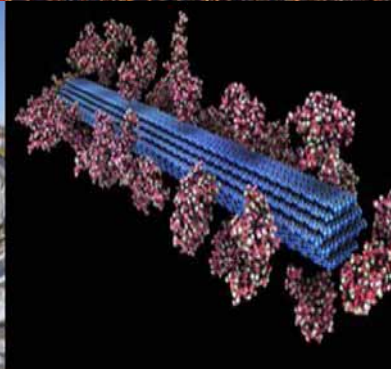




U.S. DEPARTMENT OF
ENERGY

Energy Efficiency &
Renewable Energy



1.2.2.1 Advanced Feedstock Preprocessing

March 25, 2015

Feedstock Interface

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Goal Statement

- Problem:
 - Feedstock properties are managed because they have conversion cost impacts
 - The meaning of “quality” varies with conversion pathway
 - The process impact of quality specs varies with conversion pathway
 - The cost of not meeting quality specs varies with conversion pathway
 - This is a barrier to utilizing the billion tons of biomass because there is
 - a lack of firm cost impact information for feedstock quality attributes for most conversion processes
 - little room available in feedstock cost goals for quality mitigation
 - a near-term willingness to sacrifice product yield for decreased product cost
- Goal:
 - Address biomass quality barriers by employing lowest-cost combinations of mechanical and chemical separations and formulation to produce feedstock blends that meet quality, convertibility and yield targets



Quad Chart Overview

Timeline

- Start date – 10/1/2009**
- Project end date – 9/30/2017
- Percent complete – 50%

Budget

	Total Costs FY 10- FY 12	FY 13 Costs	FY 14 Costs	Total Planned Funding (FY 15- Project End Date)
DOE Funded	\$3,800	\$1,731	\$1,469	\$1,690
This Project	\$688	\$1,232	\$1,307	\$1,690
Project Cost Share	\$0	\$0	\$0	\$0

***Current project tasks initiated on 10/1/2011.*

Barriers

- Three main barriers addressed:
 - Ft-A. Terrestrial Feedstock Availability and Cost
 - Bt-A. Biomass and Feedstock Variability
 - Tt-D. Biomass Pretreatment
- Other barriers impacted:
 - Feedstock: Ft-E, Ft-G
 - Biochemical Conversion: Bt-B, Bt-D, Bt-H
 - Thermochemical Conversion: Tt-C, Tt-G

Partners

- Collaborations
 - NREL
 - LBNL
 - SNL
 - Cascadia Consulting
 - Michigan Tech



1 - Project Overview

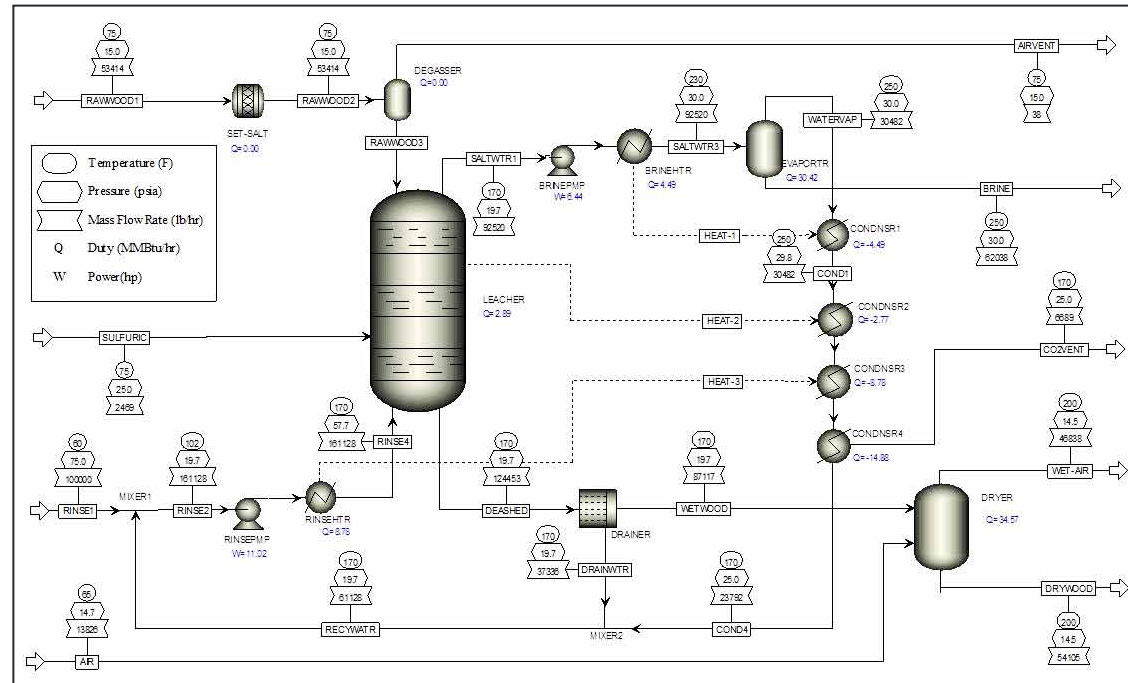
Project Objectives

- Identify and develop advanced technology solutions to meet quality requirements
 - Spanning the entire space of potential feedstocks, blend stocks, and BETO-relevant conversion technologies
 - Definition of quality and cost:benefit relationships over this space are not well characterized
- Develop, test & improve quality management technologies
 - Informed by economic analysis of quality mitigation costs
 - Order of magnitude economic estimates of the cost of not meeting a given quality parameter for a given conversion process
- Develop data-driven quality management approach
 - Combinations of existing & new technologies or strategies
 - Modification of feedstock structure to reduce feedstock logistics operations and/or their costs
 - Formulation with other feedstocks and fractions of feedstocks
 - Potential for integration with biorefinery energy balance prior reactor throat

2 – Approach (Technical)

General Approach

- Identify specific biomass properties that impact feedstock or conversion costs/yields for specific conversion platform processes
- Identify affected conversion unit operations & assess the order of magnitude cost impact
- Assess the order of magnitude cost of potential quality improvement strategies & compare with the conversion cost and yield impacts
- Collect experimental data and develop predictive models for economically promising single or multi-step strategies
- Assess the cost per ton of preprocessing by those methods for a range of feedstocks
- Integrate the combinations of preprocessing methods with feedstock formulation approaches to meet various quality specifications

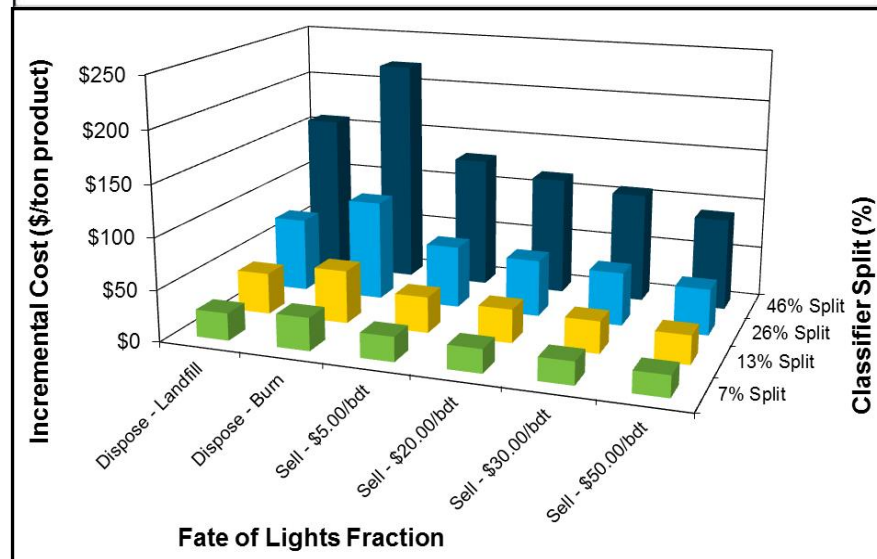
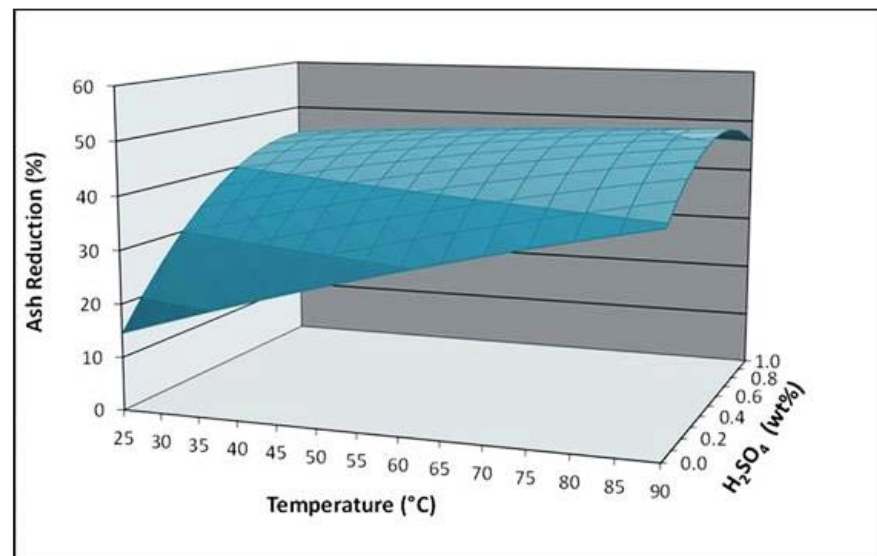


2 – Approach (Management)

- Integrated tasks have related milestones and are considered complementary rather than separate
- Schedule-Tracking Milestones and Annual Milestones monitor progress and define deliverables that document and disseminate research products
- Go/No-go Decision Points guide selection of promising preprocessing technology candidates for further development
 - Based on attainment of minimum achievable quality improvements, and
 - Order of magnitude economic comparisons for potential preprocessing technologies with potential cost/yield improvements for impacted conversion unit operations
- Samples and data for process models and conversion testing are provided as needed to Conversion Interface projects

3 – Technical Accomplishments/Progress/Results

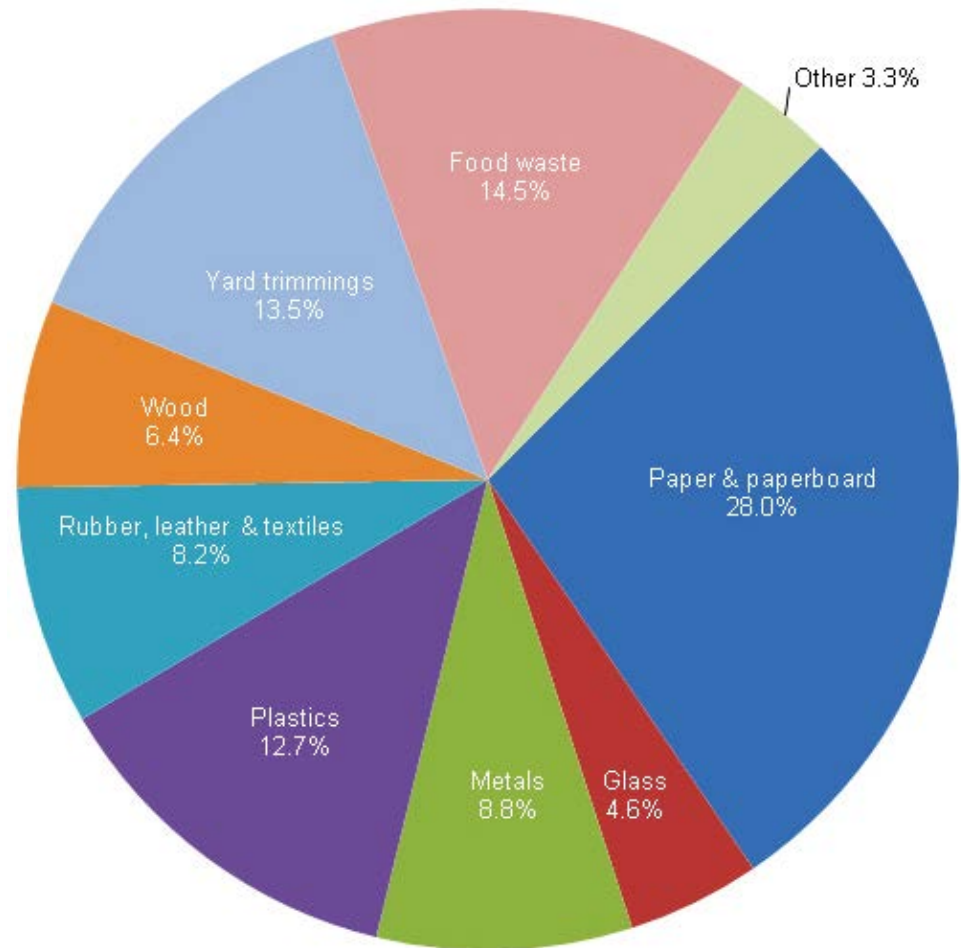
- Feedstocks: Corn stover, woody residues
- Quality focus: Total ash, Na, K, Ca, Mg and SiO₂
- Approach: Combinations of air or particle classification, leaching and blending
- Conversions (effects):
 - Uncatalyzed Fast Pyrolysis (Bio-oil yield ↓)
 - Fermentation Ethanol (Feedstock replacement ↑, equipment wear ↑, ash disposal ↑)
- Activities:
 - Data collected for mechanical & chemical separations for selected feedstocks
 - Process models developed for the options
- Product cost per ton to meet quality specifications: Estimated for raw & preprocessed blend stocks
- Currently developing estimates for combinations of methods and blending approaches to meet a range of quality specifications



3 – Technical Accomplishments/Progress/Results

Formulation – Potential low quality, low cost feedstocks

- Waste streams
 - Food wastes
 - Agriculture processing wastes – cotton gin trash, nut shells, peels, husks
 - Forestry processing wastes – thinnings, tops, branches
 - Municipal solid waste
- Wastes applicability
 - Often point sources
 - Seasonal
 - Quality issues



2011 Total MSW Generation by material, 250 Million Tons before recycling (US EPA, Municipal Solid Waste in the United States, 2011 Facts and Figures, EPA530-R-13-001, 2013)

3 – Technical Accomplishments/Progress/Results

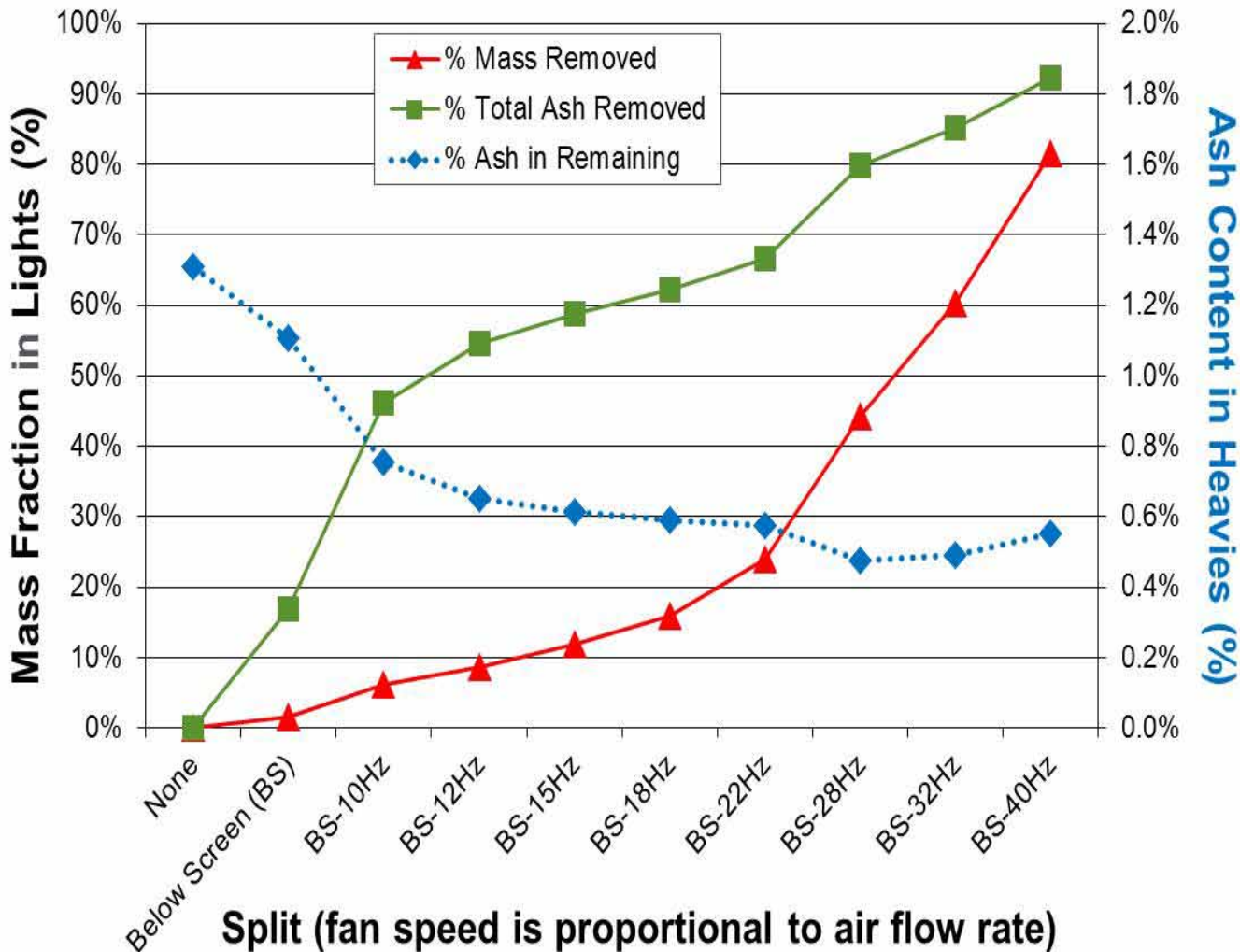
Loblolly Pine Residue – Gross Tissue Hand Separation

- Tissue fractions hand-separated from 2-in. chipped loblolly pine forest thinnings
- Distribution of ash as a function of tissue
- Highest ash content is in needles, bark and twigs
- Suggests that an analogous mechanical separation like air classification could be used for management of total ash

Separable Fraction	% of Plant Mass	Ash Content (%)
Needles	0.69	2.21
Bark	3.64	1.42
Twigs	--	1.09
Branches	--	0.57
Fines	20.2	--
Cambium with bark	17.6	0.38
Sapwood	57.9	0.31
Composite	100	1.27

3 – Technical Accomplishments/Progress/Results

Loblolly Pine Thinnings Air Classification Results (2-in. chips)

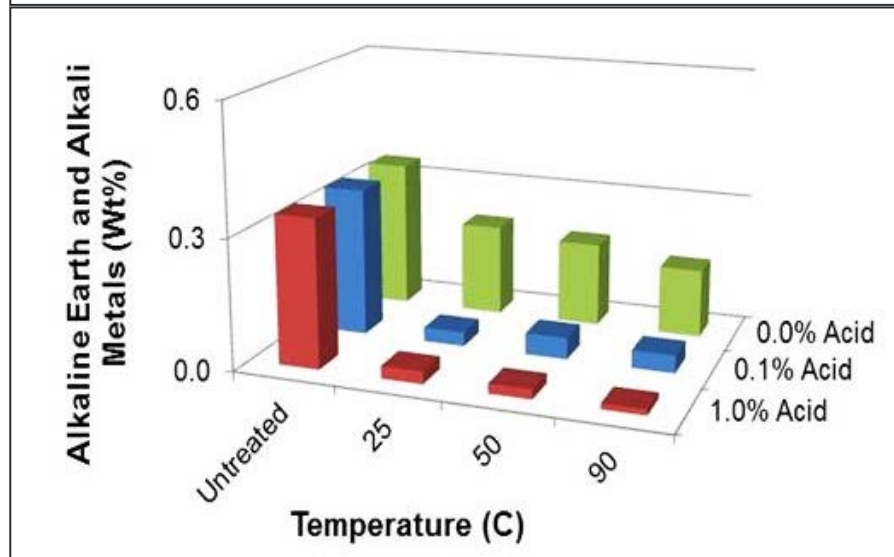
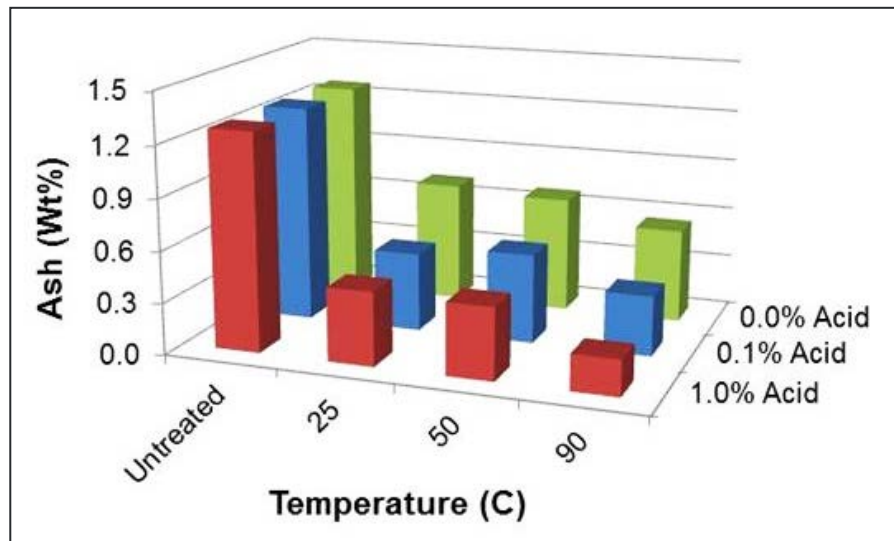


- **Blue curve:** Can successfully partition high ash fractions from those meeting 0.9% ash spec
- **Green curve:** Nearly half the ash can be partitioned into the lights split with fan speed at 15 Hz
- Ash species removed are those from non-physiological sources
 - SiO_2 , Al_2O_3 , TiO_2 , Fe_2O_3 , Na_2O

3 – Technical Accomplishments/Progress/Results

Equilibrium Dilute Acid Leaching Studies for Loblolly Pine Thinnings

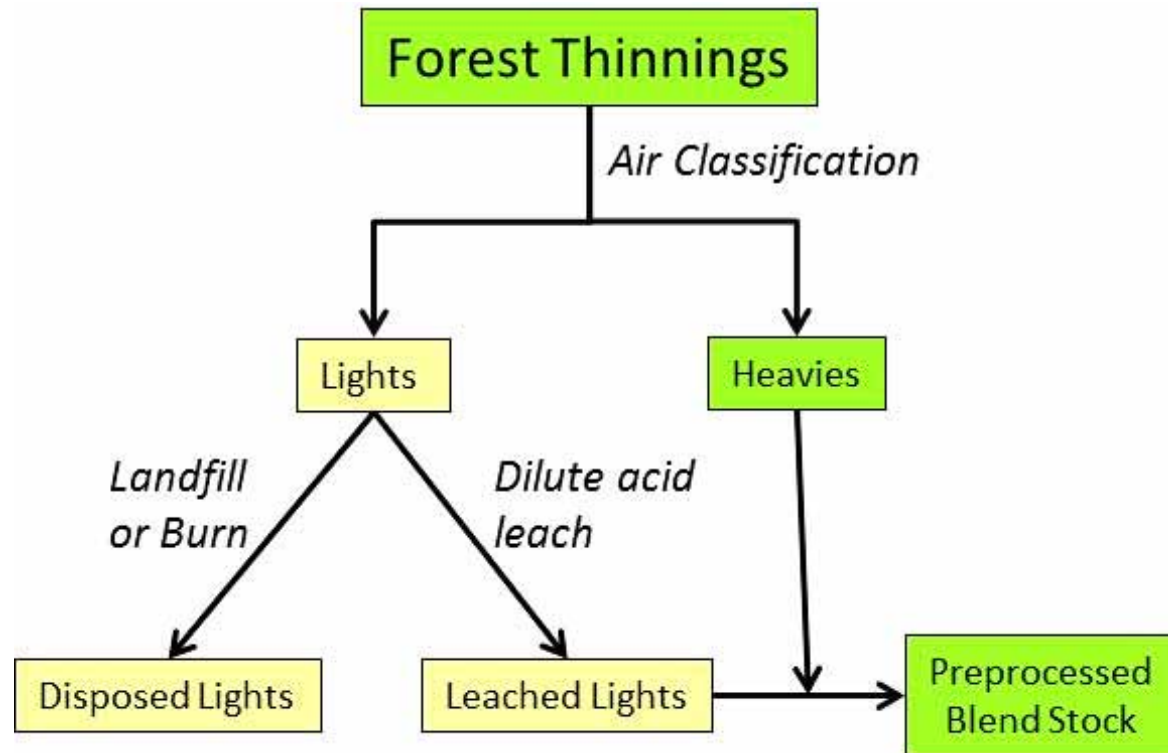
- Ash removal in general increased with higher temperature and lower pH
- Relative removal of physiological ash species (Ca, Mg, Na, K) was dependent on pH
- Similar trends observed for leaching of other pine residues
- Percentages of ash species removed via dilute acid leaching of pine residues were similar for corn stover as well
 - Suggests that physiological sources of ash species may be similar among these feedstocks
 - Equilibrium leaching can likely be modeled for prediction of efficacy with a wider range of feedstocks



3 – Technical Accomplishments/Progress/Results

Example Analysis

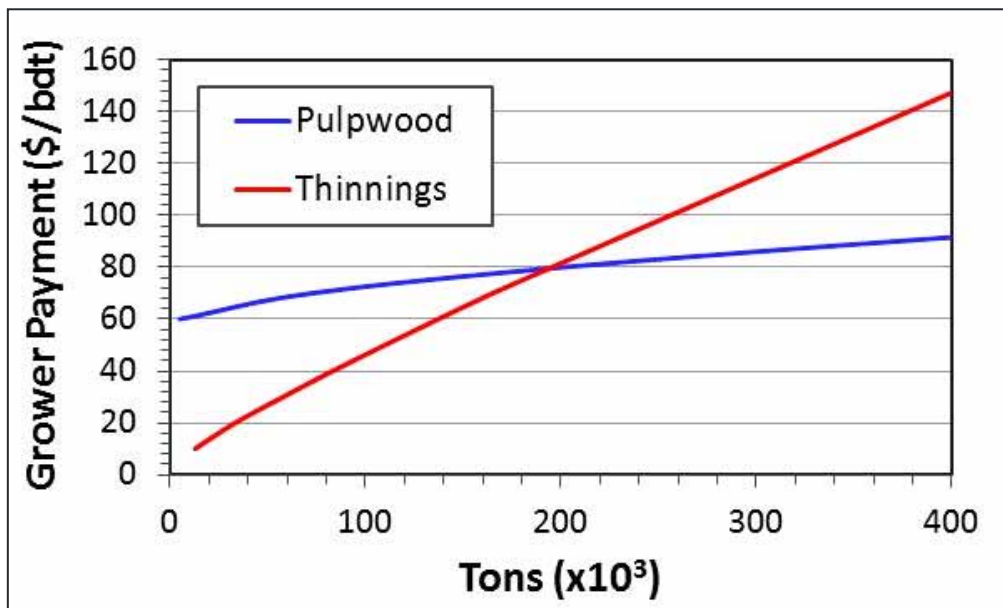
- Two choices of preprocessing treatments: air classification & dilute acid leaching
- Lights fraction disposed or leached
- Leached lights fraction solids dried and recombined with heavies fraction
- Blend stocks from thinnings shown in **green**
- Pulpwood also a blend stock, subject to availability limits
- Linear optimization model used to predict lowest cost blends based on blend product cost



3 – Technical Accomplishments/Progress/Results

Assumptions

- Depot producing ~200,000 bdt/y (25 tph, 340 operating days/y)
- Input feedstock costs include grower payment + existing processing
 - Grower payment: ORNL supply curves
 - Processing:
 - Processing costs taken from the 2014 INL Woody Feedstock Design State of Technology (SOT)
 - Includes: Storage, Handling, Drying, Chipping, Grinding, Pelleting, Pellet Storage, and Transportation
- Costs estimated for additional depot operations (air classification, leaching)
- Incremental cost per ton of product includes added cost of replacement feedstock

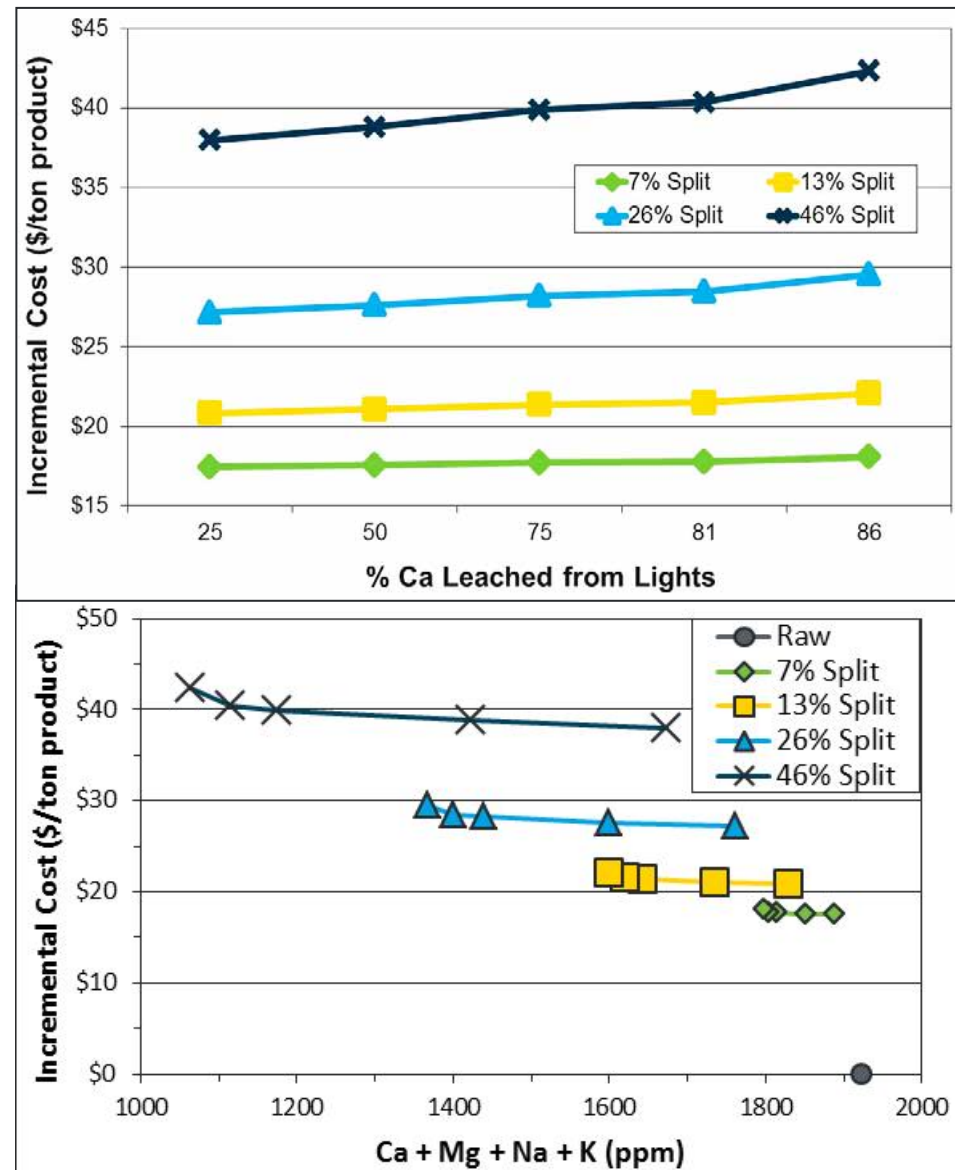


Feedstock Logistics Process	Pulpwood (\$/bdt)	Thinnings (\$/bdt)
Harvest and collection	\$16.01	\$0.00
Landing preprocessing/sorting	\$0.00	\$8.73
Transportation	\$19.37	\$19.37
Preprocessing	\$37.21	\$18.24
Storage	\$1.96	\$1.96
Handling	\$1.90	\$1.90
Total	\$76.45	\$50.20

3 – Technical Accomplishments/Progress/Results

Effect of Amount of Thinnings Leached on Added Cost

- Example for gross input thinnings cost of \$140.20/bdt
- Incremental (additional) costs are insensitive to leaching time when small amounts of feedstock are treated
- Because leaching was sized for maximum possible throughput of 25 tph
 - Capital outlay dominates over operating costs for small amounts
 - Operating costs become more important for larger amounts
- For air classified thinnings, leaching larger amounts is necessary to have a significant impact on a spec based on Ca + Mg + Na + K



3 – Technical Accomplishments/Progress/Results

- Two starting blend stocks: Pulpwood and Thinnings
- Eight cases considered
 - Pulpwood fixed at annual availabilities from 0-200,000 bdt
- Additional blendstocks generated via air classification and leaching
 - Four from air classification (heavies from 7%, 13%, 26% and 46% splits)
 - Four from equilibrium leaching of the lights from the 7%, 13%, 26% and 46% splits

Blend Stock	Total ash (%)	Ca + Mg + Na + K (ppm)
Pulpwood	0.24	1,028
Thinnings	1.27	1,923
Thinnings, 7% Split, Heavies	0.61	1,891
Thinnings, 7% Split, Heavies + Leached Lights	0.86	1,796
Thinnings, 13% Split, Heavies	0.49	1,776
Thinnings, 13% Split, Heavies + Leached Lights	0.77	1,600
Thinnings, 26% Split, Heavies	0.46	1,715
Thinnings, 26% Split, Heavies + Leached Lights	0.70	1,367
Thinnings, 46% Split, Heavies	0.46	1,730
Thinnings, 46% Split, Heavies + Leached Lights	0.63	1,064

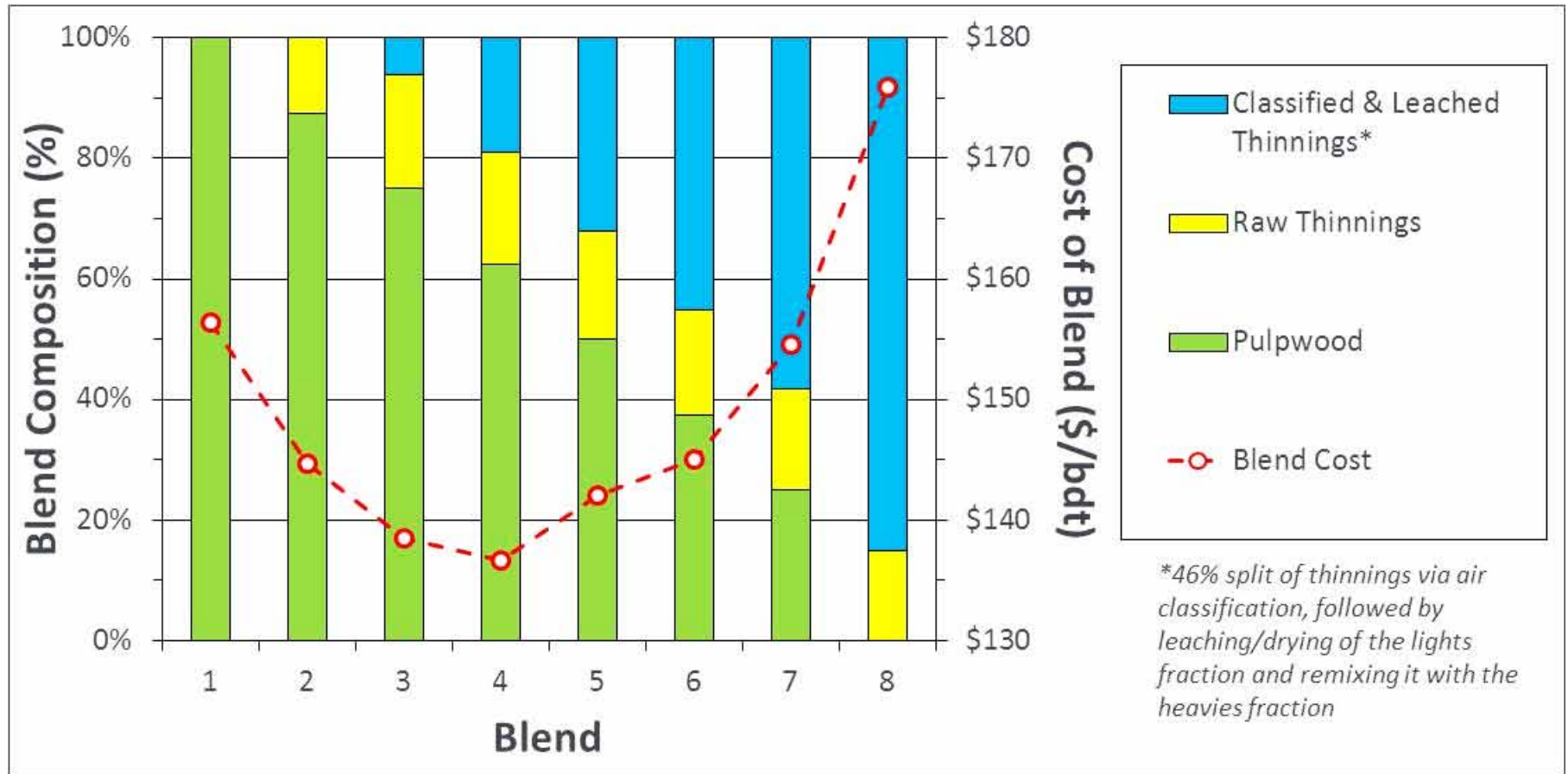
3 – Technical Accomplishments/Progress/Results

- Blends were optimized for lowest product cost, with two quality specifications
 - 0.9% total ash spec
 - an assumed 1,200 ppm Ca+Mg+Na+K spec
 - 1,200 ppm is ~20% > 1028 ppm, the average of 8 lit. values for pulpwood with RSD = 28%
- Linear optimization model used iteratively with the cost models to predict lowest cost blends on a bdt blend product basis

Blend	Pulpwood			Raw Thinnings		
	Amount (×10 ³ bdt)	Grower Payment (\$/bdt)	Cost (\$/bdt)	Amount (×10 ³ bdt)	Grower Payment (\$/bdt)	Cost (\$/bdt)
1	200	\$80.00	\$156.45	0	\$0.00	\$0.00
2	175	\$78.00	\$154.45	25	\$25.00	\$75.20
3	150	\$75.00	\$151.45	50	\$40.00	\$90.20
4	125	\$73.00	\$149.45	75	\$46.00	\$96.20
5	100	\$72.00	\$148.45	100	\$60.00	\$110.20
6	75	\$70.00	\$146.45	125	\$65.00	\$115.20
7	50	\$70.00	\$146.45	150	\$75.00	\$125.20
8	0	\$0.00	\$0.00	200	\$90.00	\$140.20

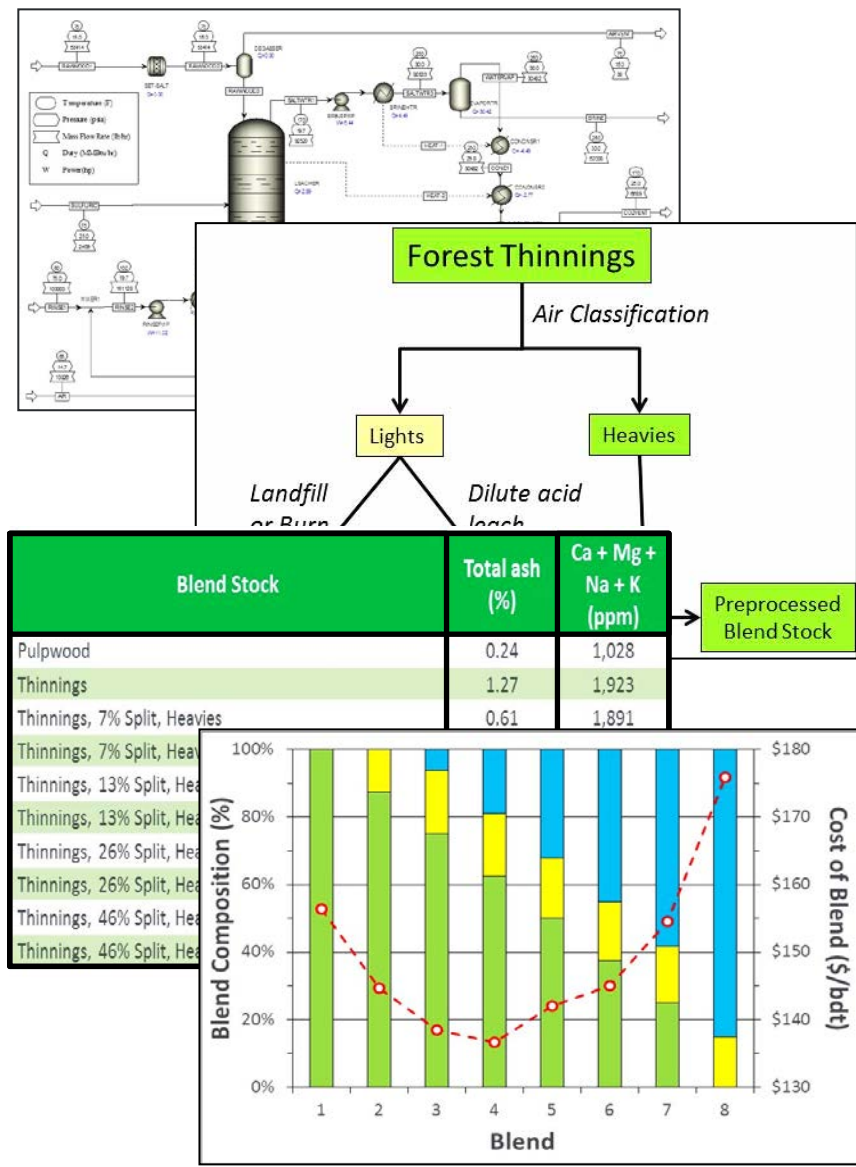
3 – Technical Accomplishments/Progress/Results

Goal: Address biomass quality barriers by employing lowest-cost combinations of mechanical and chemical separations and formulation to produce feedstock blends that meet quality, convertibility and yield targets



4 – Relevance

- Directly relevant to BETO, MYPP goals, and overall bioenergy industry
 - Characterizing and studying feedstock composition
 - Defining feedstock quality characteristics that can be cost effectively managed prior to conversion
 - Developing feedstock quality specifications based on cost:benefit
 - Developing cost effective strategies to produce feedstocks at quality specs
- Assigns unambiguous costs to quality
- Precursor to the development of feedstock commodity specifications
- Develops a data-driven feedstock quality management approach that maximizes feedstock utilization and conversion yield while minimizing overall product cost





5 – Future Work

- Estimate effects and magnitudes of manageable feedstock characteristics on costs and yields for a range of conversion technologies
 - Convertibility effects of feedstock preprocessing for range of conversions
 - Expand impact using strategic collaborations (Labs & other partners)
- Perform economic assessments for potential preprocessing methods that affect the identified feedstock characteristics
- Upcoming key milestones
 - Go/No-Go Milestone on fractionation of high and low ash fractions of woody feedstocks using air classifications (3/31/2015)
 - Annual Milestone on the formulation of mechanically separated fractions of herbaceous, woody and MSW feedstocks (9/30/2015)
 - Milestone to submit a manuscript that details correlations between feedstock quality and conversion yields (9/30/2015)
- The Go/No-Go Decision Point criterion has been met (Go)
 - Air classification will be used as a preprocessing tool to generate high and low quality feedstock fractions

Summary

- Objectives of Advanced Feedstock Preprocessing
 - Identify and develop advanced technology solutions to meet quality requirements
 - Develop, test & improve quality management technologies
 - Develop a data-driven feedstock quality management approach that spans the range of potential feedstocks and conversion processes
- Goal: Address biomass quality barriers by employing lowest-cost combinations of mechanical and chemical separations and formulation to produce feedstock blends that meet quality, convertibility and yield targets
- Recent challenges and what was learned
 - Lack of firm cost impact information for feedstock quality attributes
 - Little room available in feedstock cost goals for quality mitigation
 - Near-term willingness to sacrifice product yield for decreased product cost
- Estimated impact on current State of Technology or on bioenergy industry
 - Enables the assignment of unambiguous costs to quality specifications
 - Precursor to the development of feedstock commodity specifications



Responses to Previous Reviewers' Comments

- Comment: “Net cost reduction and improved quality; plan to do optimization, but not really evident from presentation or answers to questions.”
 - Response: This project was new in 2012 and we were still trying to effectively target what we were doing to be of the highest relevance. The optimization strategy should be clear from the 2015 Peer Review.
- Comment: “Considerable work remains to be completed in this project, but funding is apparently assured for several more years. The future work described was appropriate for addressing the critical success issues, but was quite general in nature. PI should have a more detailed plan for each of the four areas.”
 - Response: We have worked hard to integrate the individual areas within this project into a single multi-faceted plan to address quality management. Further, much has been done to develop a clear approach to defining cost:benefit relationships for quality management that are based on conversion cost impacts as well as biofuel yields.

Publications, Patents, Presentations, Awards, and Commercialization

Publications of work performed on this project

- Impact of mixed feedstocks and feedstock densification on ionic liquid pretreatment efficiency. J. Shi, V.S. Thompson, N.A. Yancey, V. Stavila, B.A. Simmons, S. Singh. *Biofuels* (2013), 4(1), 63-72.
- Chemical preconversion: Application of low-severity pretreatment chemistries for commoditization of lignocellulosic feedstock. D.N. Thompson, T. Campbell, B. Bals, T. Runge, F. Teymouri, L. Ovard. *Biofuels* (2013), 4(3), 323-340.
- Ash reduction strategies in corn stover facilitated by anatomical and size fractionation. J.A. Lacey, R.M. Emerson, T.L. Westover, D.N. Thompson. Submitted to *Biomass and Bioenergy*, in review.
- Removal of introduced inorganic content from chipped forest residues via air classification. J.A. Lacey, J.E. Aston, T.L. Westover, R.S. Cherry, D.N. Thompson. Submitted to *Fuel*.

Manuscripts in preparation

- Selective recovery of solubilized organics and silica from the alkaline leachate of corn stover. J.E. Aston, J.A. Lacey, D.N. Thompson. In preparation for submission to *Biofuels, Bioproducts and Biorefining*.
- Effect of dilute-acid and dilute-alkaline preconversion on conversion-relevant ash species in corn stover. J.E. Aston, T.L. Westover, D.N. Thompson. In preparation for submission to *Fuel*.

Acknowledgements

Project Task Leads & Teams

- Dr. Jeffrey A. Lacey – Mechanical Separations Lead
 - Karen Delezene-Briggs
 - Seth Ashby
 - Melanie Lynn Powell
- Dr. John E. Aston – Chemical Separations and Preconversion Lead
 - Sandy Fox
 - Seth Ashby
 - Melanie Lynn Powell
- Dr. Vicki S. Thompson – Formulation Lead
 - Sandy Fox
 - Seth Ashby

Ancillary Information Slides for Reviewers



3 – Technical Accomplishments/Progress/Results

Key Accomplishments – FY13

- Demonstrated mechanical cob removal from ground corn stover
- Milestone Report on mechanical fractionation technologies for quality improvement
- Procured a Chemical Preconversion System useful over wide range of conditions
- Two Milestone Reports on the development of an analysis tool to identify the highest impacts of chemical preconversion in the feedstock supply chain
- Exceeded milestone metric of 30% ash reduction by leaching vs. untreated stover
- Published a peer reviewed manuscript with MBI and U. Wisconsin on the potential for application of chemical preconversion to enable commoditization of biomass feedstocks and industry expansion
- Two milestones met on delivery of blended feedstock to LBNL on a collaborative project
- Milestone Report demonstrating the value of formulation in the feedstock supply chain
- Case Study of regional MSW generation/composition for least cost formulation model
- Published a peer reviewed manuscript with LBNL on the impact of blended feedstocks and feedstock densification on ionic liquid pretreatment efficiency

3 – Technical Accomplishments/Progress/Results

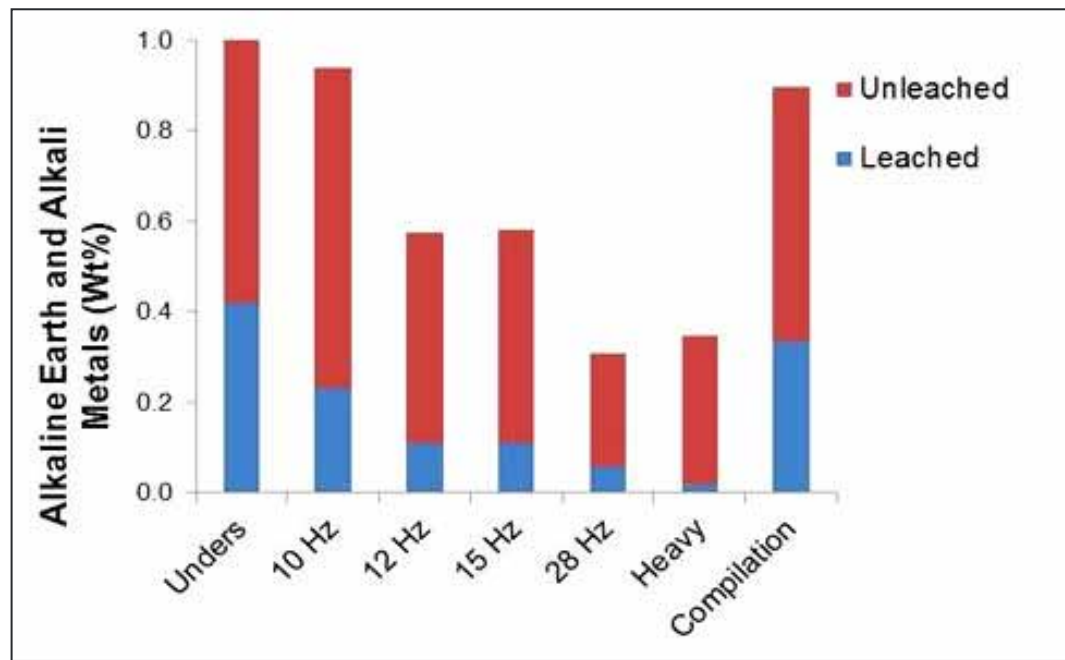
Key Accomplishments – FY14

- Anatomical separation opportunities shown for ash for corn stover and pine residues
- Air classification of chipped woody residues shown promising for removal of soil ash
- Maximum achievable reduction of ash via leaching or extraction determined as a function of solid loadings, catalyst loadings, and temperature for corn stover and pine residues
- Feasibility of fractionating silica & lignin from alkaline extract of corn stover shown
- Non-recyclable paper waste and yard waste examined to date for the Biochemical conversion pathway
- Paper waste blended with corn stover shown to have no negative impacts on sugar yields after enzymatic hydrolysis
- Studies with MSW processors in Washington State and Alberta initiated to assess the variation of a variety of waste streams over time and how waste materials impact other conversion processes

3 – Technical Accomplishments/ Progress/Results

Equilibrium Dilute Acid Leaching Studies for Air Classifier Light Fractions of Thinnings

- Select fractions of air classified material can be leached
- Leaching effectively lowered the ash content and concentrations of specific ash components
- Leaching select fractions may allow for the recombination of leached material with unleached fractions such that the entire feedstock meets a spec after leaching a relatively small amount of it



3 – Technical Accomplishments/Progress/Results

Tissue Separation Results for Corn Stover

- Tissue fractions hand-separated from baled multi-pass corn stover
- Obvious sources of soil were not collected
- Physiological ash for corn stover typically is 3-4 wt%
- Highest ash content is in leaves and sheaths
- Suggests that mechanical separation of these fractions could potentially be used for management of total ash depending on their grinding characteristics

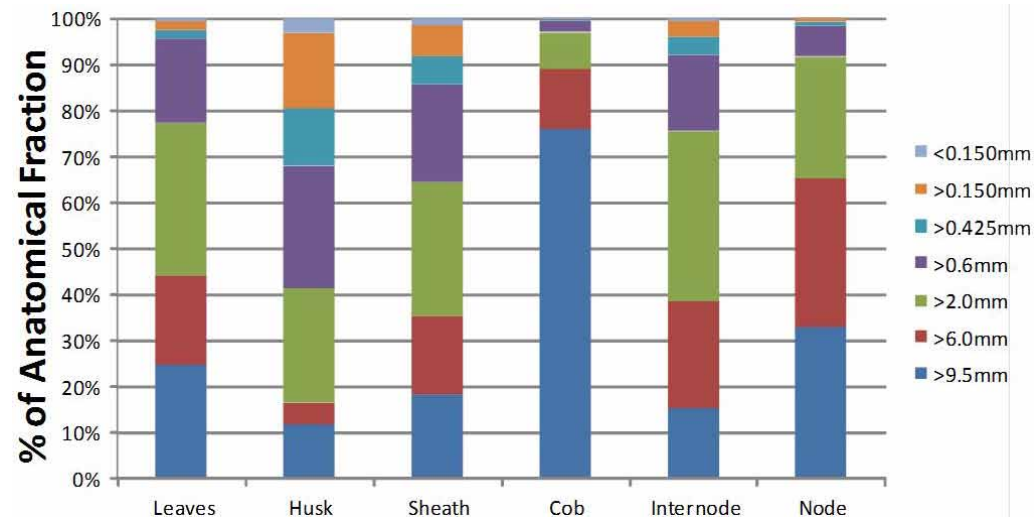
Tissue Fraction	% of Plant Mass	Ash Content (%)
Leaves	20	10.4 ± 0.5
Sheaths	10	6.9 ± 0.2
Nodes	10	4.0 ± 0.3
Husk	10	3.7 ± 0.2
Internodes	30	3.5 ± 0.2
Cobs	20	1.5 ± 0.2
Composite	100	4.9 ± 0.1

3 – Technical Accomplishments/Progress/Results

Selected Size Separation Results for Corn Stover

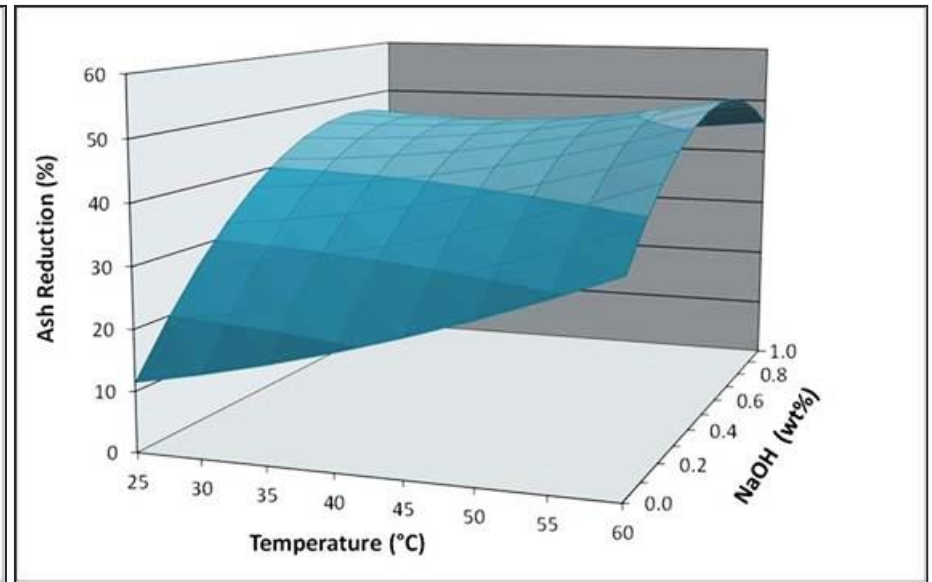
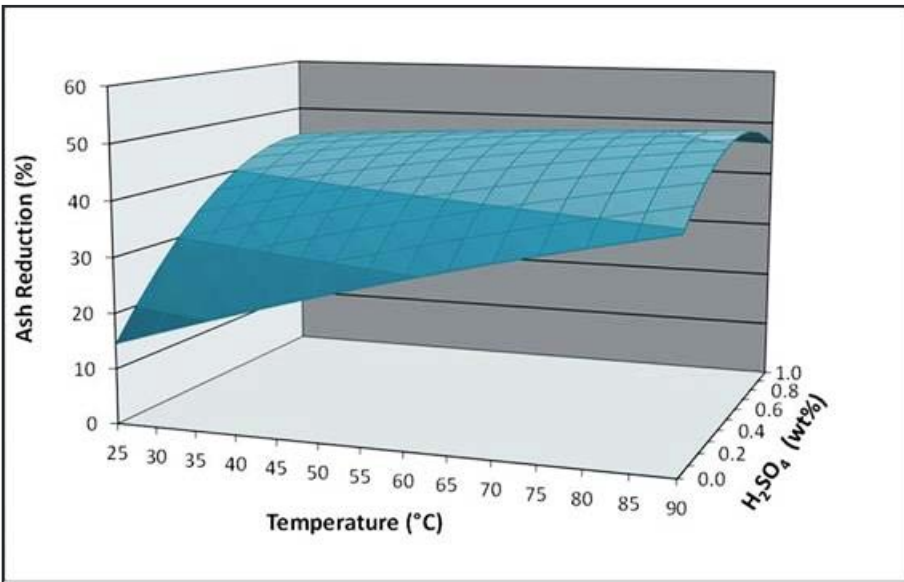
- Baled multi-pass corn stover Wiley-milled to pass a ¾-in. screen
- Ground stover then sieved
- Highest ash content is in the two smallest particle size fractions
- Bulk of the partitioned ash from non-physiological sources
- Further testing was done to identify size distribution of similarly ground and sieved individual corn stover tissues

Size Fraction (mm)	% of Plant Mass	Ash Content (%)
≥ 9.5	31.0 ± 4.2	3.5 ± 0.4
≥ 6.0	18.8 ± 3.7	4.6 ± 0.9
≥ 2.0	27.5 ± 1.4	5.1 ± 0.5
≥ 0.6	14.4 ± 6.0	5.9 ± 0.2
≥ 0.425	3.5 ± 0.9	4.8 ± 0.7
≥ 0.150	3.9 ± 0.9	7.5 ± 2.5
< 0.150	0.8 ± 0.2	22.5 ± 10.8
Composite	99.9 ± 8.4	4.8 ± 0.6



3 – Technical Accomplishments/Progress/Results

Response Surface Plots Developed From Comprehensive Corn Stover Leaching/Extraction Studies

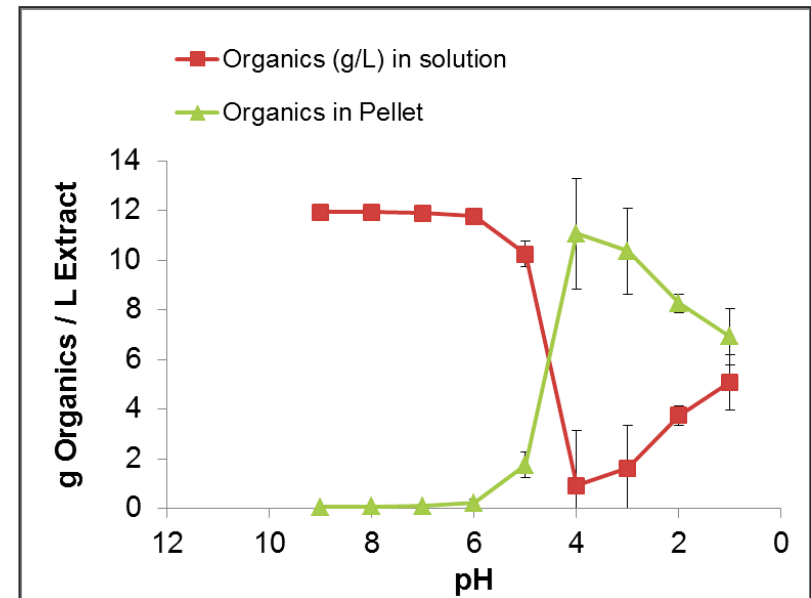
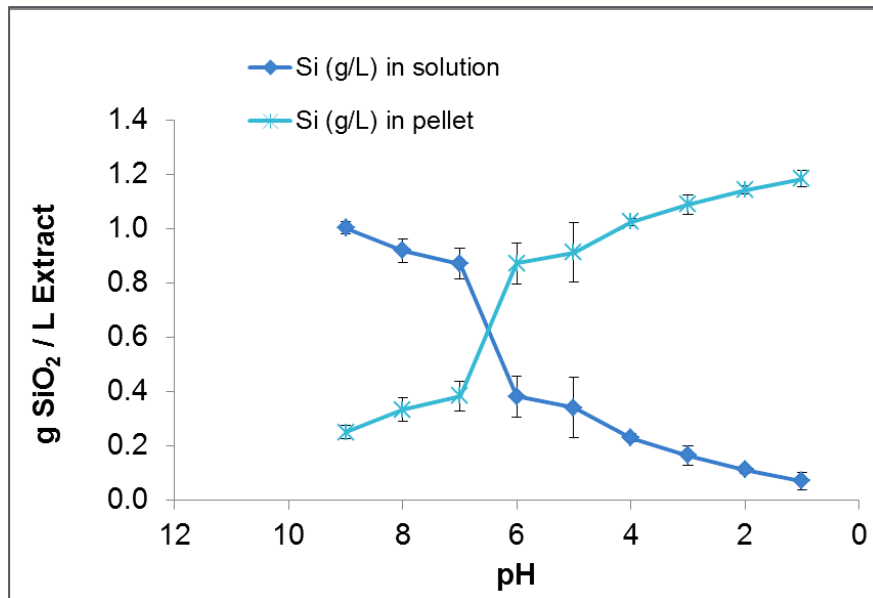


- Power series fits to data
- Equilibrium ash reduction generally increased with temperature & acid concentration

- Similar patterns with alkaline extraction
- Overall ash reduction is higher for alkali even at lower temperatures due to silica dissolution

3 – Technical Accomplishments/Progress/Results

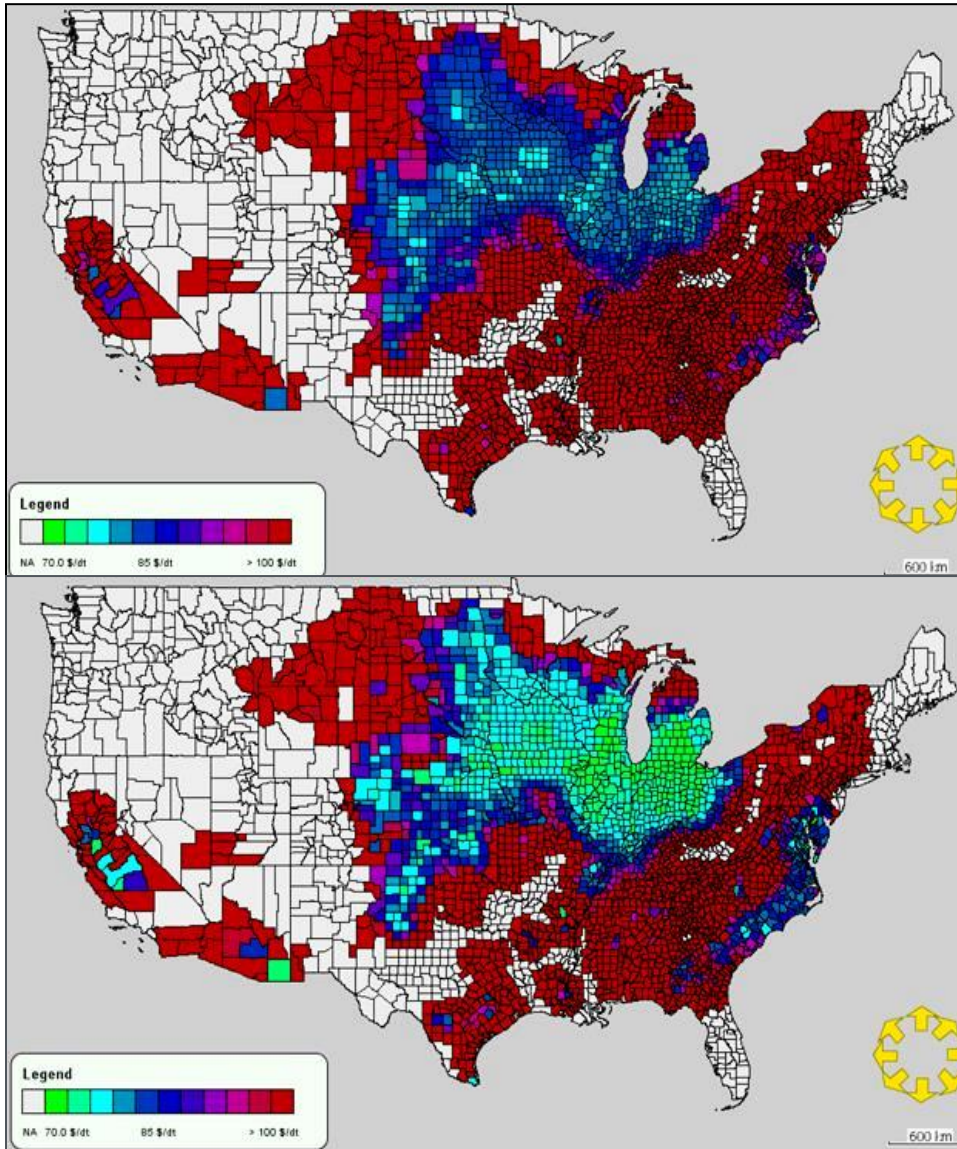
Selective Precipitation and Recovery of Material Solubilized During Alkaline Extraction of Corn Stover



- Ability to selectively precipitate silica versus solubilized organics
- Silica precipitates between pH 7 and 5
- Organics precipitate between pH 5 and 4
- Represents a possible tool for the recovery of value products to decrease the final feedstock → conversion costs

3 – Technical Accomplishments/Progress/Results

Effect of Blend Utilization on Feedstock Costs



- Corn stover only meets cost targets in high production areas
- Corn stover-based biorefineries would be limited to a few areas in the Midwest
- Replacing 20% of corn stover with MSW results in most of Midwest meeting cost targets
- Areas outside of the Midwest have sufficient availability to support biorefineries

3 – Technical Accomplishments/Progress/Results

Paper Waste Compositional Analyses

Samples	Lignin (%)	Glucan (%)	Xylan (%)	Extractives (%)	Ash (%)
Corrugated Cardboard	18.0	59.2	8.7	2.3	3.3
Glossy Cardboard	14.8	50.8	8.1	1.7	12.7
Office Paper	7.2	60.1	10.0	2.1	11.0
Glossy Paper	11.1	43.3	7.5	1.0	25.1
Corn Stover	n/a	33.3	20.3	n/a	n/a
80:20 Corn Stover and Paper Mix	14.1	39.0	18.5	5.3	5.5

n/a not available

3 – Technical Accomplishments/Progress/Results

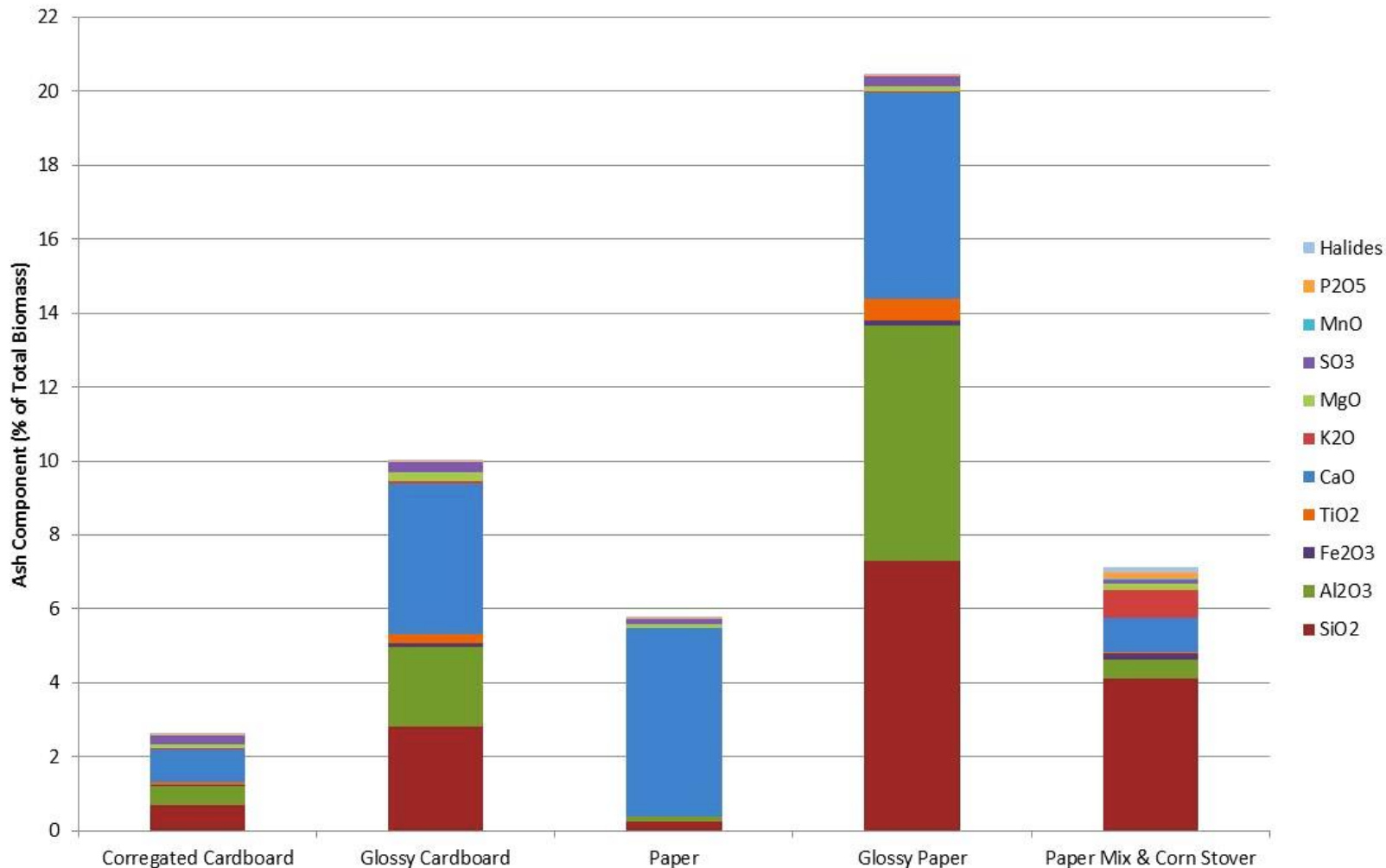
Yard Waste Compositional Analyses

Samples	Lignin (%)	Glucan (%)	Xylan (%)	Extractives (%)	Ash (%)	Protein (%)
Grass (ID) fresh	16.7	19.6	8.5	21.6	12.8	10.2
Grass (FL) fresh	11.7	15.3	6.3	23.6	12.5	10.3
Grass (AZ) dormant	18.9	20.4	13.8	15.0	11.8	n/a
Composted grass (ID)	18.1	22.8	10.3	21.1	12.6	10.3
Pine cones	42.8	18.6	4.4	10.9	6.3	2.9
Christmas trees	33.6	21.3	4.0	19.9	2.9	n/a
Tree leaves	14.2	15.1	4.8	32.0	7.2	10.2

n/a not available

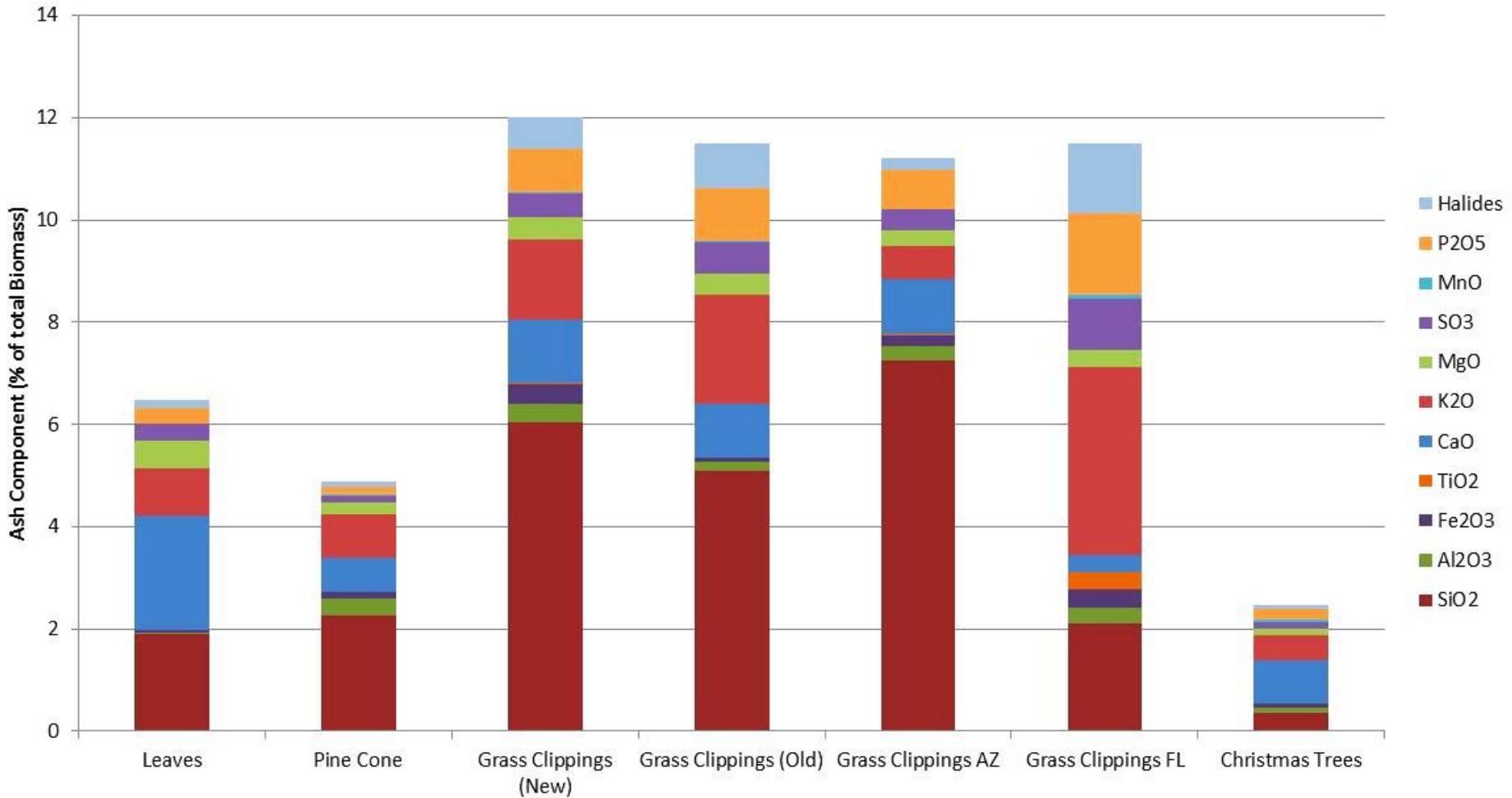
3 – Technical Accomplishments/Progress/Results

Ash Composition – Paper Waste



3 – Technical Accomplishments/Progress/Results

Ash Composition – Yard Waste



3 – Technical Accomplishments/Progress/Results

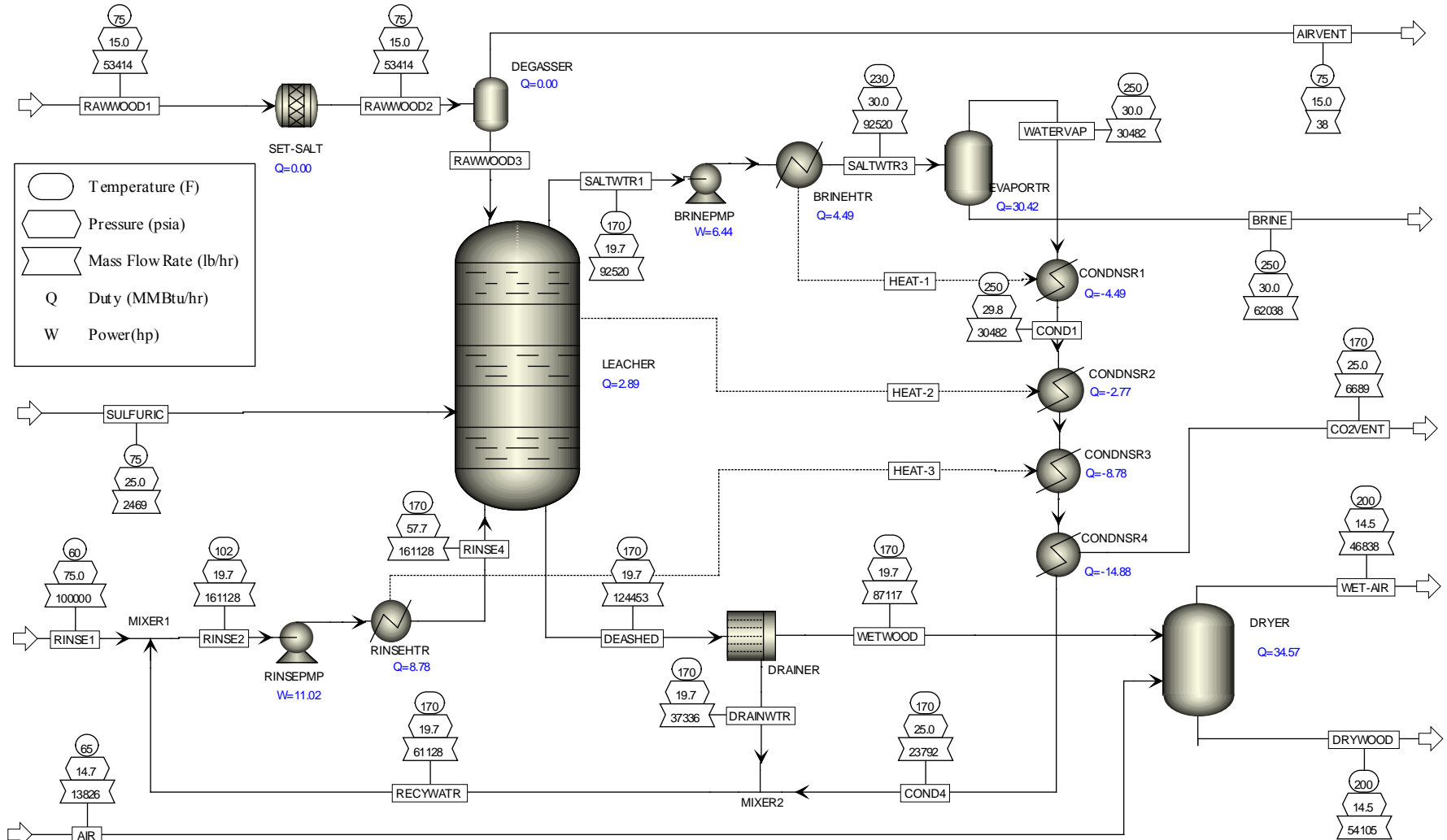
Acid Pretreatment and Enzymatic Hydrolysis – Yard Waste

Samples	% glucan yield	% xylan yield
Grass (ID) fresh	83.6	63.2
Grass (FL) fresh	145	79.1
Grass (AZ) dormant	62.8	37.4
Composted grass (ID)	80.9	60.5
Pine cones	10.7	25.9
Christmas trees	9.0	31.2
Tree leaves	51.0	26.6
Corn stover control	71.1	73.6

- Dionex ASE 350 Accelerated Solvent Extractor
- 10% solids loading
- 1% H₂SO₄ (w/w)
- 130°C for 7 minutes
- Severity (Log R₀) = 1.73
- Novozyme CTec2 and HTec2
- 50°C and 5 days

3 – Technical Accomplishments/Progress/Results

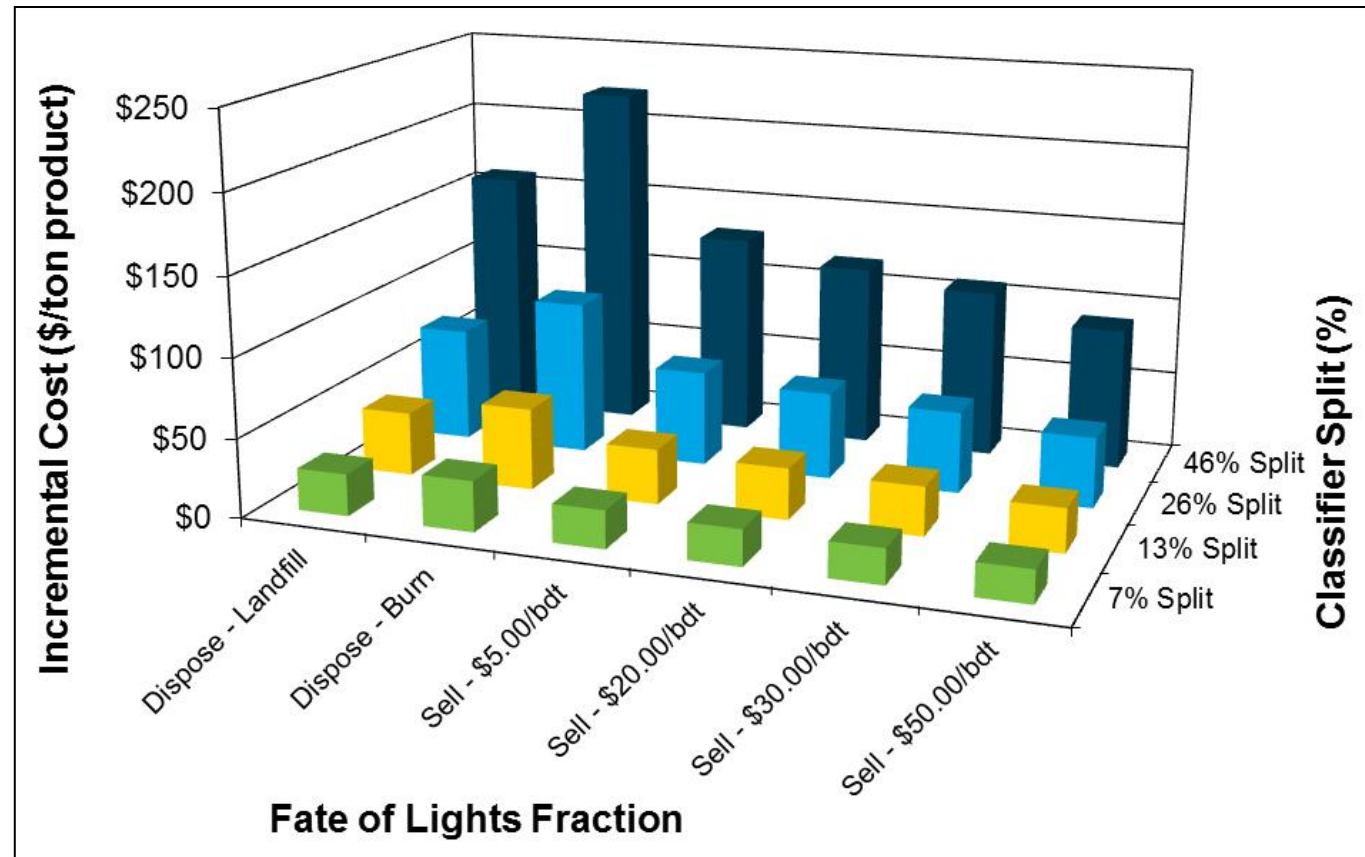
Aspen Plus Process Model – Dilute Acid Leaching of Biomass



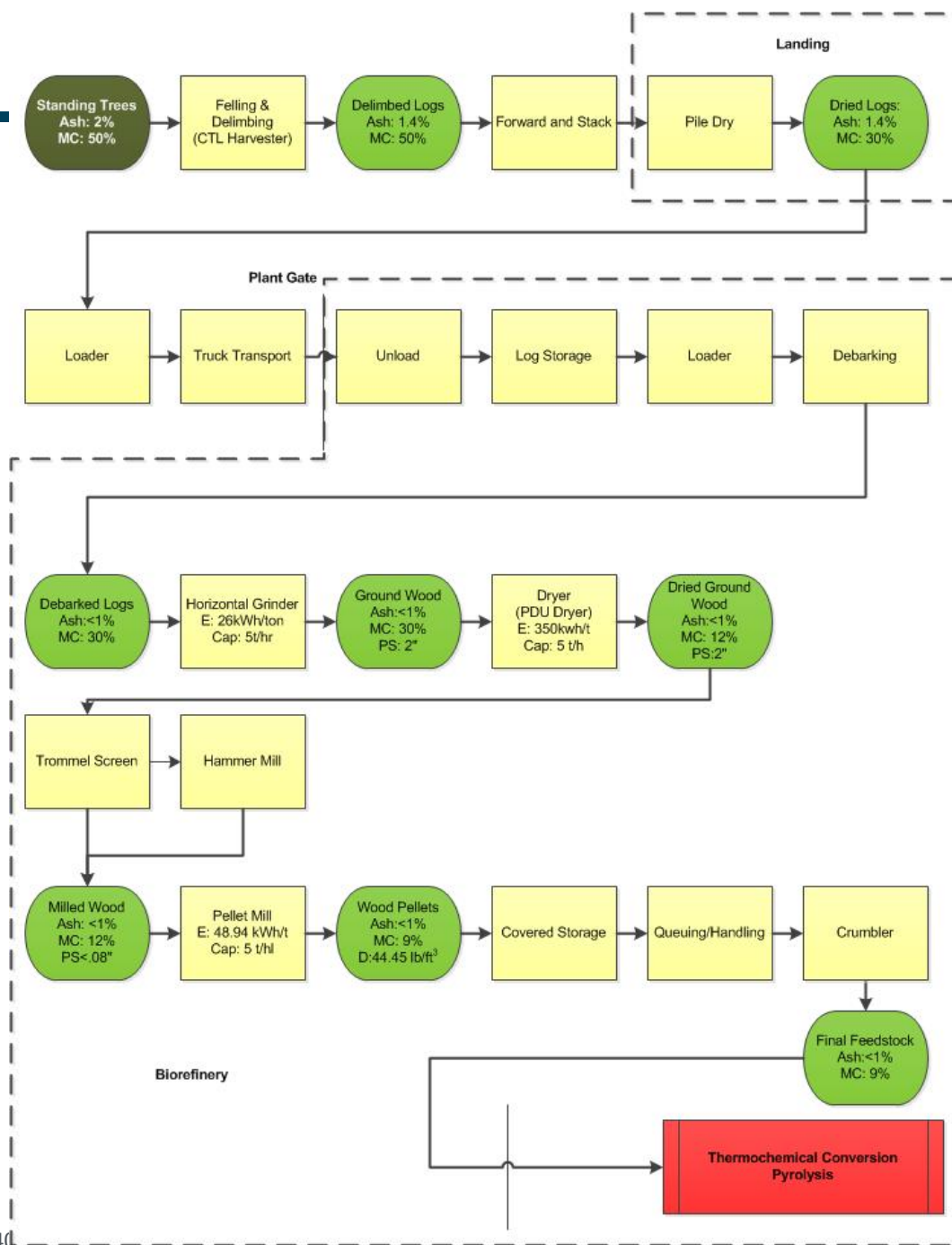
3 – Technical Accomplishments/Progress/Results

Effect of Co-Product Value on Non-Leaching Options for Light Fraction

- Example for input thinnings cost of \$140.20/bdt
- Burning the lights fraction is the most costly option
 - Due primarily to feedstock replacement cost
- Local co-product value is the best option if lights not leached



3 – Technical Accomplishments/ Progress/Results



Flowsheet – 2014 Woody Feedstock State of Technology (SOT)