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U.S. Department of Energy (DOE) Bioenergy Technologies Office (BETO) 2017 Project Peer Review

2.2.1.101-102 Feedstock Process Interface & Biochem Blended Feedstock Development

March 9, 2017

Feedstock Conversion Interface Consortium

Allison Ray (INL)

Nick Nagle (NREL)

Goal Statement

<u>GOAL</u>: To employ blending and densification strategies that enable feedstock consistency for efficient conversion to sugar and lignin-derived fuels and chemicals in biochemical conversion pathways

<u>OUTCOME:</u>

- Qualification of a blended feedstock that meets cost, quality and performance for 2022 BC verification

<u>RELEVANCE:</u> Integrated biorefineries (IBRs) have struggled with problems that stem from variability, feeding and handling, and cost

- Develop consistent feedstocks for BC pathway
- Evaluate preprocessing strategies aimed at solving challenges associated with feeding & handling
- Reduce cost through incorporation of low-cost biomass resources
- Expand resource availability through blending

Quad Chart Overview

Timeline

- Start: October 2015
- End: September 2018
- Percent complete: 50%

Budget

	FY 16 Costs	Total Planned Funding (FY 17-Project End Date)
DOE Funded NREL	$568.5K	$1.2M
DOE Funded INL	$975.5K	$2.0M

Barriers

- Barriers addressed
  - Ft-A, Feedstock Availability and Cost
  - Ft-G, Feedstock Quality and Monitoring
  - Bt-A, Biomass and Feedstock Variability

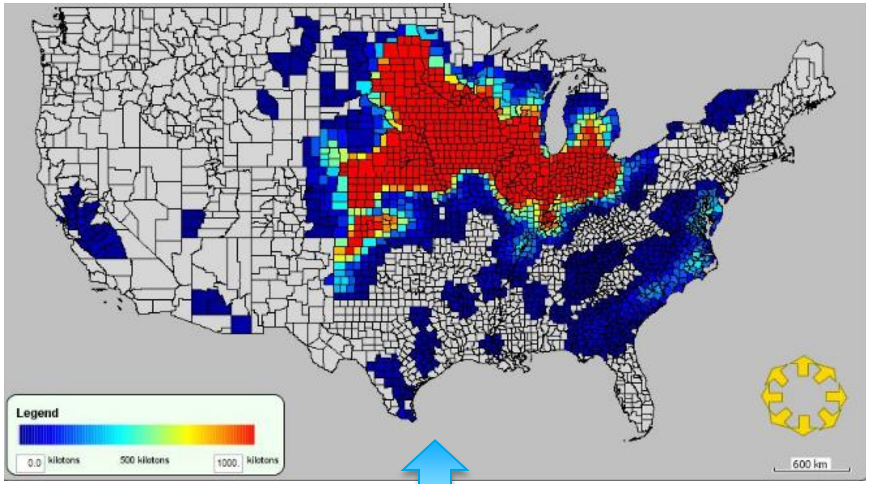
Partners

- INL
- NREL
- LBNL
- Bliss Industries
- U.S. Department of Agriculture
- North Carolina State University
- University of Kentucky
- University of Georgia CCRC

1 – Project Overview

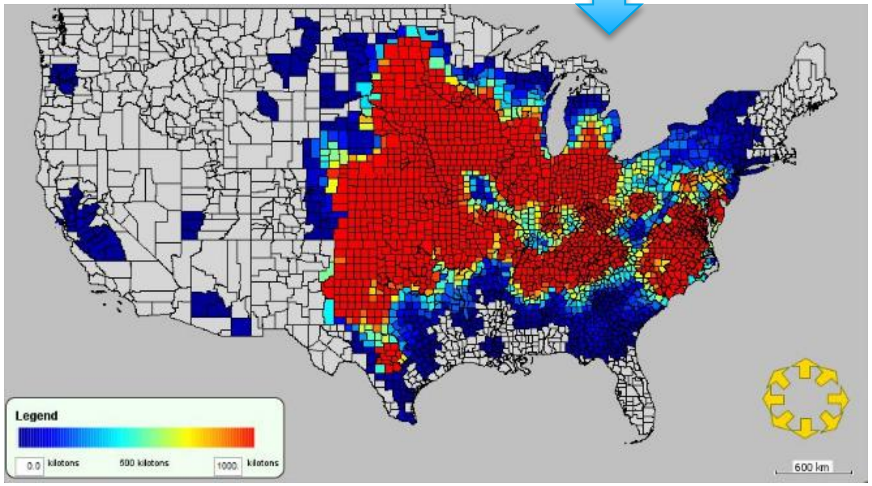
CONTEXT: WHY BLEND?

- Diversifies biomass supply to reduce cost and risk to the supply chain
- Bridges seasonal gaps in availability
- Incorporates low-cost, low-quality biomass resources to reduce cost



Projected **corn stover** density in 2030, available for \$50/DMT and 50-mile harvest radius.

Projected density for **corn stover** and **perennial grass** **blend** in 2030, available for \$50/DMT and 50-mile harvest radius.



- Overcomes biomass variability challenges related to feedstock handling and conversion performance

A successful blending strategy has to meet cost, volume, and performance targets AND be relevant to industry. This project is focused on performance.

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https://bioenergylibrary.inl.gov/Research/LCF.aspx

1 – Project Overview

HISTORY

- INL and NREL initiated the feedstock conversion interface project in 2006
- Partnership studied the impact of densification on the biochemical pathway in 2011

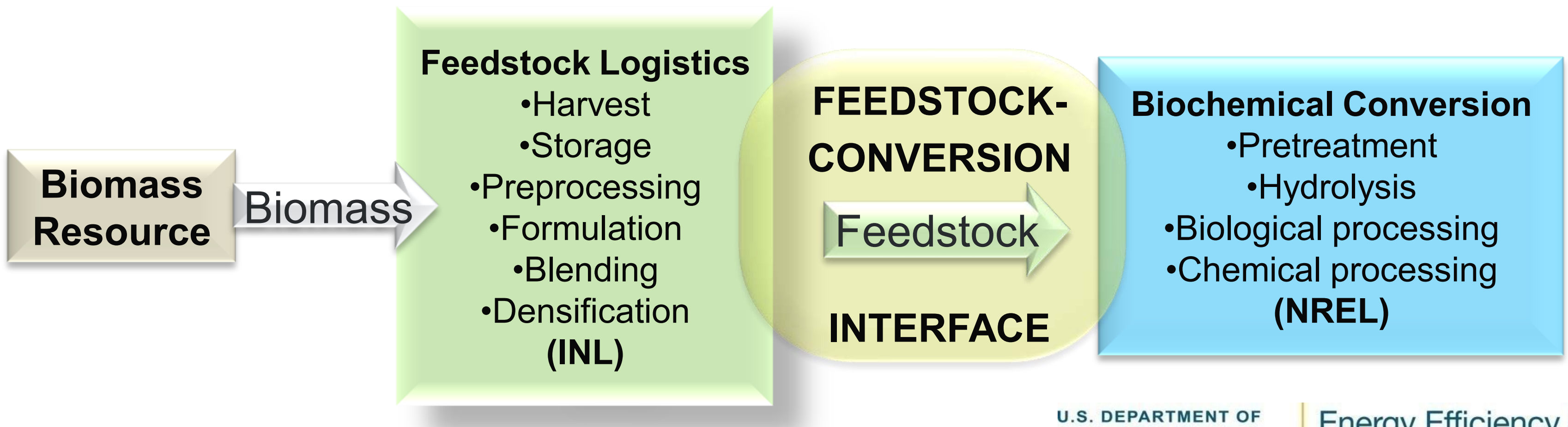
OBJECTIVES

- Qualify a blend for 2022 biochemical pathway verification
- Develop consistent, conversion-ready feedstocks for BC pathway
- Develop models to predict blend performance
- Formulate blend capable of attaining of equivalent sugar and lignin yields to a model feedstock (corn stover)
- Demonstrate continuous conversion and operation of blended/densified feedstocks
- Understand the impact of feedstock properties on conversion performance

2 – Approach (Management)

APPROACH:

- Joint AOP and milestone development
  - FY16 Q2 NREL Go/No Go milestone: Demonstrate continuous pretreatment of blended and densified feedstocks
  - FY17 Q2 INL Go/No-Go: Demonstrate 90% sugar release compared to corn stover at lab-scale for two candidate blends in deacetylation, DAP and enzymatic hydrolysis
- Collaboration between INL and NREL strengthens impact of results
  - Transfer of information between projects accelerates progress
  - Integration of targets for both biochemical conversion and feedstock logistics
  - Response to industry challenges as they emerge



2 – Approach (Technical)

APPROACH:

- Extensive characterization of biomass feedstocks
- Blend formulation & densification to improve consistency (INL)
- Lab-scale pretreatment & enzymatic hydrolysis (INL/NREL/LBNL)
- Evaluation of conversion performance
- Larger-scale continuous conversion testing under process-relevant conditions (200 kg per day) (NREL)

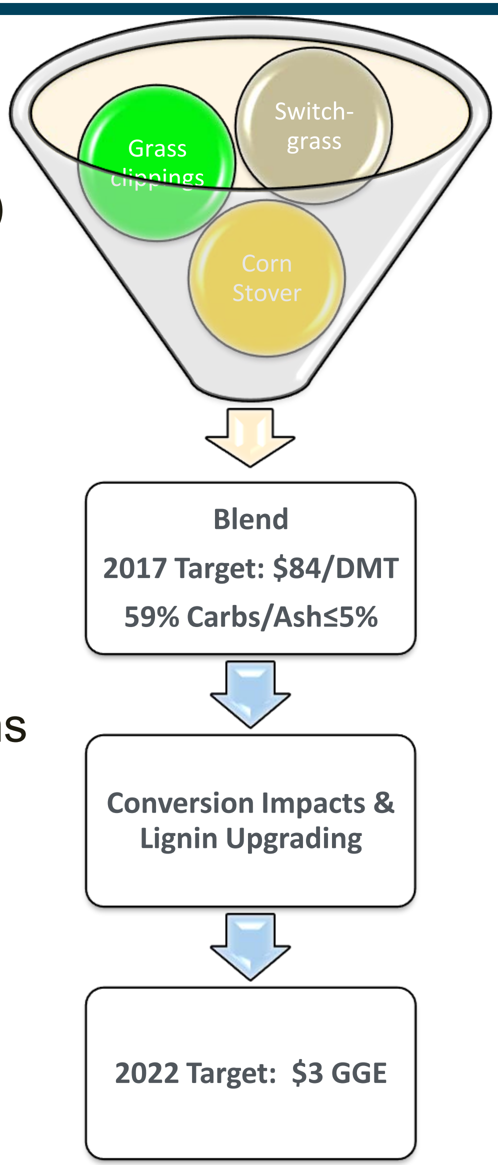
CHALLENGES:

- Managing feedstock variability (e.g., fines)
- Operating under larger-scale, continuous conversion conditions

CRITICAL SUCCESS FACTORS:

Blends in densified formats must have…

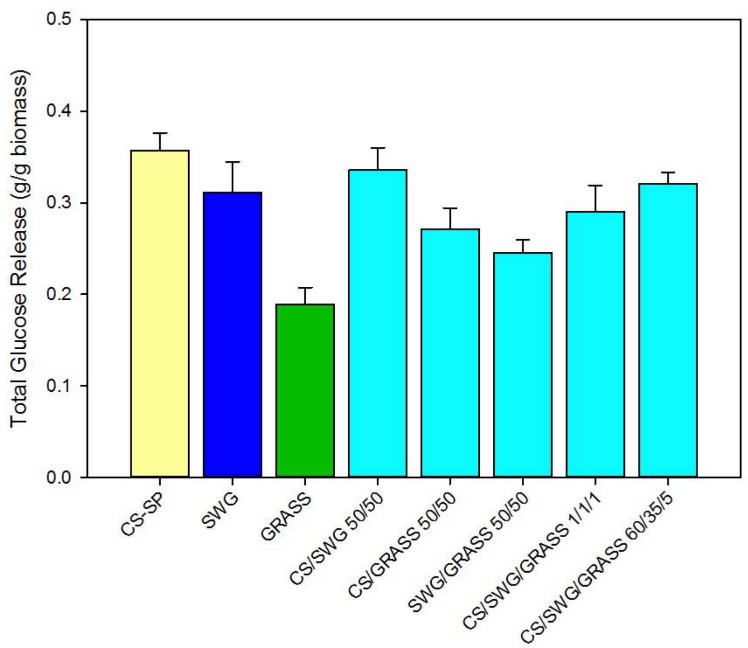
- Predictable conversion performance
- Sufficient sugar and lignin yields compared with model feedstock
- Value-added chemicals derived from sugars and lignin



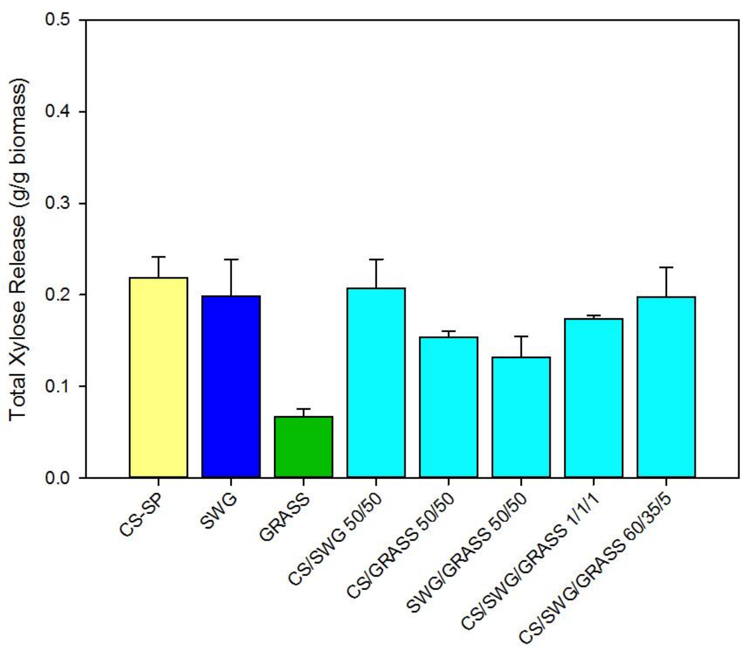
3 – Technical Accomplishments/Performance Evaluation

Blends of CS/SWG (50/50) and CS/SWG/Grass (60/35/5) achieved equivalent sugar release to corn stover (p>0.05).

Glucose



Xylose



Glucose and xylose release per gram of biomass from combined dilute-acid pretreatment and enzymatic hydrolysis.

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3 – Technical Accomplishments/Predictive Model

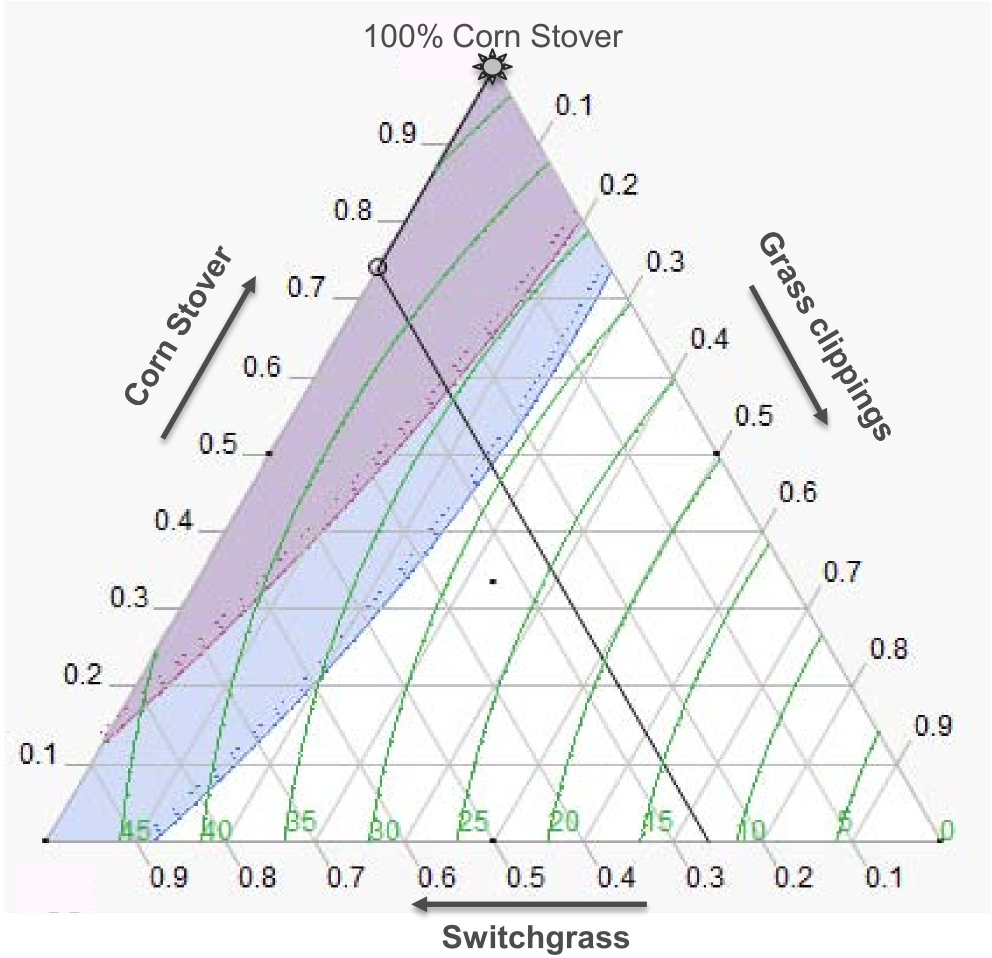
- Ternary plot of sugar release and cost for blends of corn stover, switchgrass, and grass clippings from a mixture design

- Corn stover was used as the baseline for performance

- Response limits for glucose (purple) and xylose (blue) release from dilute-acid pretreatment and enzymatic hydrolysis are shown

- Maximum sugar release and minimum cost corresponded to a blend formulation…

CS/SWG = 73%/27%



Predictive models enable blend formulation as a function of cost, quality, and conversion performance.

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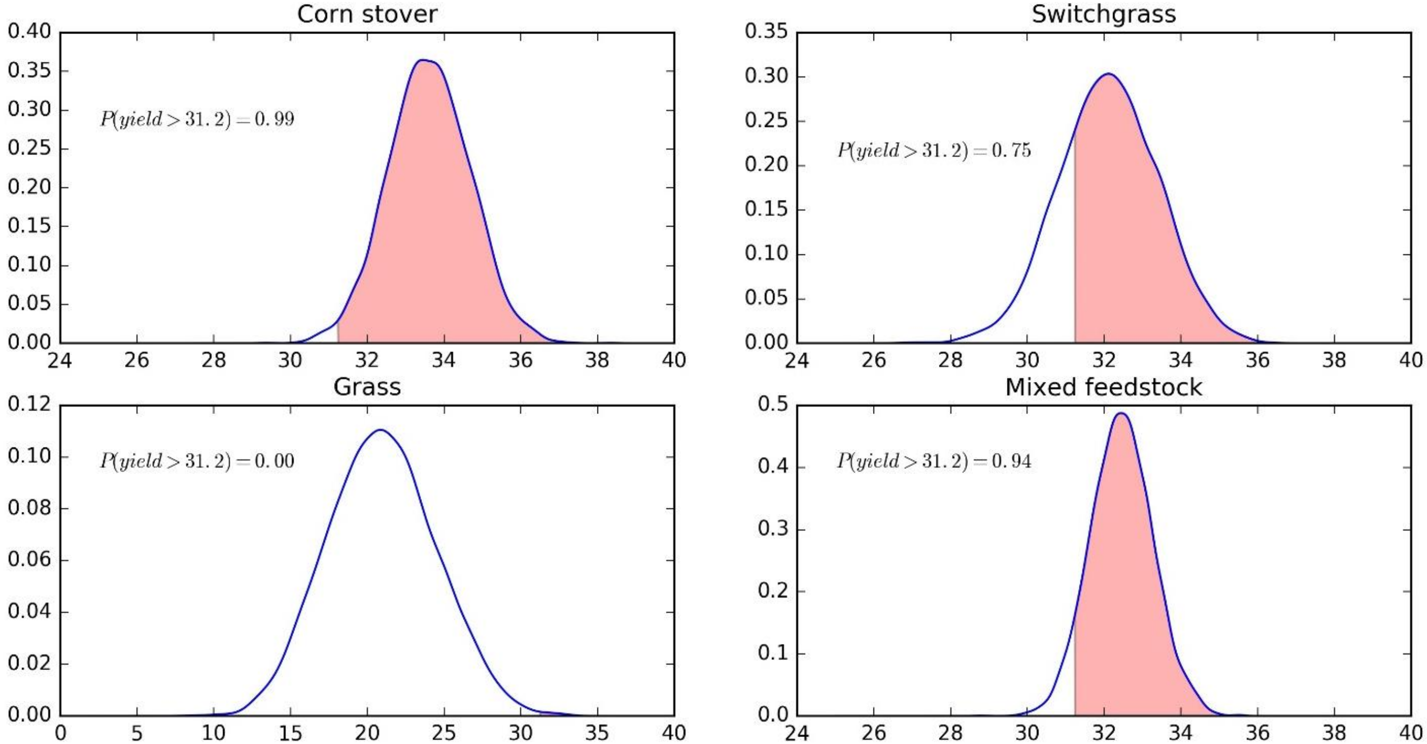
3 – Technical Accomplishments/Uncertainty Analysis

- Reduce the impact of variability on performance
- Blending results in more consistent yield performance

Probability Density vs. Glucose Yield (g/100 g biomass)



With a blended feedstock, availability is improved, variability is reduced, and probability of achieving target sugar yield remains high.

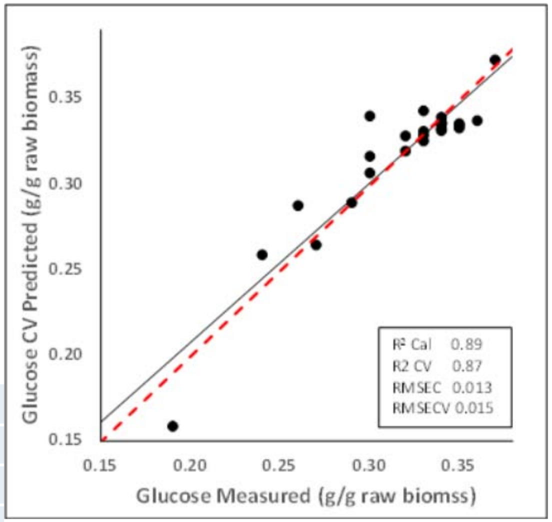
3 – Technical Accomplishments/Blend Linearity

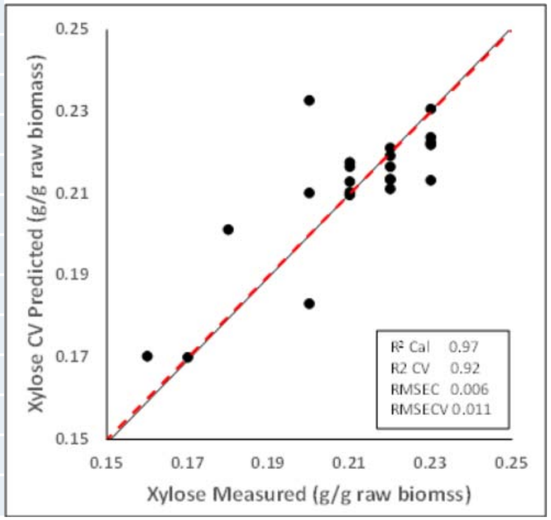
Blends are predictable

Chemical properties of 26 samples were used to develop predictive models

- 9 biomass types
- 17 blends
- Chemical properties as predictor variables

#	BLEND	RATIO
1	CS/SWG	50/50
2	CS/SWG	60/40
3	CS/SWG	80/20
4	CS/SWG	70/30
5	CS-MP/SWG	70/30
6	CS-MP/SWG	80/20
7	CS MIX/SWG/MSW	60/35/5
8	CS MIX/SWG/ID grass	60/35/5
9	CS MIX/SWG/SORG	60/35/5
10	CS MIX/SWG/AR grass	60/35/5
11	CS MIX/SWG/WHE	60/35/5
12	CS MIX/SWG/MIS	60/35/5
13	SWG/ID Grass	50/50
14	CS/SWG/ID Grass	1/1/1
15	CS/ID Grass	50/50
16	CS/SWG	50/50
17	CS/SWG/ID Grass	60/35/5





Sugar release for blends can be predicted from the composition and sugar release of pure biomass.

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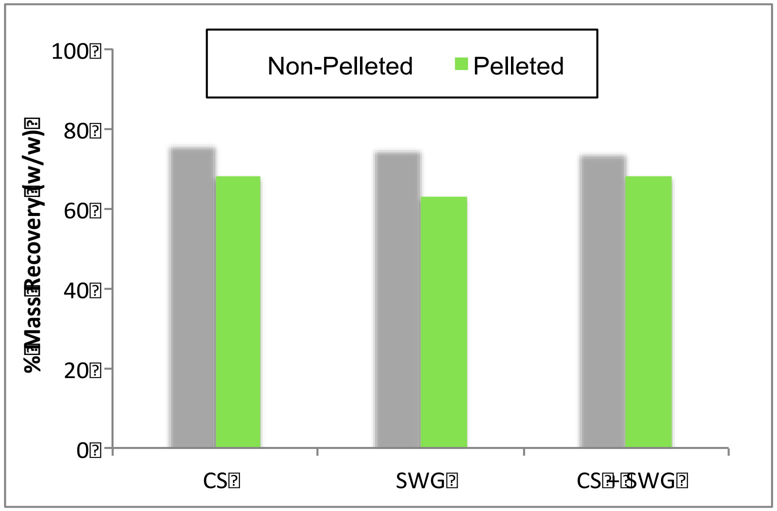
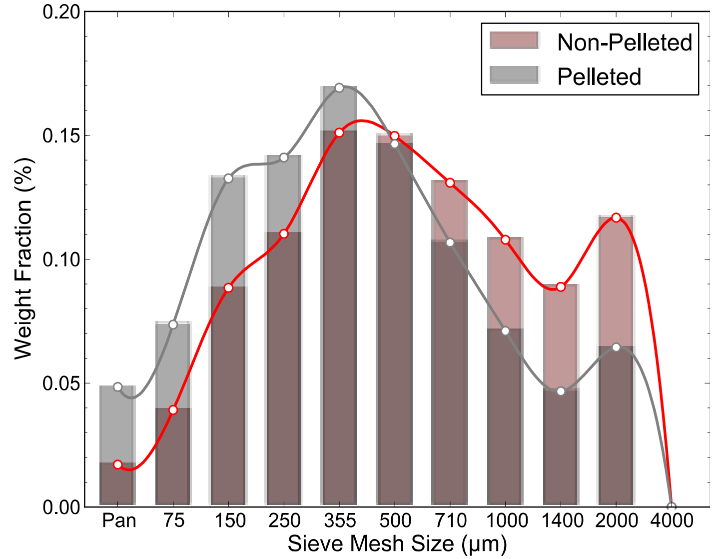
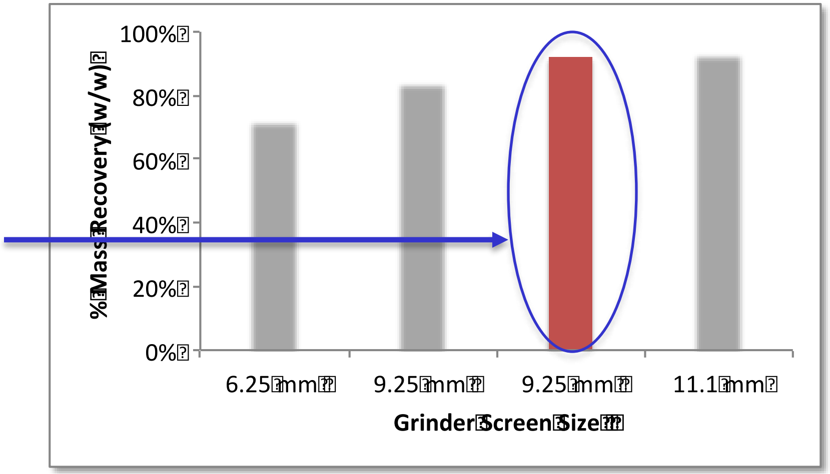
3 - Technical Accomplishments/Mass recovery after deacetylation and acid impregnation



Mass recovery after deacetylation w/ 0.2% NaOH (w/w), 1% Acid (w/w) impregnation



- **Preprocessing that targets fines removal improves mass recovery in pretreatment**
- Larger particle sizes improve solids recovery after deacetylation

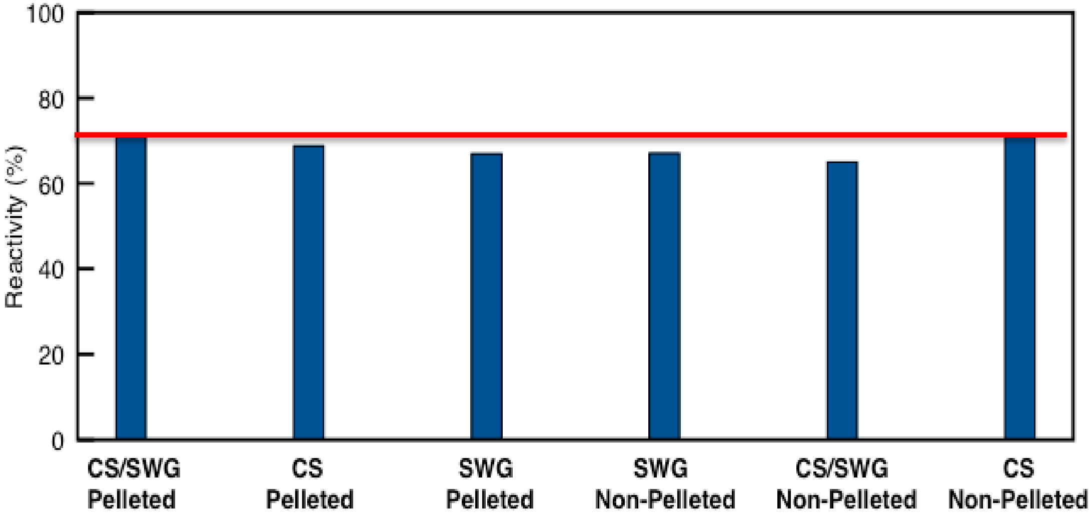


Improve in-feed properties → improve mass recovery

Mass recovery after deacetylation and acid impregnation of ground corn stover prepared with different screen sizes.

3 – Technical Accomplishments/Continuous Pretreatment

- **Go/No-Go: Demonstrated continuous dilute-acid pretreatment (DAP) for blends, pellets under process-relevant (high solids loading) conditions**
- Pelleted blends improved feeding (reduced motor torque in screw conveyors)
- Pelleted blend produced comparable sugar yields to model feedstock



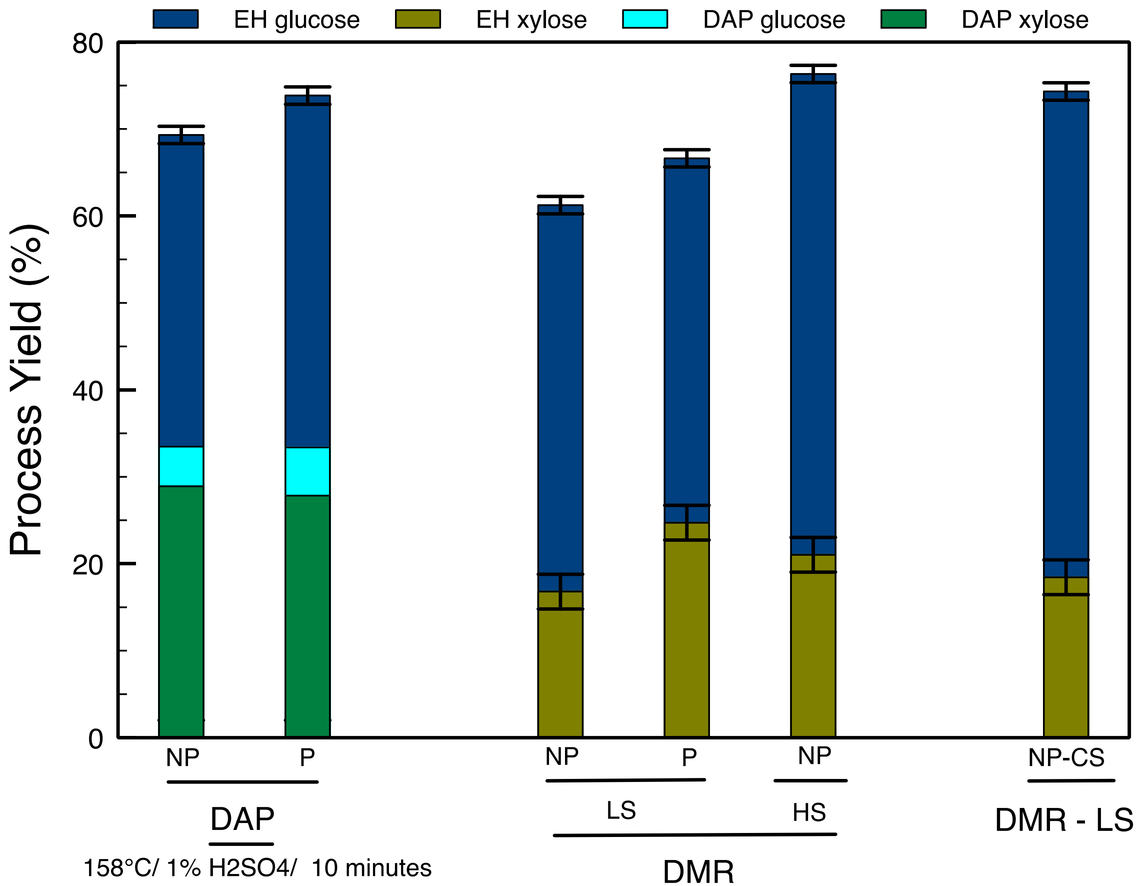
Reactivity is a weighted measure of monomeric xylose and glucose recovered from dilute-acid pretreatment (DAP) and enzymatic hydrolysis.

200kg/day, NREL horizontal pretreatment reactor

Run Conditions:

- 200 kg/day horizontal reactor
- 1% Acid (w/w) pre-impregnated
- Feed rate -175g/min
- Solids loading 40%-45%(w/w)
- Reaction temp-155^{o}C
- Residence time-10 min
- Run time 2 hr./condition

3 – Technical Accomplishments/Blend Conversion to Sugars



Total sugar yields from CS/SWG (50/50) blend, pelleted and non-pelleted using two pretreatment processes, dilute acid (DAP) and deacetylated and mechanically refined (DMR**)**.

Low Severity-DMR: 50 kg NaOH/metric ton, 80 C; 10% solids

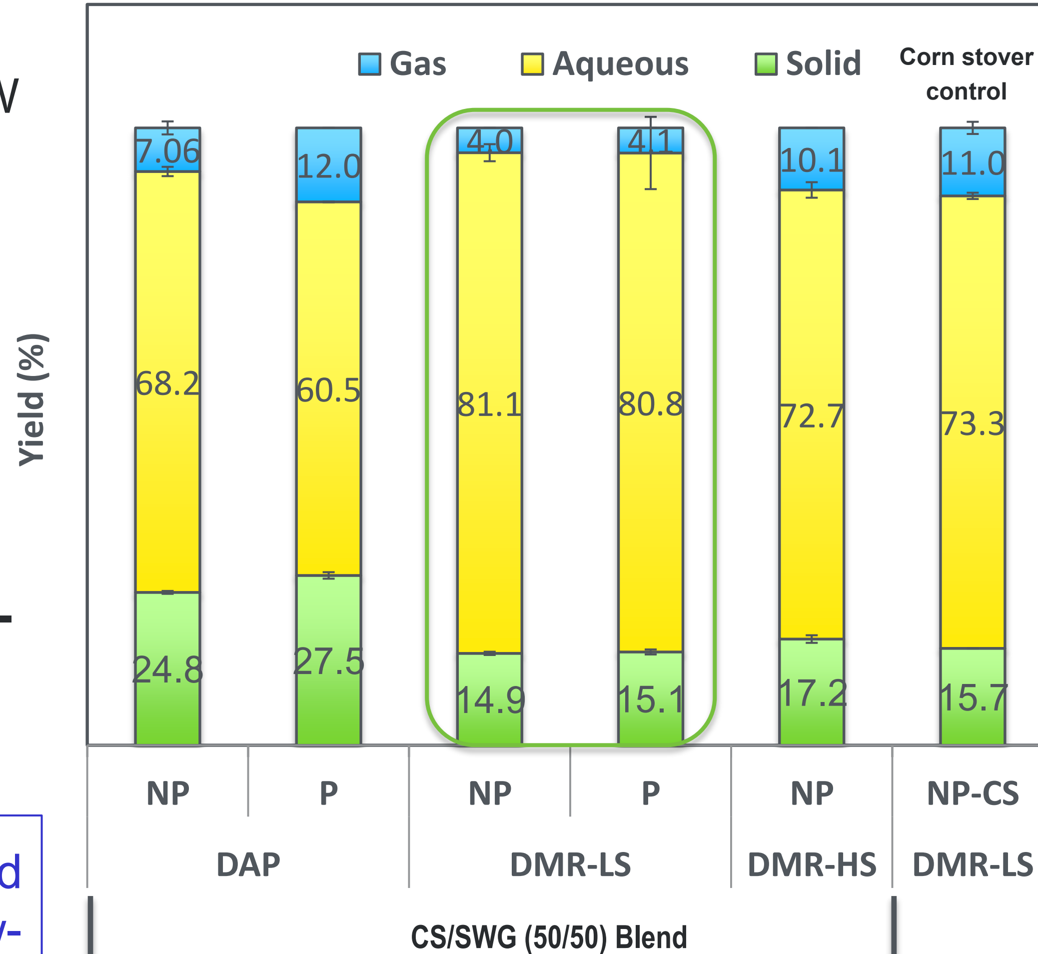
High Severity-DMR: 70 kg NaOH/metric ton, 92 C; 10% solids

- Evaluated sugar yields from blends using DAP and deacetylation and mechanical refining (DMR)
- **Pelleting did not increase biomass recalcitrance**
- **Higher severity deacetylation increased sugar release (glucose and xylose) for DMR compared to DAP**

3 – Technical Accomplishments/Depolymerization of lignin-rich residues

- Evaluate blended and pelleted feedstocks for production of low-MW compounds from lignin
- Base-catalyzed depolymerization (BCD) on pretreated and enzymatically hydrolyzed residues
- **Low-severity deactylated and mechanically-refined (DMR-LS) pretreatment produced more low-MW intermediates than DAP and DMR-HS**

Blends and pellets demonstrated compatibility with production of low-MW compounds from lignin: DMR-LS blends had higher yields than non-pelleted corn stover



Yields (%) from base-catalyzed depolymerization (BCD) of pretreated and enzymatically-hydrolyzed feedstocks (NP: non-pelleted; P: pelleted; DAP: dilute-acid pretreatment; DMR-LS: low-severity deacetylation; DMR-HS: high-severity deacetylation).

4 – Relevance

GOAL: To employ blending and densification strategies that enable feedstock consistency for efficient conversion to sugar and lignin-derived fuels and chemicals in biochemical conversion pathways

For industry, blended and densified feedstocks…

- Reduce cost and risk to the supply chain, bridge gaps in seasonal availability, incorporate low-cost biomass, and overcome variability challenges
- Enable high sugar AND aqueous lignin yields for production of fuels AND high-value co-products—value-added co-products are requisite for biorefinery development and process economics

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4 – Relevance

BETO Cost and Performance Goals:

- Qualification of a blend for the 2022 BC verification
- Delivered feedstock cost of $84/dry ton
- Meet volume requirement of 800,000 dry tons within biorefinery draw radius
- Incorporates low-cost biomass resources

Blending and densification improve utilization of diverse biomass resources to ensure biomass availability and quantity to achieve the billion ton goal and enable a robust biorefining industry for biofuels and bio-products.

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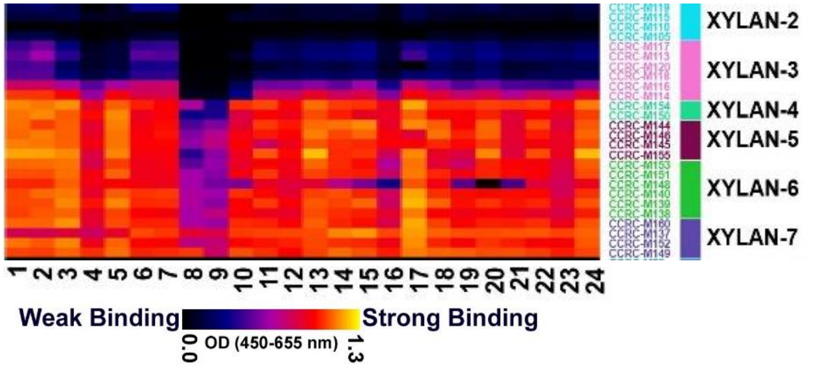
5 – Future Work

A better understanding of feedstock properties as they relate to…

- Feed, handling and flow behavior of pretreated solids
- Cell wall structure and chemistry for biomass compatibility and blend formulation
- Selection of pretreatment chemistry
- Conversion performance

Key milestones include:

- Evaluation of densified formats to enable high-solids loading in pretreatment
- Coupling supply chain and BC conversion to quantify the impact of blended, densified feedstocks on Minimum Fuel Selling Price (MFSP)
- Blend qualification for 2022



Courtesy of S. Pattathil, University of Georgia, Complex Carbohydrate Research Center.

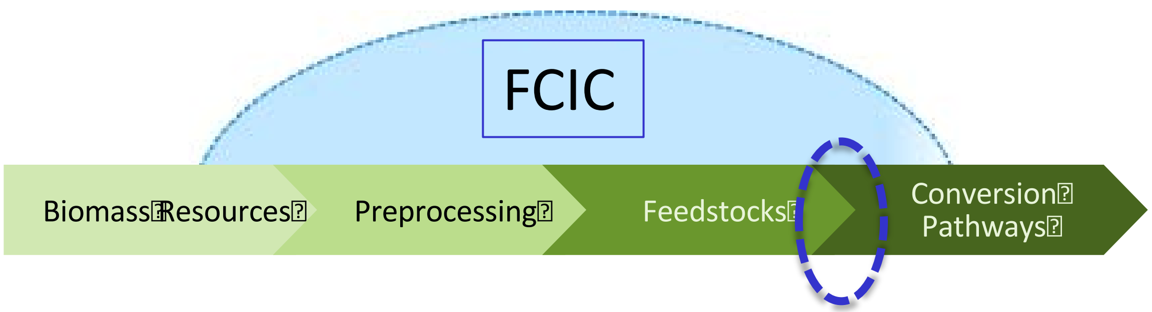
Lane #	Biomass Type or Blend
1	Corn Stover (CS)
3	Switchgrass (SWG)
4	Wheat Straw (WS)
8	Hybrid Poplar
16	CS/Poplar (50/50)
20	CS/SWG/WS (80/10/10)

Glycome profiling is a rapid method that employs a diverse suite of antibodies to detect major non-cellulosic polysaccharides present in plant cell walls (hemicelluloses) to enable selection of pretreatment chemistry and determination of biomass compatibility for blending.

5 – Future Work

Support Industry Goals:
- Examine the efficacy of blends for conversion to final product
- Serve as a resource to solve industry problems and challenges
- Leverage user facilities to scale laboratory results to high impact findings for industry

Support FY18 FCIC Goals:
- Address near-term industry challenges related to variability in feedstock properties, feeding and handling issues
- Support biomass grading and specifications development



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Summary

1. **Overview:** Feedstock blending and densification strategies for efficient conversion to sugar and lignin-derived fuels and chemicals in biochemical conversion pathways

2. **Approach:** Testing feedstock blends and pellets under process-relevant conditions and developing feedstock/conversion predictive models for performance and formulation

3. **Technical Accomplishments:**
   - Predictive model developed for blend formulation ($, quality, and performance)
   - Process-relevant, continuous pretreatment was demonstrated for blended, pelleted feedstocks (Go/No-go milestone)
   - DMR-LS blends and pellets had higher yields of low-MW compounds than corn stover—compatible with upgrading to co-products.

4. **Relevance:** Blending and densification improve utilization of diverse biomass resources to ensure biomass availability and quantity to achieve the billion ton goal and enable a robust biorefining industry for biofuels and bio-products.

5. **Future Work:** An emphasis on understanding how fundamental feedstock properties translate to performance.

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BETO

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Additional Slides

Comments from previous review

Reviewer: Current work has been batch; project needs to look at continuous processes, which has been indicated as a project goal. Staffing and time constraints listed as a challenge, need to focus on prioritization to get most out of project in schedule timeline.

Response: *We agree with the reviewers regarding the scale up and operation under continuous conditions. We have plans in the both the FY16 and FY17 AOP, to demonstrate bioconversion using additional processes such as alkaline pretreatment or by DMR (deacetylation and mechanical refining) under continuous operating conditions. In addition we'll be mindful of the resources needed to accomplish this research plans, identifying and focusing on key elements needed to meet the schedule timeline*.

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Publications, Patents, Presentations, Awards, and Commercialization

Publications

- Edward J. Wolfrum, Nicholas J. Nagle, Ryan M. Ness, Darren J. Peterson, Allison E. Ray, Daniel M. Stevens. The Effect of Biomass Densification on Structural Sugar Release and Yield in and Biofuel Feedstock and Feedstock Blends. 2017, BioEnergy Research, (doi:10.1007/s12155-017-9813-z)
- Allison E. Ray, Chenlin Li, Vicki S. Thompson, Dayna L. Daubaras, Damon S. Hartley, and Nick J. Nagle. Biomass Blending and Densification: Impacts on Feedstock Supply and Biochemical Conversion Performance. Biomass Volume Estimation and Valorization to Energy. 2017, ISBN 978-953-51-4909-5
- Godin, B., Wolfrum, E., Nagle, N., Sattler, S., Agneessens, A., Delcarte J. Improved sugar yields from biomass sorghum feedstocks: Comparing low-lignin mutants and pretreatment chemistries. Biotechnology for Biofuels. November 2016. DOI: 10.1186/s13068-016-0667-y
- Lischeske, J., Crawford, N.C., Kuhn., K., Nagle, N.J., Schell, D.J., Tucker, M.P., McMillan, J.D., Wolfrum, E.J. Assessing pretreatment reactor scaling through empirical analysis. Biotechnology for Biofuels. October 2016. DOI: 10.1186/s13068-016-0620-0.
- Payne, C., Wolfrum, E., Nagle, N., Brummer, J.E. and Hansen, N.C. Cool Season Grasses as Biofuel Feedstocks: NIR/PLS Predictions of Carbohydrate Yields. Submitted to the Agronomy Journal. Submitted September 2016.
- Crawford, N.C., Nagle, N.,. Sievers, D.A., Stickel , J. 2016. The effects of physical and chemical preprocessing on the flowability of corn stover. Biomass and Bioenergy 85, 126-134.
- N. C. Crawford, A. E. Ray, N. A. Yancey, N. Nagle. Evaluating the pelletization of “pure” and blended lignocellulosic biomass feedstocks. Fuel Processing Technology 140, 46-56 (2015)
- Akash Narani, Phil Coffman, James Gardner, NVSN Murthy Konda, Chyi-Shin Chen, Firehiwot Tachea, Chenlin Li, Allison E. Ray, Allison Stettler, Blake Simmons, Todd Pray, and Deepti Tanjore. Predicting optimal feedstock blends and process parameters to de-risk bio-based manufacturing (Submitted February 2017)
- Allison E. Ray, Daniel Stevens, Kara Cafferty, Dayna L. Daubaras, Kastli Schaller, Lorenzo Vega Montoto, Rachel M. Emerson, Vicki S. Thompson, and Chenlin Li. Impact of blending corn stover, switchgrass, and MSW grass on biochemical conversion performance and feedstock cost. (In preparation)

Publications, Patents, Presentations, Awards, and Commercialization

DOE LAB-CORPS Cohort 4 Team Selection: Team Opti-blend (INL)

- Allison Ray (Principal Investigator), Hongqiang Hu (Entrepreneurial Lead), and Ryan Bills (Industry Mentor) of Team Opti-blend graduated from LAB-CORPS Cohort 4 on December 5-8, 2016 in Denver, CO. Team OptiBlend conducted 75 interviews during the 6-week course with contacts from industry, national laboratories, and academia.

Conference Presentations

- Allison E. Ray, Daniel Stevens, Kara Cafferty, Dayna L. Daubaras, Kastli Schaller, Lorenzo Vega Montoto, Rachel M. Emerson, Vicki S. Thompson, & Chenlin Li. Impact of blending corn stover, switchgrass, and MSW grass on biochemical conversion performance and feedstock cost. Frontiers in Biorefining. November 8-11, 2016 in St. Simon's Island, GA.
- Allison E. Ray, Chenlin Li, Vicki S. Thompson, Dayna Daubaras and Nicholas J. Nagle. Biomass Blending & Densification: Impacts on Biochemical Conversion Performance. American Institute of Chemical Engineers (AIChE) 2016 Annual Meeting. San Francisco, CA; November 2016
- Akash Narani, Phil Coffman, Matthew Miller, Firehiwot Tachea, Chyi-Shin Chen, Chenlin Li, Allison E. Ray, Todd Pray and Deepti Tanjore. Predictive Model and Bioconversion of Mixed Feedstocks. American Institute of Chemical Engineers (AIChE) 2016 Annual Meeting. San Francisco, CA; November 2016
- Nick Nagle, Edward Wolfrum, David Templeton, Allison E. Ray, Neal Yancey, Jaya Tumuluru and Nathan Crawford. Impact of preprocessing on bioconversion yields from densified and blended feedstocks. Symposium on Biotechnology for Fuels and Chemicals. Baltimore, MD, April 25-28,2016
- Impact of blending corn stover, switchgrass, and MSW grass on biochemical conversion performance and feedstock cost, Allison E. Ray, Daniel Stevens, Kara Cafferty, Kastli Schaller, Dayna L. Daubaras, Lorenzo Vega Montoto, Rachel M. Emerson and Vicki S. Thompson, presented at the Symposium on Biotechnology for Fuels and Chemicals. Baltimore, MD, April 25-28,2016
- Deacetylation and dilute acid pretreatment of corn stover, switchgrass, and stover-switchgrass mix. E.M. Kuhn, X. Chen, N.J. Nagle, M.P. Tucker, N. Yancey, A. Ray. Symposium on Biotechnology for Fuels and Chemicals. Baltimore, MD, April 26, 2016

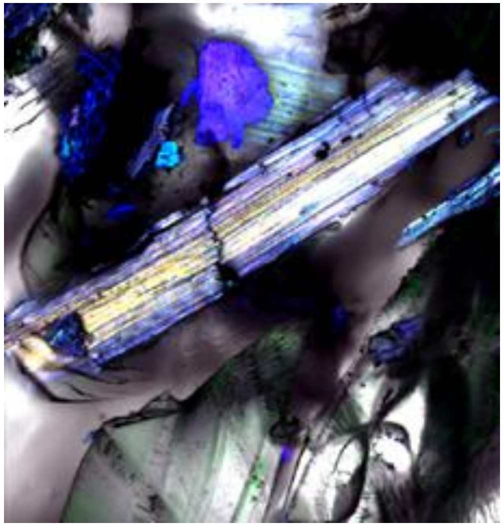
Publications, Patents, Presentations, Awards, and Commercialization

- Amber Hoover, Rachel Emerson, Daniel Stevens, Allison Ray, Jeffrey Lacey, Marnie Cortez, Courtney Payne, Robert Kallenbach, Matthew Sousek and Rodney Farris. Effect of drought on composition and bioconversion for Miscanthus, mixed perennial grasses, and switchgrass as bioenergy feedstocks. 38th Symposium on Biotechnology for Fuels and Chemicals. Baltimore, MD, April 26th, 2016.
- Allison E. Ray, Daniel Stevens, Ross Hays, Kastli D. Schaller, Amber Hoover, Chenlin Li, Ingrid Hoeger & Sunkyu Park. Development of a Blended Feedstock Strategy to Address Challenges of Feedstock Cost for Biochemical Conversion of Lignocellulosic Biomass. American Institute of Chemical Engineers (AIChE) 2015 Annual Meeting. Salt Lake City, UT; November 2015
- Kastli D. Schaller, Daniel Stevens, Gage Sowell, Dayna Daubaras, Kara G. Cafferty, Amber Hoover and Allison Ray. Comparison of Predicted and Measured Conversion Performance of Corn Stover, Switchgrass, Miscanthus, and Paper Blends Following Pretreatment and Enzymatic Hydrolysis. Idaho Falls, ID. American Institute of Chemical Engineers (AIChE) 2015 Annual Meeting. Salt Lake City, UT; November 2015
- Nathan C. Crawford, Nicholas J. Nagle, David A. Sievers, Jonathan J. Stickel and Allison E. Ray, Flowability of Biomass Solids: The Effects of Preprocessing. American Institute of Chemical Engineers (AIChE) 2015 Annual Meeting. Salt Lake City, UT; November 2015
- Vicki S. Thompson, Allison Ray, Daniel Stevens, Amber Hoover, Rachel Emerson, Suchada Ukaew, Bethany Klemetsrud, Jordan Klinger, David R. Shonnard and Dominic Eatherton. Municipal Solid Waste As a Potential Feedstock for Biochemical and Thermochemical Conversion Processes. American Institute of Chemical Engineers (AIChE) 2015 Annual Meeting. Salt Lake City, UT; November 2015
- Ingrid Hoeger, Allison Ray, Daniel Stevens, Amber Hoover, Chenlin Li, and Sunkyu Park. Property Assessment of Blended Biomass Feedstocks for Biosugar Production. American Institute of Chemical Engineers (AIChE) 2015 Annual Meeting. Salt Lake City, UT; November 2015
- Amber Hoover, Daniel Stevens, Allison E. Ray, Sunkyu Park, Ingrid Hoeger, Rachel Emerson, Sabrina Morgan and Garold L. Gresham. Predicting Bioconversion from Physical and Chemical Characteristics of Single and Blended Lignocellulosic Biomass. American Institute of Chemical Engineers (AIChE) 2015 Annual Meeting. Salt Lake City, UT; November 2015

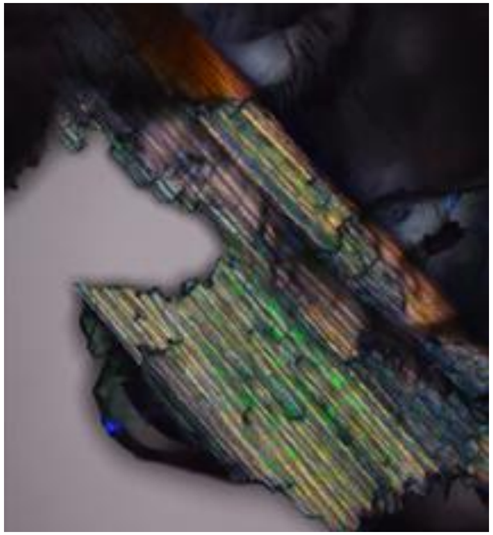
Publications, Patents, Presentations, Awards, and Commercialization

Awards

- BETO-funded research activities from WBS 2.2.1.102 (INL) awarded 1st and 3rd place in the Center for Advanced Energy Studies (CAES) ARTernative Energy contest; Spring 2016, Idaho National Laboratory, Idaho Falls, ID.



CAES ARTernative Energy Contest
1st place winner: Biomass in Blue



CAES ARTernative Energy Contest
3rd place winner:
Corn Stover Streaking

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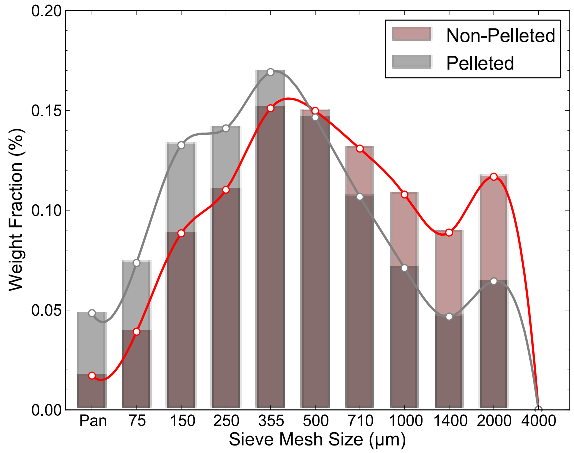
Acronym Guide

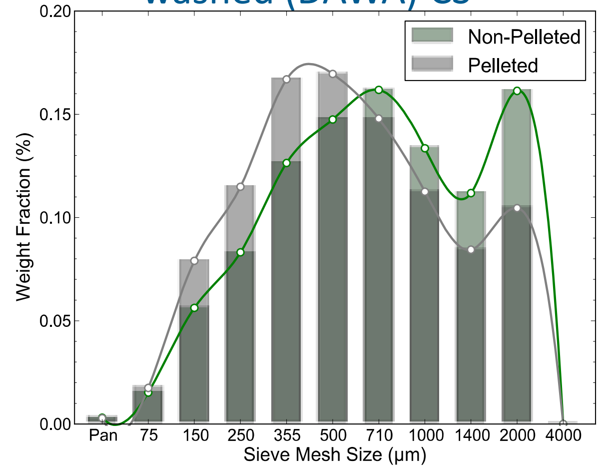
- Integrated Biorefineries (IBRs)
- State of Technology (SOT)
- Least Cost Formulation (LCF)
- Biochemical (BC)
- Feedstock Conversion Interface Consortium (FCIC)
- Biomass Feedstock National User Facility (BFNUF)
- Dilute-acid pretreatment (DAP)
- Pretreatment (PT)
- Enzymatic hydrolysis (EH)
- Base catalyzed depolymerized (BCD)
- Corn stover (CS)
- Switchgrass (SWG)
- Grass clippings (G)
- Deacetylation and mechanically refined (DMR)
- Minimum fuel selling price (MFSP)

Impact of deacetylation an acid impregnation on particle size distribution

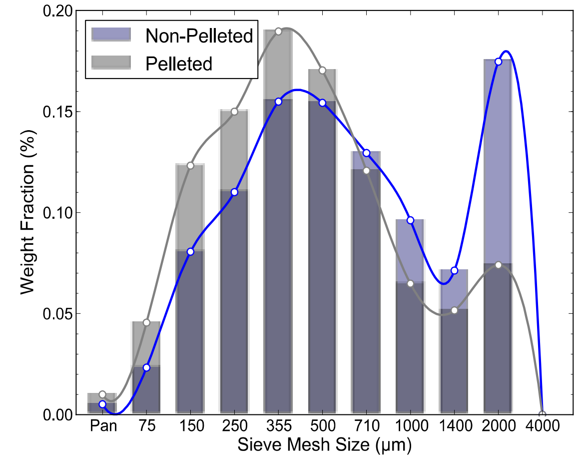
Ground CS

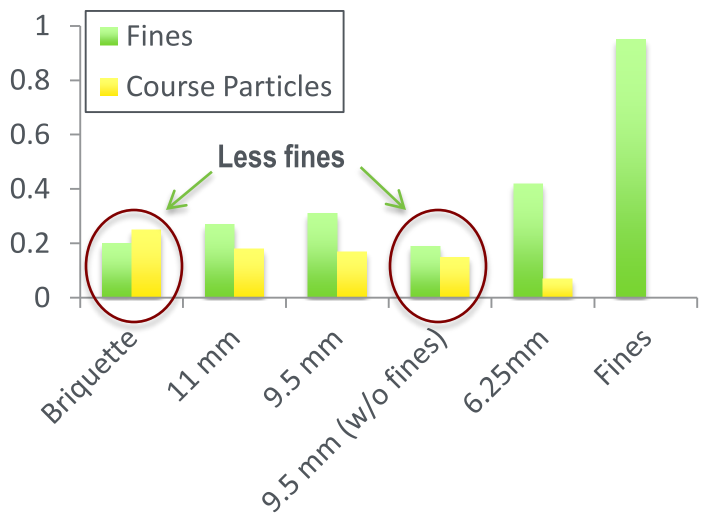


Deacetylated and washed (DAWA) CS



Acid Impregnated (AI) CS





Mass fraction of particles (500< and >2000 µm) in CS fraction and briquetted CS

- **Distribution in pelleted material is wider with lower average particle size compared to non-pelleted for CS, SWG and CS:SWG blend**
- Average particle size increases after DAWA and AI for CS, no change in SWG, the particle size n blend is average of CS and SWG

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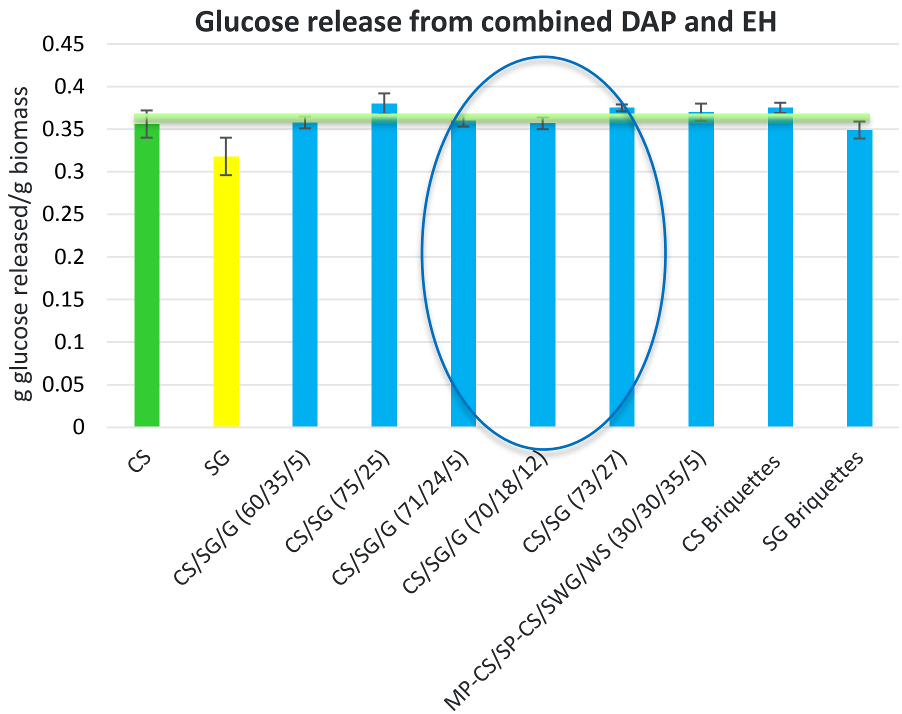
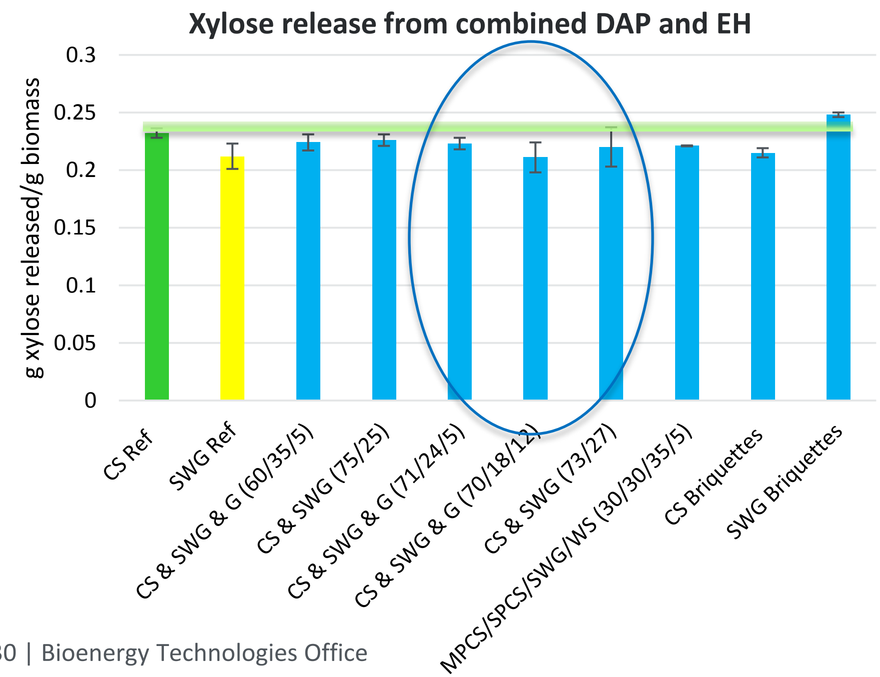
Performance Evaluation of Blends and Formats

- Characterization & performance evaluation of > 25 blends & formats (composition, lab-scale DAP & EH)
- Blends that meet cost and quality targets
  - Quality ≥59% CHO, <5% ash
  - $84/DMT





- Densified formats of had equivalent or increased glucose release
- LCF blends had sugar yields that were equivalent to CS in lab-scale screening studies

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Process schematic