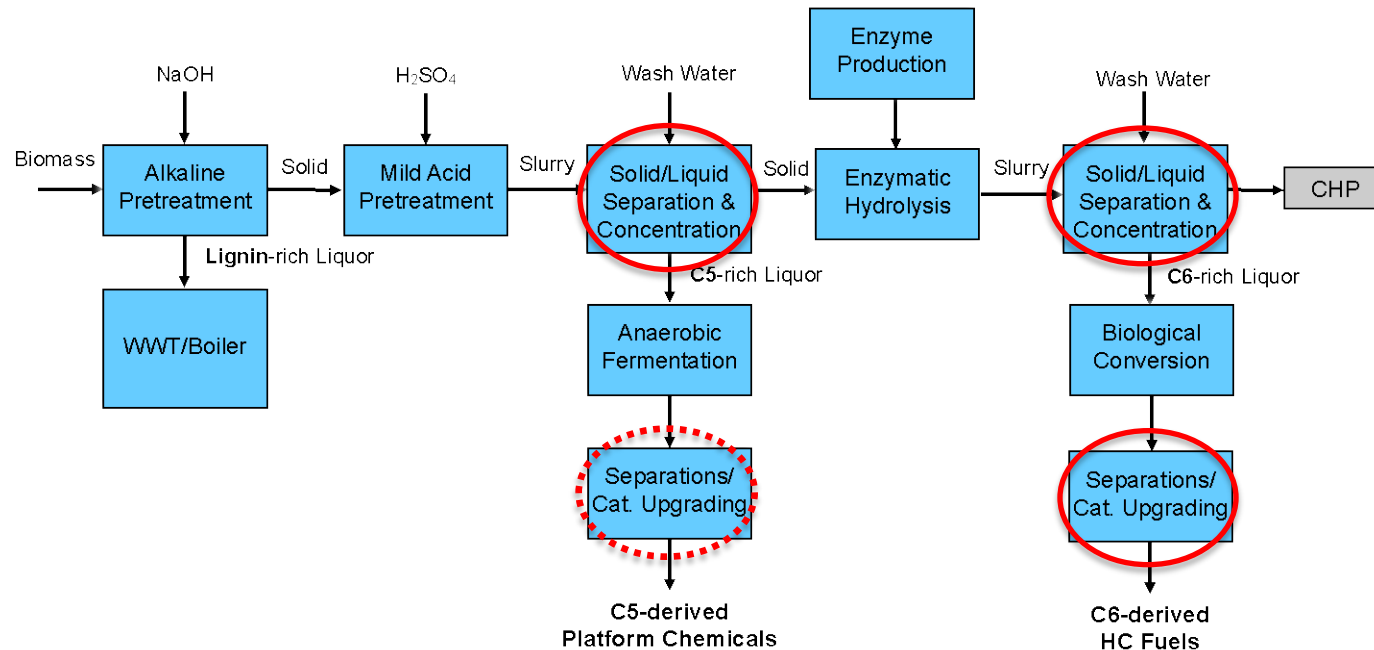
# Overall Project Goal

Overall goal: Define, develop and apply separation processes to enable demonstration of sugars to SA and HC fuel precursor



Envisioned biomass conversion process to be demonstrated in 2017.

# SDA Project Overview



- **Support achieving 2017 and 2022 advanced biofuels production cost goal of \$3/GGE (\$2011)** by developing and applying separations processes to improve the efficiency and economics of producing biofuels and value-added co-products from biomass.
- **Major foci:** 1) upstream liquor clarification and concentration to prepare C5-rich and C6-rich sugar streams for biological upgrading; and 2) downstream recovery of the lipid product (fuel precursor) produced from the C6-rich sugar stream. Secondary foci are slurry characterization and continuous enzymatic hydrolysis.
- Co-product (SA) recovery removed from scope in FY15 AOP Merit Review.

# Starting Point and Major Project Elements

**Starting point:** Saccharification technology from NREL's 2012 integrated demonstration of cellulosic ethanol

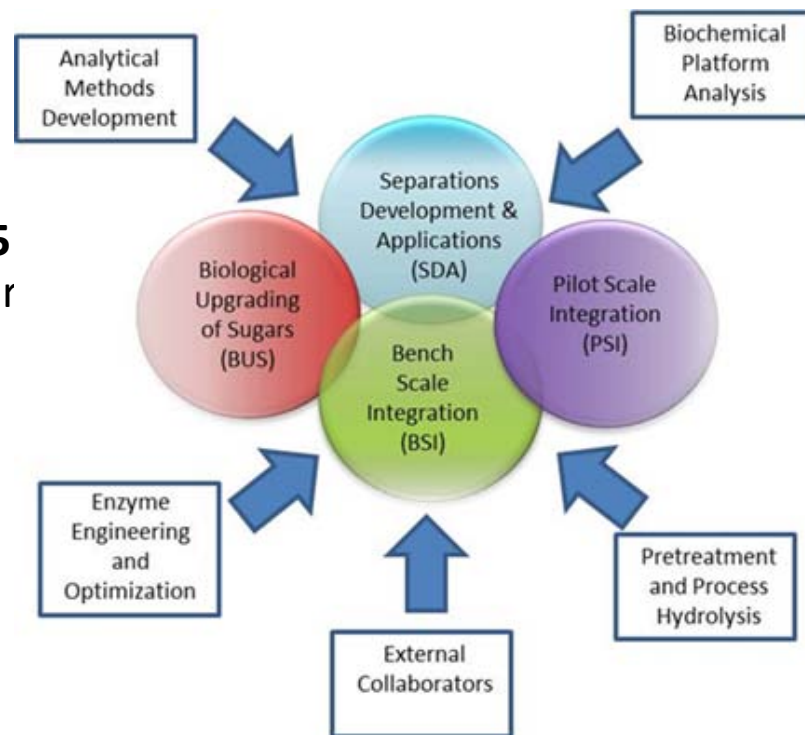
- No solid-liquid, concentrative or suitable product recovery separations developed in this work; it was a whole slurry process with product recovered by distillation.

## Project elements and timing

**(FY13 start)** as Slurry Characterization and Separation Science task w/in PPH task

- **Renamed Sugar Stream Production task in FY15** with main focus on separations to produce sugar streams for 2017 demo
  - C5-rich stream for SA coproduct production
  - C6-rich stream for HC precursor production
- **(FY15 start)** Fuel Precursor Recovery
  - Develop lower cost lipid recovery technology (superior to homogenizer-based methods)

➔ **R&D plan coordinated with and interdependent on NREL's BUS, BSI and PSI projects**



# Quad Chart Overview

## Timeline

**Start: FY13** (as PPH – Integration)

- Slurry characterization → sugar stream production related separations

**End: FY17** (projected)

**Percent complete: 25%**

## Budget

	Total Costs FY10 – FY12	FY13 Costs	FY14 Costs	Total Planned Funding (FY15-Project End Date)
DOE Funded	\$0	\$449,000 (slurry characteriz- ation and separations science task)	\$428,281 (out of \$1,435,942)	\$3,023,996
Project Cost Share (Comp.)*	--	--	--	--

## Barriers

Primary focus on addressing upstream and downstream separations-related barriers:

- **Bt-H. Cleanup/Separation**
- **Bt-J. BC Process Integration**

## Partners

- Subcontractors
  - CSM (biomass slurry rheology)
  - U. Colorado, U. Arkansas (Continuous EH and membrane-based concentration)
  - Pall Corporation (Cross flow filtration)
  - Other(s) (Not yet identified)
- National Laboratories
  - LBNL (ABPDU) – homogenizer data
  - ANL – EDI organic acid recovery data
- Related NREL projects
  - Biochemical Platform Analysis (BPA)
  - Biological Sugars Utilization (BUS)
  - Bench Scale Integration (BSI)
  - Pilot Scale Integration (PSI)

# Approach (Technical)

**Overall approach is cost-driven R&D:** TEA assessment guides research priorities; statistical design of experiments data informs ongoing TEA refinement.

## Where prior relevant data exists

Use to develop initial performance and cost sensitivity information (jointly with BC Platform Analysis) for key separation processes, e.g., for filtration to remove solids from post pretreatment and post enzymatic hydrolysis slurries.

## Where prior data unavailable

Identify/review best available public domain literature (papers and patents), and use this information to inform preliminary techno-economic analyses to guide R&D prioritization.

	Design Report Basis	2014 SOT	2015 Projection	2016 Projection	New 2017 Target
<b>Minimum Fuel Selling Price (\$/GGE, 2011\$)</b>	<b>\$5.10</b>	<b>\$12.97</b>	<b>\$10.14</b>	<b>\$7.43</b>	<b>\$5.03</b>
Feedstock Contribution (\$/GGE, 2011\$)	\$1.76	\$3.88 <sup>1</sup>	\$3.20 <sup>1</sup>	\$2.47 <sup>1</sup>	\$1.87 <sup>1</sup>
<b>Conversion Contribution (\$/GGE, 2011\$)</b>	<b>\$3.33</b>	<b>\$9.09<sup>1</sup></b>	<b>\$6.93<sup>1</sup></b>	<b>\$4.97<sup>1</sup></b>	<b>\$3.16<sup>1</sup></b>
RDB Fuel Yield (GGE/dry ton)	45	18	20	20	22
Succinic Acid Yield (lb/dry ton)	NA	197	206	232	270
<b>Feedstock</b>					
Feedstock Cost (\$/dry ton) <sup>2</sup>	\$80	\$130	\$115	\$95	\$80
Feedstock Blend	Blend	Stover	Stover	Blend	Blend
<b>Pretreatment/Separation</b>					
Solids Loading (wt%)	30%	30%	30%	30%	30%
Xylan to Xylose (including conversion in C5 train)	>73%	73%	75%	78%	78%
Hydrolysate solid-liquid separation	No	Yes	Yes	Yes	Yes
Xylose Sugar Loss (into C6 stream after acid PT separation)	NA	5%	4%	2.5%	1%
<b>Enzymes</b>					
Enzyme Loading (mg/g cellulose)	10	14	12	10	10
<b>Enzymatic Hydrolysis &amp; Bioconversion – C6 Train</b>					
Total Solids Loading to Hydrolysis (wt%)	20%	15%	15%	17.5%	17.5%
Enzymatic Hydrolysis Time (d)	3.5	3.5	3.5	3.5	3.5
Hydrolysis Glucan to Glucose	90%	77%	85%	85%	90%
Hydrolysis Residual Xylan to Xylose	>30%	30%	30%	30%	30%
Glucose Sugar Loss (into solid lignin stream after EH separation)	1%	5%	4%	2.5%	1%
<b>Expt'l bioconversion scale/method</b>					
Bioconversion Volumetric Productivity (g/L-hr)	1.3	0.29	0.3	0.35	0.4
Lipid Content (wt%)	NA	57%	57%	60%	60%
Glucose to Product (total glucose utilization) <sup>3</sup>	87% [95%]	75% [100%]	75% [100%]	78% [100%]	78% [100%]
Xylose to Product (total xylose utilization) <sup>4</sup>	82% [86%]	74% [98%]	74% [98%]	76% [98%]	76% [98%]
C6 Train Bioconversion Metabolic Yield (Process Yield) (g/g sugars)	0.34 (0.28)	0.26 (0.26)	0.26 (0.26)	0.27 (0.27)	0.27 (0.27)
Intermediate Product Recovery	97%	90%	90%	90%	90%
Carbon Yield to RDB from Biomass	26.2%	10.4%	11.4%	11.8%	12.5%
<b>Coproduct Production Performance – C5 Train</b>					
Bioconversion Volumetric Productivity (g/L-hr)	NA	0.3	1	1.5	2.0
C5 Train Bioconversion Metabolic Yield (Process Yield) (g/g sugars)	NA	0.63 (0.59)	0.64 (0.60)	0.66 (0.65)	0.795 (0.74)
Succinic Acid Recovery Efficiency	NA	80%	80%	80%	80%
Carbon Yield to Succinic Acid from Biomass	NA	8.9%	9.3%	10.5%	12.2%

**Metrics: Recovery yield** is the most important performance measure for bulk products (fuels or chemicals), followed by CAPEX and OPEX; see **Target Table**.



# 2017 BC Demo Targets Table (FY14 SOT)

	<i>Design Report Basis</i>	<b>2014 SOT</b>	<b>2015 Projection</b>	<b>2016 Projection</b>	<b>New 2017 Target</b>
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C5 Stream  
Production

C6 Stream  
Production

Lipid  
Recovery

SA  
Recovery

# Approach (Management)

## Critical Success Factors:

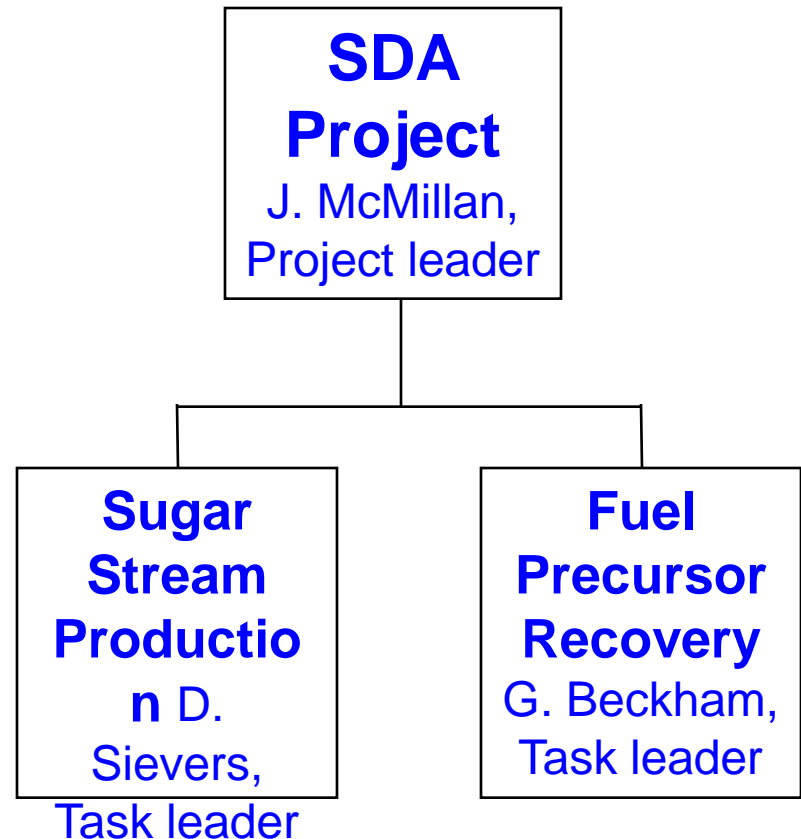
Demonstrate separation process recovery yields  $\geq$  design case targets and at capital and operating costs  $\leq$  design case (i.e., technically and commercially near viable).

## Top 2-3 Challenges:

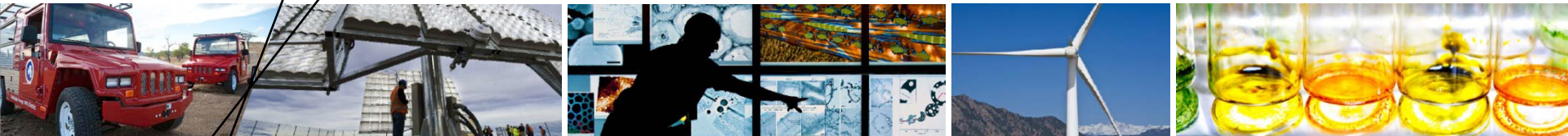
- 1) Difficulty optimizing separations for processing steps still being refined, i.e., both pretreatment and lipid production;
- 2) Ability to adequately assess at lab scale methods to be applied at pilot/larger scale;
- 3) Access to small scale (“table top”) systems for testing/optimizing S/L separations, cell disruption, etc.

**Project management** occurs within NREL’s Biomass Program. Follows AOPs format, with established progress milestones and go/no-go decision points.

## FY15 Restructure







# Technical Accomplishments

# FY13 Accomplishments / Results

## Slurry Characterization and Separation Science subtask

- Advanced methods to measure slurry rheological properties
- Began developing and testing methods for fractionating and dewatering biomass slurries into component process streams

## FY13 Milestone Table:

Type	Title/Performance Measure	Date
D	Develop membrane-based water removal (sugar concentrating) process technology. <i>Performance Measure:</i> Demonstrate a 4-fold increase in solute concentration with a flux decline of less than 50%.	2/28/13
E	Develop experimental and data analysis methods for routinely measuring the rheological properties of biomass slurries by means of large amplitude shear rheometry. <i>Performance Measure:</i> Document methods in 1-2 manuscripts and submit to a peer-reviewed journal.	6/30/13

?

➔ **Both milestones successfully completed on time, within budget. Results documented in milestone reports and multiple publications.**

# FY13 Accomplishments / Results (2)

- **Objective:**

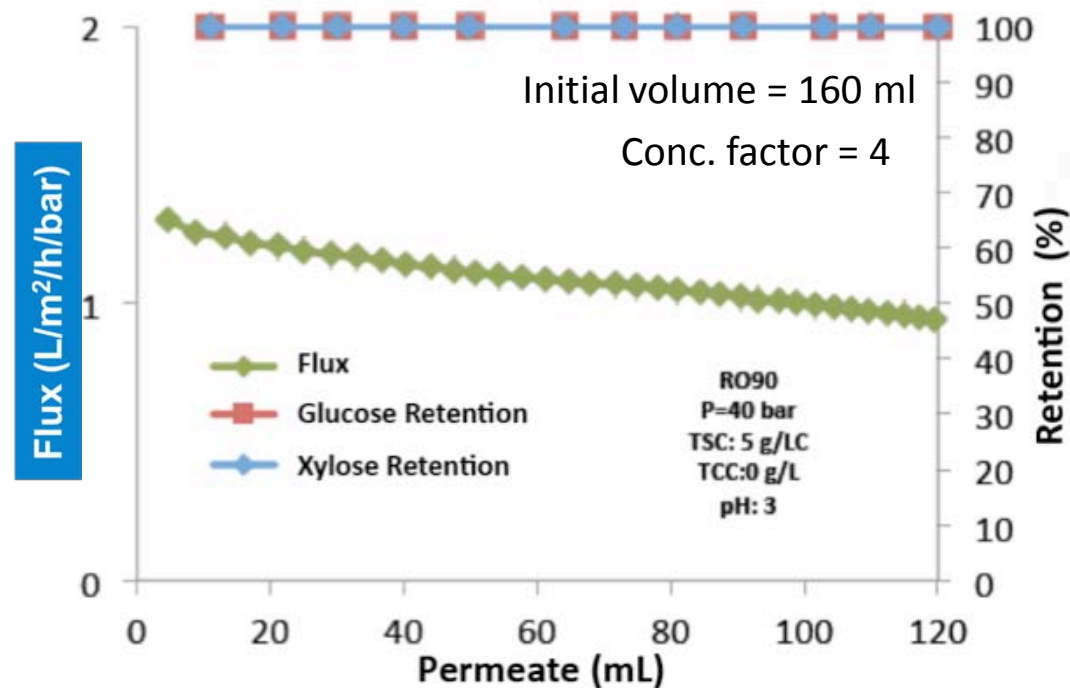
Develop membrane-based water removal (sugar concentrating) process technology.

- **Approach:**

Conducted dead-end filtration experiments using model and PCS hydrolysates, testing multiple nanofiltration membranes from two manufacturers. (U. Arkansas s/c)

- **Outcomes:**

- 1) Verified nanofiltration effective to concentrate hydrolysate sugar streams
- 2) Found membrane selection and operating pH can be adjusted to maximize loss of toxic species (i.e., acetic, furfural, HMF).
- 3) Identified a potential method to enable/achieve continuous enzymatic hydrolysis.

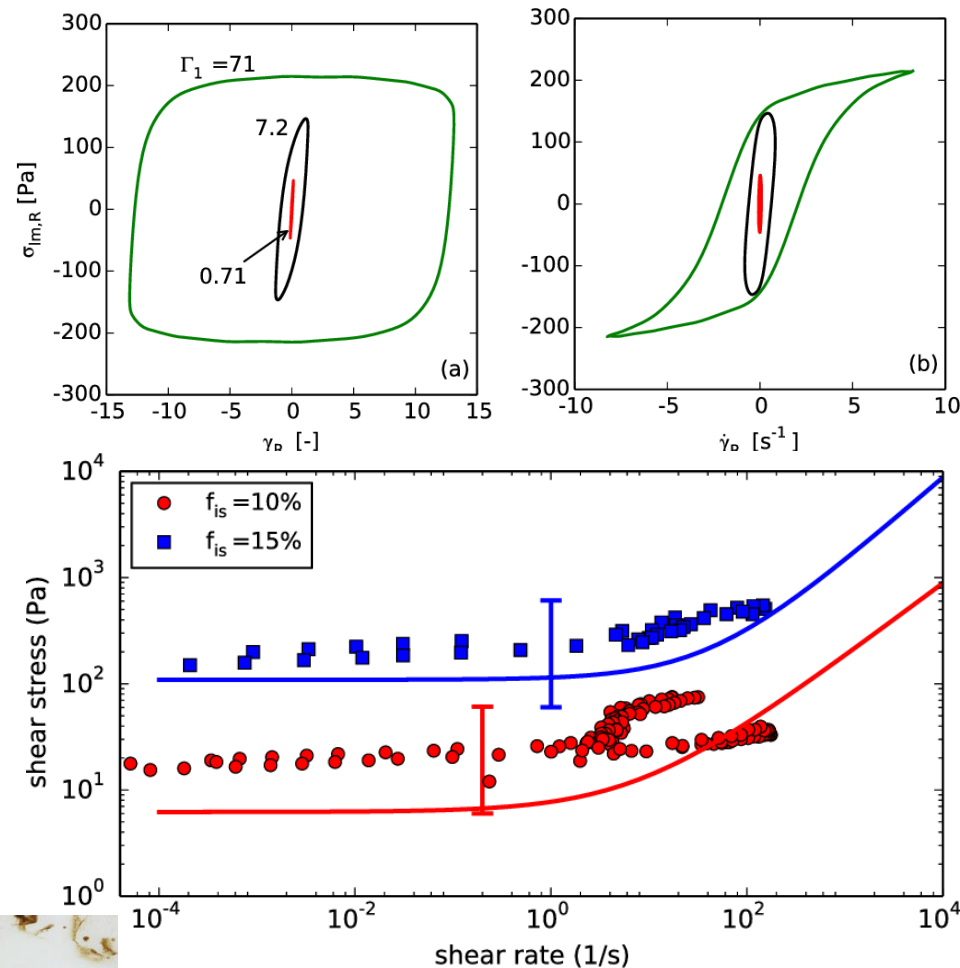


- **Significance:** While TEA remains to be performed, results show promise for NF as a lower energy method for concentrating biomass hydrolysates.

- **Publication:** Malmali *et al.* 2014. *Sep. Purif. Technol.*, **132**:655-665.

# FY13 Accomplishments / Results (3)

- **Objective:** Develop methods for routinely measuring high solids biomass slurry rheological properties using large amplitude oscillatory shear (LAOS) rheometry.
- **Approach:** Conducted experiments and data analysis modeling to enable LOAS rheometry of biomass slurries.
- **Outcome/Significance:** Advanced and validated the use of LAOS rheometry for determination biomass slurry viscosity information. This is enabling more routine and reliable characterization of slurry rheology.
- **Publications:**
  1. Stickel *et al.* 2013. J. Rheol., **57**(6):1569-1596.
  2. Stickel *et al.* 2014. Appl. Rheol., **24**(5):53075.



# FY14 Accomplishments / Results

## Slurry Characterization and Separation Science subtask

- Extend studies of slurry rheological properties and methods for fractionating and dewatering biomass slurries into component process streams.
- Develop continuous enzymatic hydrolysis (CEH) process with enzyme retention
  - Simultaneous/integrated EH and S/L filtration to produce clean sugar stream
  - Potential for higher productivity and lower costs than a batch system
  - Projected to enable use of smaller equipment (↓ CAPEX), reduced enzyme loadings (↓ OPEX) through decreased product inhibition and enzyme retention/recycle if operability can be demonstrated.

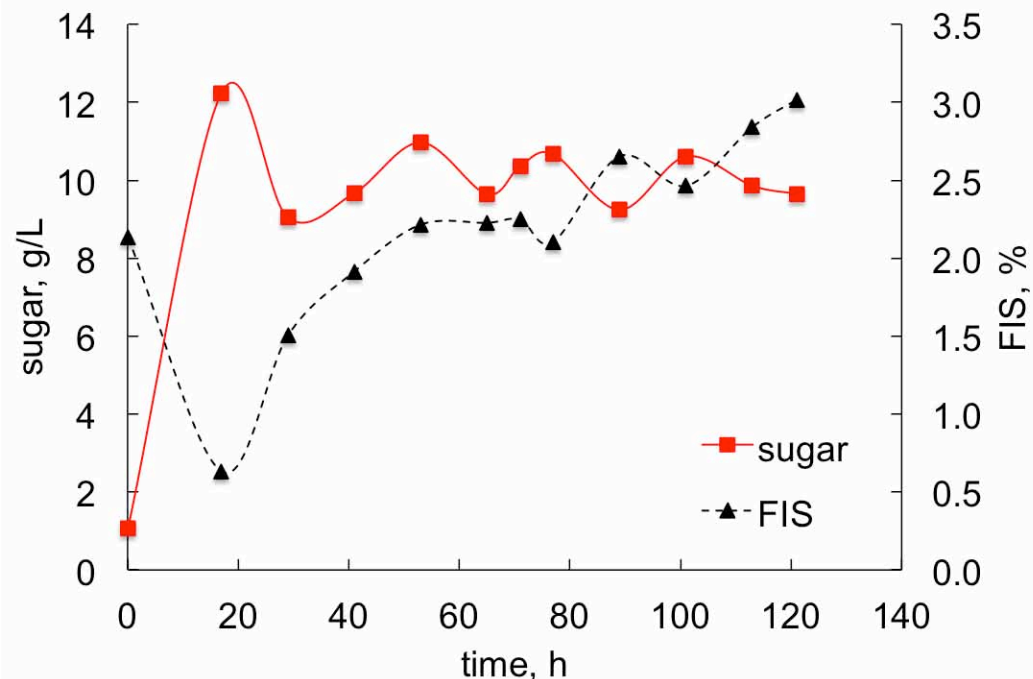
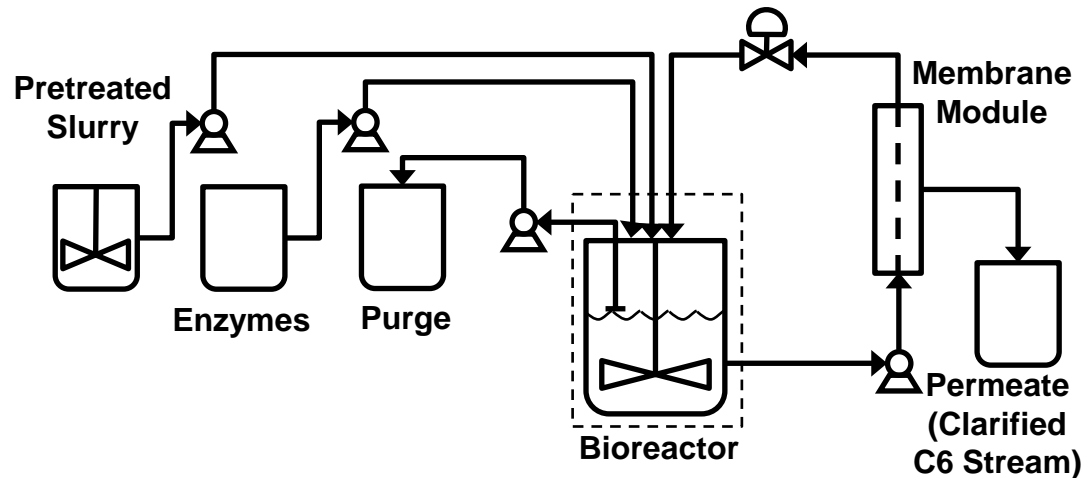
## FY14 Milestone Table:

Qtr	Due Date	Type	Milestones, Deliverables, or Go/No-Go Decision
Q4	9/30/2014	Regular	Milestone: Demonstrate successful steady state operation of a 5L continuous enzymatic hydrolysis reactor for more than 72 hours.

➔ *Milestone successfully achieved on schedule and within budget. Results documented in milestone report; publications pending.*

# FY14 Accomplishments / Results (2)

- **Objective:** Demonstrate ability to operate CEH bioreactor for  $\geq 72$  h
- **Approach:** Use nanofiltration (NF) to remove glucose while retaining enzyme-bound solids. System comprises a 5-L STR bioreactor integrated with a NF PVDF HF membrane module. (w/ CU Boulder)
- **Outcome:** Achieved pseudo steady state CEH operation on pretreated corn stover (PCS) slurry for more than 72 h; purge used to manage residual solids buildup.
- **Significance:** Demonstrated CEH proof-of-concept. Achieved 2.5 higher productivity (0.93 g/L-h) than in batch, and conversion yield of 64.5%, close to theo. max for 1 CSTR (67%). CSTRs in series will enable higher conversion yields.
- **Presentation:**  
[Adhikari et al. 2014. 36th SBFC.](#); more pres. and pubs pending

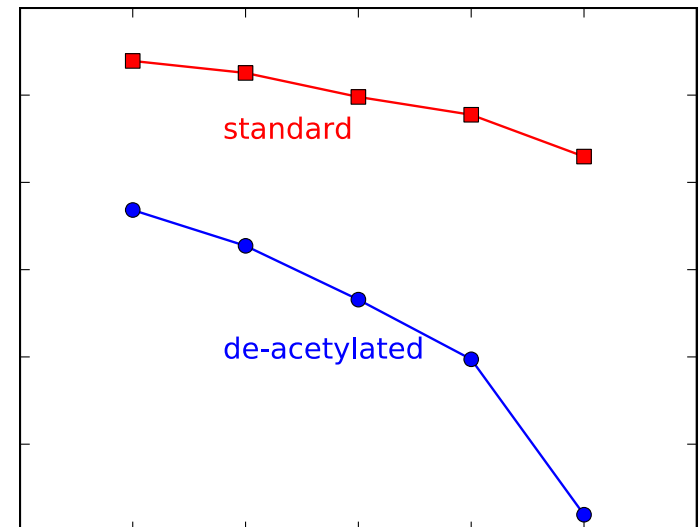
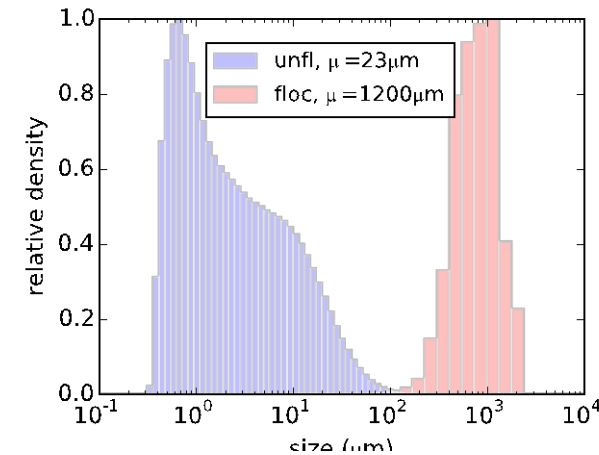




# FY14 Accomplishments / Results (3)

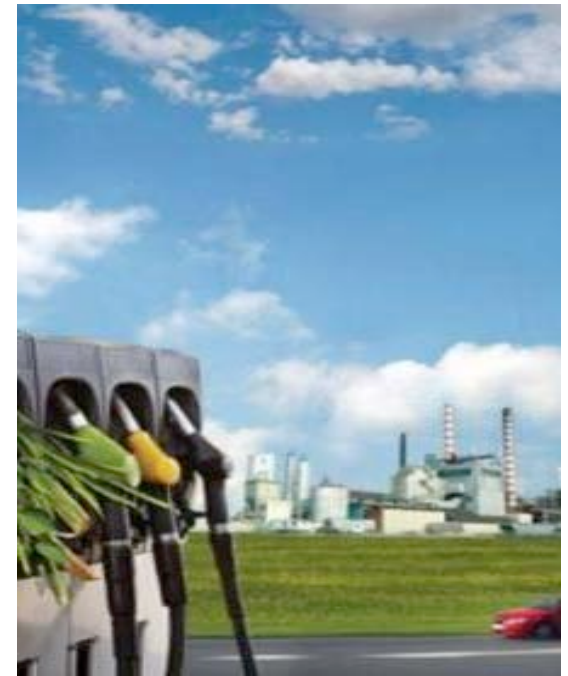
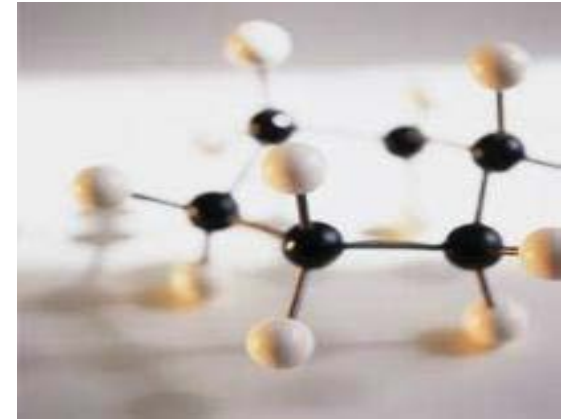
- **Objective:** Develop process to remove residual solids from post (EH) slurry.
- **Approach:** Improve slurry filterability by using: 1) polyelectrolyte flocculant; or 2) high scouring velocity cross flow filtration (with Pall). Test 2 different types of hydrolysates at bench scale (dilute acid (DA) and deacetylation + DA).
- **Outcome:** Cross flow pending. Polyelectrolyte flocculant increased mean particle size 50-fold ( $23\mu \rightarrow 1200\mu$ ) and greatly improved filterability: scaled filter capacity increased 40-fold.
- **Significance:** Potential for large cost savings. At 95% sugar recovery, MFSP decreased by \$1.35/GGE for DA pretreated and EHed hydrolysate (by \$3.40/GGE for DA+deacetylation and EH'ed materials).
- **Publication (submitted):**

[Sievers \*et al.\* \(2015\). A low-cost solid-liquid separation process for enzymatically hydrolyzed corn stover slurries. Bioresource Technol. Submitted \(January\).](#)



# Relevance

- **This project helps achieve MYPP strategic and performance goals** to convert biomass sugars (and other carbohydrate and lignin derivatives) to hydrocarbon fuels, as described in recent reports:
  - *Davis, R. et al. (2013). Process Design and Economics for the Conversion of Lignocellulosic Biomass to Hydrocarbons: Dilute-Acid and Enzymatic Deconstruction of Biomass to Sugars and Biological Conversion of Sugars to Hydrocarbons. NREL/TP-5100-60223.*
  - *Biddy and Jones. (2013). Catalytic Upgrading of Sugars to Hydrocarbons Technology Pathway. NREL/TP-5100-58055.*
- **Separations R&D supports 2017 and 2022 goals** to develop sugars to biofuels technologies at a production cost  $\leq$  \$3/GGE. Separations processes in aggregate often represent  $\geq$  50% of total production cost and can make or break techno-economic viability.
- **Reliable, cost effective separation processes are required** to be able to conduct 2017 demo.
- **Some market / commercialization barriers are indirectly addressed** through partnering, technology de-risking and dissemination of findings.



# Future Work

## High-level Gantt chart:

Project/Activity	Date	FY15				FY16				FY17			
		FY15Q1	FY15Q2	FY15Q3	FY15Q4	FY16Q1	FY16Q2	FY16Q3	FY16Q4	FY17Q1	FY17Q2	FY17Q3	FY17Q4
<b>1. Sugar Stream Production</b>													
<b>C5 Sugar Stream Production</b>													
<b>Clarification (S/L Separation)</b>													
Assess pretreatment severity impact on SLS					◆ M.1.2								
Report on selected S/L method						◆ M.1.3							
<b>Concentration outside scope (FY15)</b>													
Membrane-based (RO) (proposed)										◆ M.1.5			
Evaporator-based (proposed)										◆ M.1.5			
<b>C6 Sugar Stream Production</b>													
<b>Clarification (S/L Separation)</b>													
Flocculation			◆ M.1.1										
Cross Flow Filtration (pending w/ Pall)													
Report on selected S/L method						◆ M.1.3							
<b>Concentration</b>													
Membrane-based (RO)								◆ M.1.4					
Evaporator-based								◆ M.1.4					
<b>2. Lipid Recovery</b>													
Literature and patent review				◆ M2.1									
Demo lysis and lipid phase separation					◆ M.2.2								
Down-select recovery option (>85% yield, scalable, lowest cost)								◆ M.2.3 (Go/No-go)					
Demo continuous recovery (>90% @ >10-L)												◆ M.2.4	

# FY15: Sugar Stream Production - Clarification

**Develop process for removing residual solids**  
from post EH slurry (for C6 stream production)

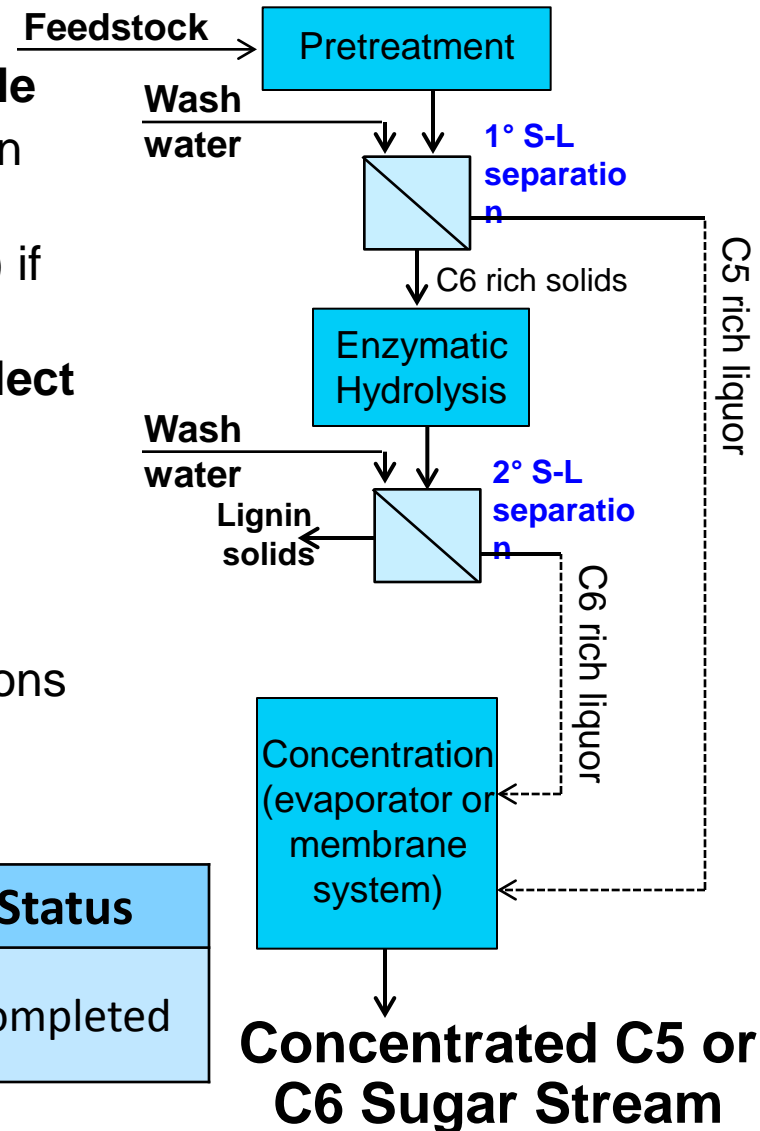
- **Evaluate 2 methods with potential for large scale**

1. Flocculation in combination with vacuum filtration using a small scale vacuum filter apparatus
2. Cross flow filtration with support of Pall (partner) if negotiations work out. (leverages NABC work)

- **Use data in comparative TEA to guide down-select**

**Assess post pretreatment SLS** for different severity PCS samples. Use results to update TEA.

**Assist PSI project in assessing C5 and C6**  
sugar stream production-related pilot scale separations  
equipment needs



Milestones (FY15)	Date	Status
Quantify improvements to post EH slurry SLS using flocculation	12/31/2014	Completed

# FY16: Sugar Stream Concentration

## Select method for concentrating C6 stream (after SLS clarification)

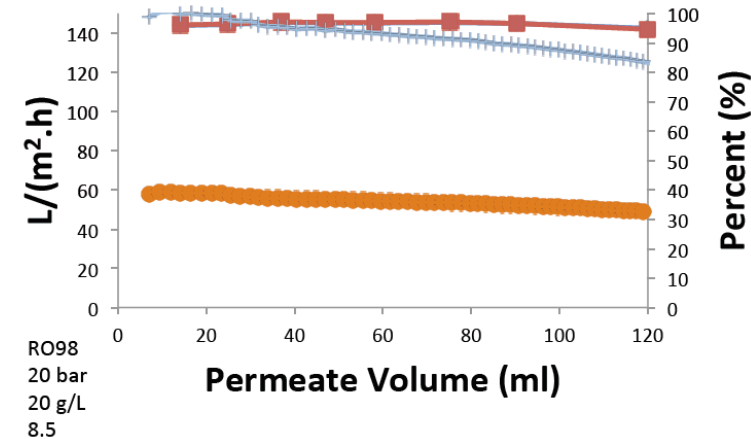
- Assess at bench scale evaporation vs NF-based methods; develop comparative performance data.
- Saw promising initial NF results: increased sugar concentration  $\geq 4X$  while maintaining high water permeate flux ( $\geq 80\%$ )
- Use comparative performance data to update TEA and guide selection of preferred method

## Revisit need to concentrate C5-rich stream

- If needed, leverage results obtained for C6 stream concentration to develop an effective method

## Bench Scale Concentration

### Initial NF results

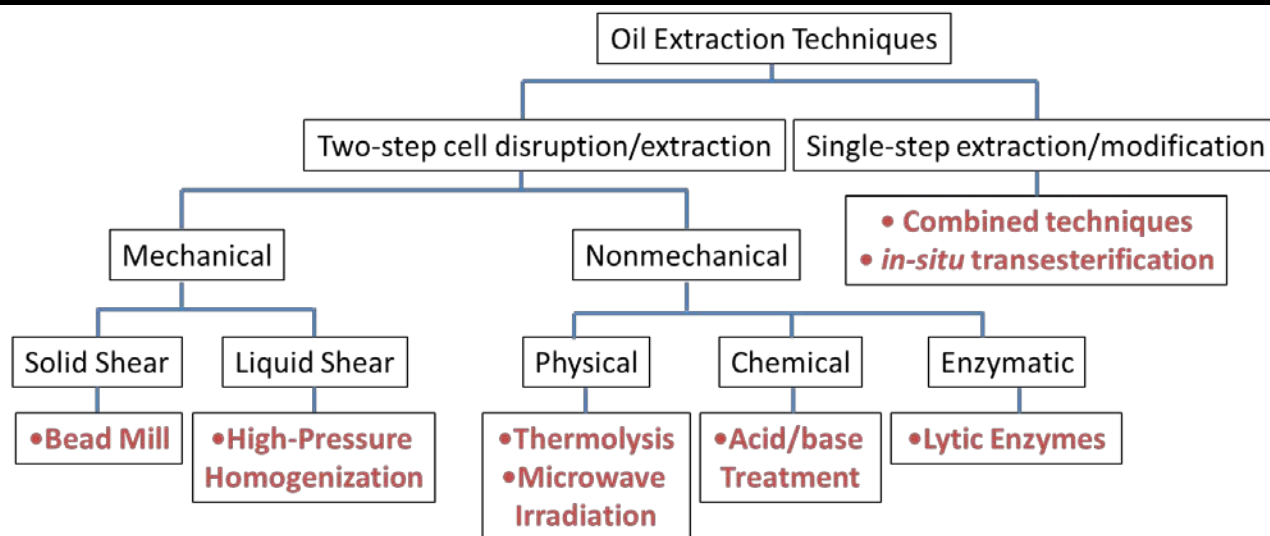


### Rotovap for evaporator concentration testing (example)



Milestone (FY16)	Date	Type
Develop comparative performance data for achieving 4x concentration of clarified C6 stream using 2 different concentration methods. Use TEA to down-select technology choice.	3/31/2016	Regular (Quarterly)

# FY15-16: Lipid Recovery Method Down-Select



## Performance screen $\geq 7$ methods, including:


- High-pressure homogenization, cell wall lytic enzymes, and acid treatment
- Other approaches to be identified/refined during Q1-Q2 literature review

Milestones (FY15-FY16)	Date	Type
Complete literature review and patent search to establish state of art for batch and continuous lipid extraction from yeast/microbes/broths. Recommend R&D.	3/31/2015	Regular (Quarterly)
Demonstrate cell lysis and lipid phase separation from production broth, and assess potential of algal lipid recovery methods (Joint w/ BUS & BSI projects)	9/30/15	Regular (Annual)
Use comparative data and literature to inform TEA and guide down-select (criteria: $\geq 85\%$ recovery, scalable, lowest cost). Rank order top 3 recovery options; further work will focus on top method meeting criteria unless it ends up not proving out.	3/31/16	Go/No-Go

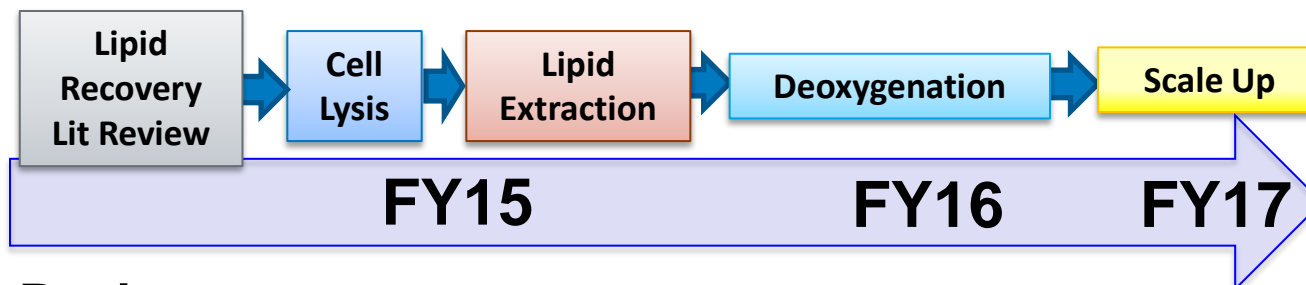


# Review of Major Literature Reviews

Review	Microbes Assessed	Applications	Recommendations for Scale-up	Recommendations against Scale-up
<b>Chisti, 1986</b>	Bacteria, Yeast, Algae, Fungi	Enzymes, proteins	High-pressure homogenization, bead milling, possibly freeze-pressing, enzymatic lysis, and combined techniques (e.g., alkali + HPH)	Sonication, chemical treatments if high selectivity and sensitive proteins are targets
<b>Middelberg, 1995</b>	Bacteria, Yeast	Proteins	High-pressure homogenization, bead milling, microfluidization, thermolysis, base or OCI-, enzymes if cheap, combined techniques (e.g., chelators + enzymes, enzymes + HPH, heat + alkali + HPH)	Possibly decompression, osmotic shock, antibiotics, direct solvent extraction; chaotropes, detergents when used alone
<b>Geciova, 2002</b>	Bacteria, Yeast	Enzymes, proteins for dairy industry	High-pressure homogenization, bead milling, microfluidization, combined techniques	Sonication, decompression, osmotic shock, thermolysis
<b>Cooney, 2009</b>	Algae	Lipids, Fuels	Possibly mechanical approaches (HPH, bead milling) in-situ transesterification, ASE and 'milking' with (do)decane	Sub- or supercritical water, methanol, or CO2 extraction; techniques that require drying or freezing; possibly microwave irradiation, osmotic pressure approaches, and sonication
<b>Halim, 2012</b>	Algae	Lipids, Fuels	High-pressure homogenization, bead milling	Techniques that require drying
<b>Yusaf, 2014</b>	Bacteria, Yeast, Algae, Fungi	Enzymes, proteins for agriculture, wastewater treatment	High-pressure homogenization, bead milling, microfluidization, hydrodynamic cavitation, physical-physical combined techniques	Rotor-stator homogenizers, French press, decompression, ultrasonication without design improvements, possibly physical-chemical combined techniques

 **Effective approaches exist: HPH, bead milling, combined techniques**

# Lipid Recovery Down-Select Approach



## Literature Review

- Mechanical and combined approaches show most promise for scale up
- Leverage NREL expertise in enzymatic, acid pretreatment

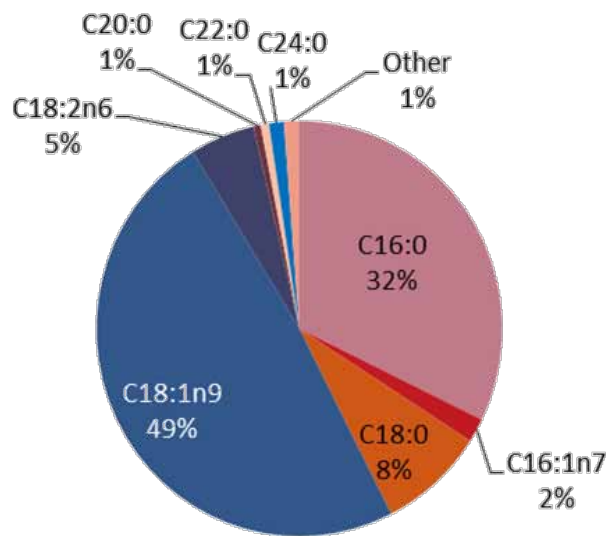
## Design of Experiments Scope:

Technique	Variables	Equipment Located
Lytic enzymes	T, t, [enzyme]	Y
Microwave irradiation	T, t, wt% solids	Y
Acid/base treatment (+m-wave)	T, t, [acid/base]	Y
Bead milling	Packing fraction, rpm, wt% solids	Y
Autoclaving	T, t, wt% solids	Y
High-pressure homogenization (HPH)	P, # ixn ch, # passes	Y
Acid + bead mill	rpm, t, [acid]	Y

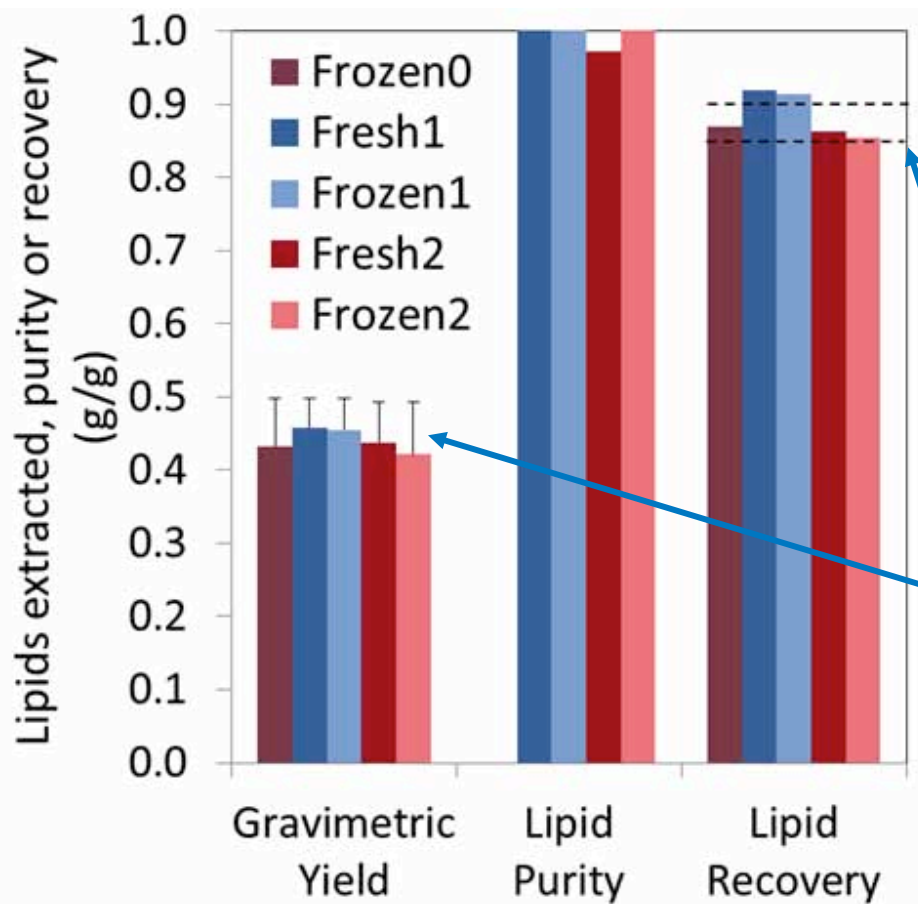
# Initial Results Encouraging

**Current cultivation conditions** produce yeast with ~50% lipids (dry wt. basis)

- Mainly C16-C18 fatty acid chains
- Lipids are recoverable in high purity and yield
- Freezing cells does not appear to increase lipid recovery



**Lipid Composition**



Design Report Basis

FY16 Q2 Go/No-go target

FAME yield of freeze-dried yeast cell mass

# Summary

## Overview/Approach: TEA-guided, cost-driven R&D

- Leverage prior relevant data, if available, to get started
- Mine literature or develop base line data where not
- Data informs TEA, which guides R&D prioritization for expt'l design

## Results:

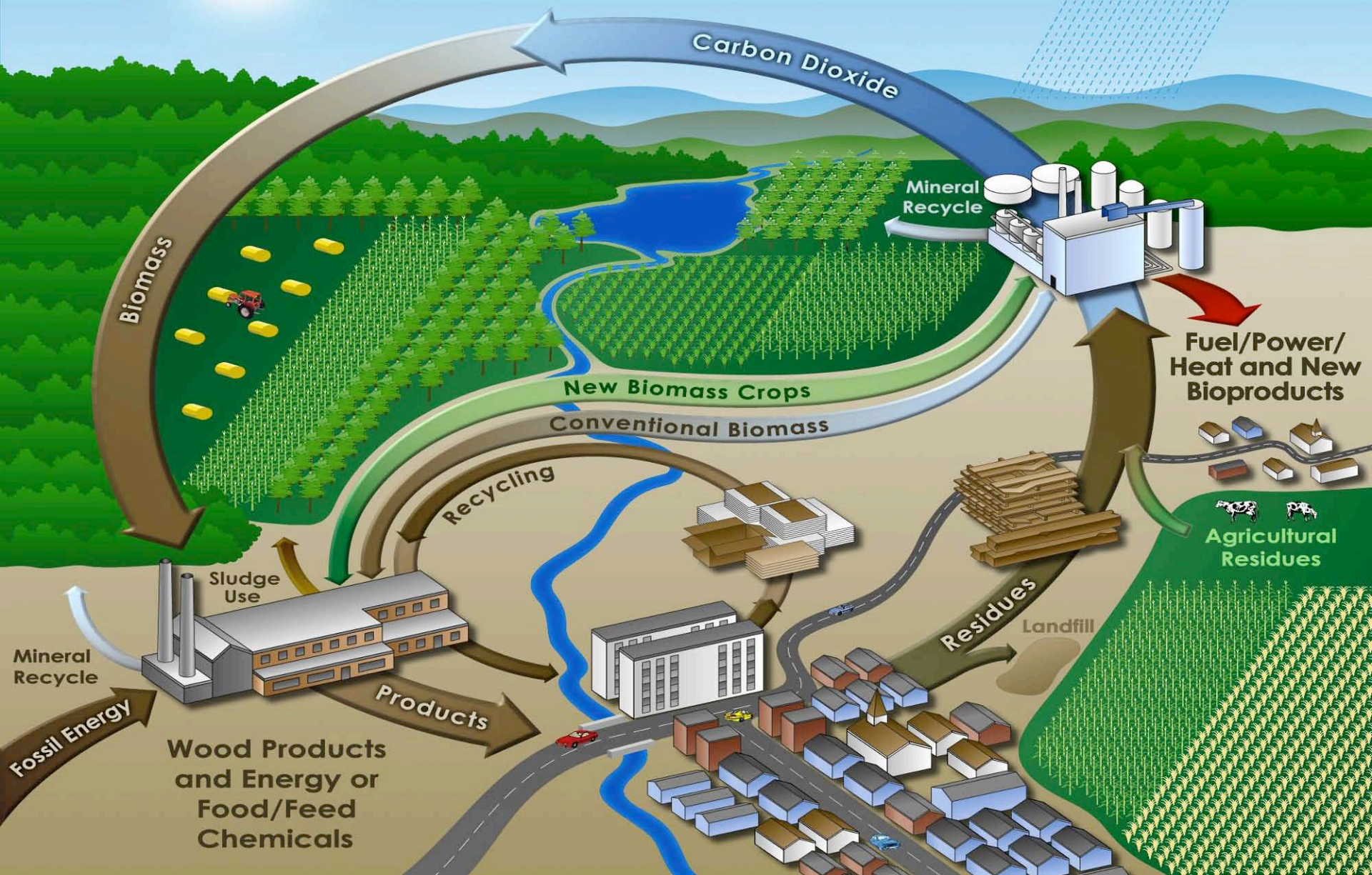
- **FY13-14: Slurry Characterization and Separation Science:** Progressed rheometry techniques (LAOS), membrane-based dewatering (NF) and showed proof of concept for advanced CEH process technology applicable to out-year targets (2022)
- **FY15: Sugar Stream Production:** Completed developing base method for SLS of problematic post EH lignin-rich fine particulate slurries
- **FY15: Fuel Precursor Recovery:** Literature review on track, with many potentially effective methods identified; early experimental results show that high extraction yields and purities possible.

**Relevance:** Low-cost, efficient separations are essential to achieving cost-competitive sugars-to-hydrocarbon biofuels process. Ability to test extraction using frozen (freeze-dried) cells facilitates more efficient experimental designs.

**Future Work:** Likely need to develop C5 stream concentration



# Questions?



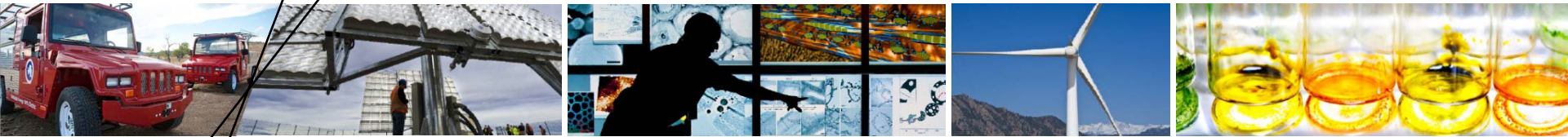
# Acknowledgments



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**SDA Project Team (NREL):** Gregg Beckham, Mary Biddy, Nick Cleveland, Nate Crawford, Tao Dong, Jake Kruger, Jim Lischeske, David Sievers, Jonathan Stickel





# **Additional Slides**

**(for reviewers, not for presentation)**

# Responses to Previous Reviewers' Comments

- There were no significant questions/criticisms of the “Slurry Characterization and Separation Science” work presented at the 2013 Peer Review (within the Pretreatment and Enzymatic Hydrolysis project).
- Reviewers noted (on p262 of 2013 Peer Review Report) that this work “*could benefit from additional direction from BETO and from greater interaction with the engineering modeling group*” [i.e., the BC Platform Analysis project (BPA)]. This is now occurring, as reflected by recent jointly authored publications and by Mary Bidy, a key member of the BPA team, also participating in the SDA fuel precursor (lipid) recovery task. The TEA guidance continues to evolve as the process design is refined.

# Publications

## **Sugar Stream Separation publications** (includes Slurry Characterization and Separations Science) (FY13-present)

1. Stickel, J.J., Knutsen, J.S., Liberatore, M.W. 2013. Response of elastoviscoplastic materials to large amplitude oscillatory shear flow in the parallel-plate and cylindrical-Couette geometries. *J. Rheology*, 57(6):1569-1596.
2. Stickel J.J., Knutsen J.S., and Liberatore M.W. 2014. Connecting large amplitude oscillatory shear rheology to steady simple shear rheology and applications to biomass slurries. *Appl. Rheol.*, 24(5):53075.
3. Lischeske, J.J. and Nelson, R.S. and Stickel, J. J. 2014. Benchtop methods for measuring the fraction of free liquid of biomass slurries. *Cellulose*, 21(4):2261-2269.
4. Malmali M., Stickel J.J., and Wickramasinghe S.R. 2014. Sugar concentration and detoxification of clarified biomass hydrolysate by nanofiltration. *Sep. Purif. Technol.*, 132:655-665.
5. Stickel J.J., Brunecky R., Elander R.T., and McMillan J.D. 2014. Enzymatic hydrolysis of lignocellulosic biomass. *In: Bisaria V.S. and Kondo A., editors, Bioprocessing of Renewable Resources to Commodity Bioproducts*, pp. 77-103. John Wiley & Sons, Hoboken, New Jersey.
6. Sievers, D. A., Tao, L., Schell, D. J. 2014. Performance and techno-economic assessment of several solid-liquid separation technologies for processing dilute-acid pretreated corn stover. *Bioresource Technology*, 167:291-296.

## **Submitted, in review**

7. Malmali M., Stickel J.J., Wickramasinghe S.R. 2015. Investigation of a submerged membrane reactor for continuous biomass hydrolysis. *Food Bioprod. Process.* In review.
8. Sievers, D.A., Lischeske, J.J., Bidy, M.J., Stickel, J.J. 2015. A low-cost solid-liquid separation process for enzymatically hydrolyzed corn stover slurries. *Bioresource Technology*. In review.

# Presentations

## Sugar Stream Separation presentations (includes Slurry Characterization and Separations Science) (FY13-FY15)

### FY13

1. Griggs A.J., Nag A., Lischeske J.J., Stickel J.J., Mittal A., Wang W., Johnson D.K. Mechanistic modeling of the enzymatic saccharification of cellulose-I<sub>β</sub> and cellulose-III<sub>1</sub>: parameter determination and model validation. Presented at the AIChE annual meeting, Pittsburgh, PA, October, 2012.
2. Lischeske J.J., Sprague M.A., Nelson R., Grosso-Giordano N., Stickel J.J. Using solute exclusion to probe structure and diffusive transport in biomass slurries. Presented at the AIChE annual meeting, Pittsburgh, PA, October, 2012.
3. Malmali M., Stickel J.J., Wickramasinghe, S.R. Sugar concentration for continuous enzymatic saccharification using nanofiltration and reverse osmosis membranes. Presented at the AIChE annual meeting, Pittsburgh, PA, October, 2012.
4. Sprague M.A., Stickel J.J., Lischeske J.J., Fischer P. Computational modeling of dilute biomass slurries. Presented at the 65th APS DFD Meeting, San Diego, CA, November, 2012.
5. Stickel, J. Response of an elastoviscoplastic material to oscillatory shear flow in the parallel plate and cylindrical Couette geometries. Presented at the 84th Annual Meeting of the Society of Rheology, Pasadena, CA, February, 2013.
6. Brodeur G., Ramakrishnan S., Wilson C., Collier J., Telotte J., and Stickel J.J. Combined dilute acid and solvent based pretreatment of agricultural wastes for efficient lignocellulosic fractionation and biofuels production. Presented at the 245th ACS National Meeting & Exposition, New Orleans, LA, April, 2013.

# Presentations, continued

## Sugar Stream Separation presentations (includes Slurry Characterization and Separations Science) (FY13-FY15, i.e., through February 2015)

### FY14

8. Lischeske J.J., Nelson R.S., and Stickel J.J. Measuring free water content and pore-size distributions in lignocellulosic biomass. Presented at the American Institute of Chemical Engineers Annual Meeting, San Francisco, CA, November, 2013.
9. Malmali M., Stickel J.J., and Wickramasinghe S.R. Continuous enzymatic hydrolysis of biomass in a membrane-assisted reactor. Presented at the American Institute of Chemical Engineers Annual Meeting, San Francisco, CA, November, 2013.
10. Adhikari, B. Development of a membrane-based separation process for the continuous enzymatic saccharification of lignocellulosic biomass. Presented at the 36th Symposium on Biotechnology for Fuels and Chemicals, Clearwater, FL, April 29, 2014.

### FY15

11. **Crawford, N. The non-monotonic torque response of a model cellulosic biomass slurry during settling and resuspension.** Poster presented at the 86th annual meeting of the Society of Rheology, Philadelphia, PA, October 8, 2014
12. Stickel J.J., Sievers D.A., Lischeske J.J., Crawford N.C., and Biddy M. Flocculation assisted clarification of enzymatically hydrolyzed corn stover slurries. Presented at the AIChE annual meeting, Atlanta, GA, November, 2014.
13. Crawford, N.C., Sievers, D.A., Nagle, N., Stickel, J.J., Ray A., and Yancey, N. Flowability of biomass solids: The effects of feedstock preprocessing. Presented at the AIChE annual meeting, Atlanta, GA, November, 2014.

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