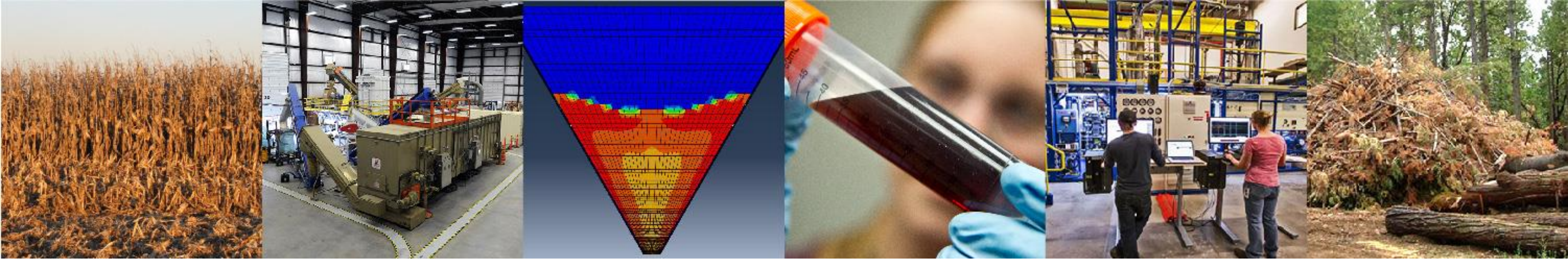


DOE Bioenergy Technologies Office (BETO) 2019 Project Peer Review

Feedstock-Conversion Interface Consortium (FCIC)
Fiscal Year 2018
WBS 2.2.1.50X Process Integration
FCIC Review Session
March 7, 2019

Edward J. Wolfrum, Ph.D. (PI)

FEEDSTOCK-CONVERSION INTERFACE CONSORTIUM

Process Integration (PI) Team

An inter-disciplinary and cross-NL team with expertise in **PREPROCESSING, CONVERSION, MATERIALS SCIENCE, COMPUTATIONAL MODELING**


Vicki Thompson, Ph.D.


Erik Kuhn, P.E.


Dan Carpenter


Peter Ciesielski, Ph.D.

NREL
NATIONAL RENEWABLE ENERGY LABORATORY


Tyler Westover, Ph.D.


Jim Keiser, Ph.D.


Kristen Smith


Jim Parks, Ph.D.


Neal Yancey

INL Idaho National Laboratory

Argonne NATIONAL LABORATORY


Ed Wolfrum, Ph.D.


Nick Nagle


Mel Tucker, Ph.D.


John Aston, Ph.D.


Jonathan Stickel, Ph.D.


Jun Qu, Ph.D.


George Fenske, Ph.D.

OAK RIDGE National Laboratory

FCIC Overall Goal

Identify and address the impacts of feedstock variability – chemical, physical, and mechanical – on biomass preprocessing and conversion equipment and system performance, to move towards 90% operational reliability

FCIC Process Integration Goal

FY18 Goal

Document the current state of **throughput** and **conversion performance** with feedstocks having widely variable properties by executing **Experimental Baseline Runs** for preprocessing, low-temperature conversion, and high-temperature conversion

Outcomes

- Robust data sets documenting the effect(s) of feedstock variability on throughput and conversion performance for preprocessing, low-temperature conversion, and high-temperature conversion, and
- Well-characterized physical samples (>1000) available for future work by FCIC teams

Relevance

This work provided pilot-scale data on feedstock effects which provided the experimental basis for future FCIC work

Quad Chart Overview

Timeline

- Project Start Date: Nov. 2017
- Project End Date : Dec. 2018 (anticipated)
- Percent Complete: 100%

	Total Costs Pre FY17**	FY 17 Costs	FY 18 Costs	Total Planned Funding (FY 19-Project End Date)
DOE Funded	NA	NA	$3.26M M	$0.4MM
Project Cost Share*	NA	NA	NA	NA

Partners: INL ($1.666MM, NREL $1.4MM, ORNL $200K)

Barriers addressed

Ft-E. Feedstock Quality: Monitoring and Impact on Conversion Performance

Ct-A. Defining metrics around feedstock quality

Ct-B. Efficient Preprocessing and Pretreatment

ADO-A. Process Integration

ADO-D. Technical Risk of Scaling

Objective

The overall goal of the Feedstock Conversion Interface Consortium (FCIC) was to develop improvements to feedstock supply-preprocessing-conversion processes that enable >90% operational reliability

FY18 Goal

Document the current state of throughput and conversion performance with feedstocks having widely variable properties by executing Experimental Baseline Runs for preprocessing, low-temperature conversion, and high-temperature conversion

1 - Project Overview

Feedstock Variability and Specification Development
Allison Ray (INL)

Feedstock Physical Performance Modeling
Tyler Westover (INL)

Biomass Quality

Process Integration
Ed Wolfrum (NREL)

Upstream-Downstream Process Integration

System-wide Throughput Analysis
Dave Thompson (INL)

Process Control and Optimization
Quang Nguyen (INL)

System Readiness Evaluation

Characterizing Biomass & Mining Data

Improving Operational Performance

Implementing Integrated Control Strategies

1 - Project Overview (2)

Experimental Baseline Runs

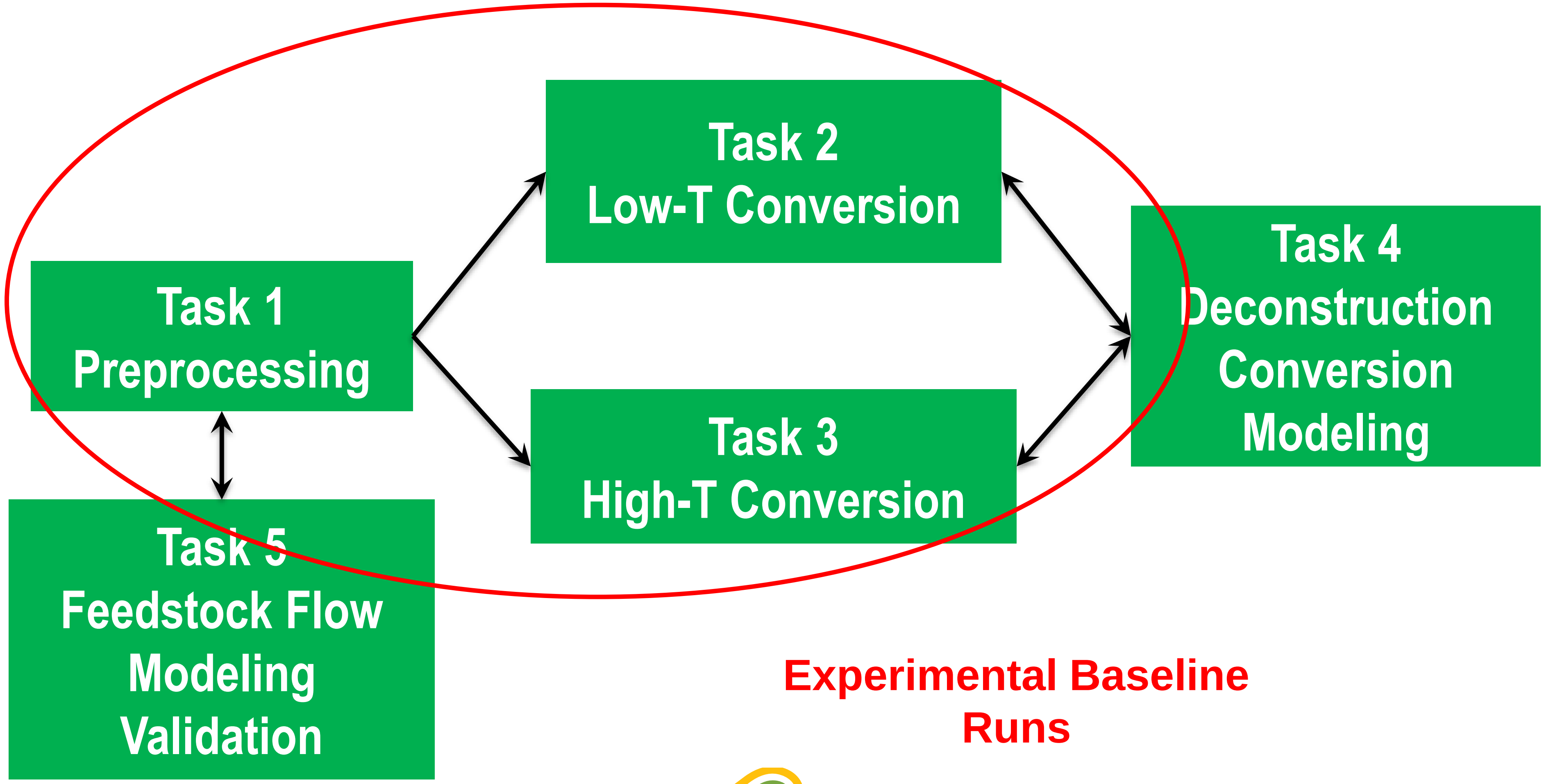
These runs were designed to document the current state of **throughput** and **conversion performance** at process-relevant scales with commercially-relevant feedstocks having widely variable properties

Key Features

- Examined biomass <u>preprocessing through first-stage deconstruction</u>
  - High-Temperature – pyrolysis of pine/pine residues
  - Low-Temperature – dilute sulfuric acid pretreatment of corn stover
- Operated the pilot plants in an integrated fashion
- Collected robust data sets including
  - Process Data
  - Characterization Data on Samples
  - Observational Data on Process Operations
- Included detailed examination of selected equipment wear

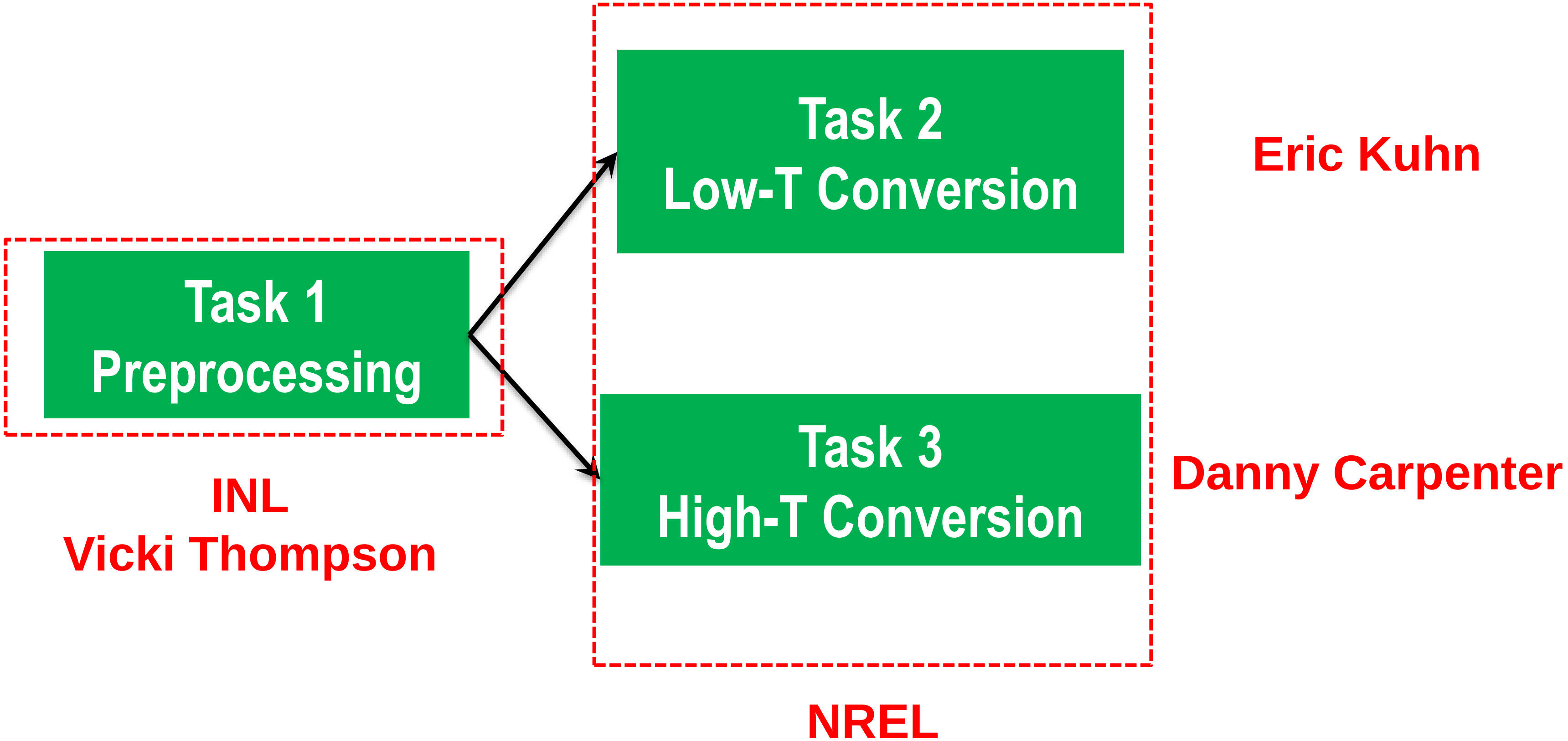
2 - Management Approach

The project was a collaboration among four different National Laboratories
INL, NREL, ORNL, ANL



2 - Management Approach (2)

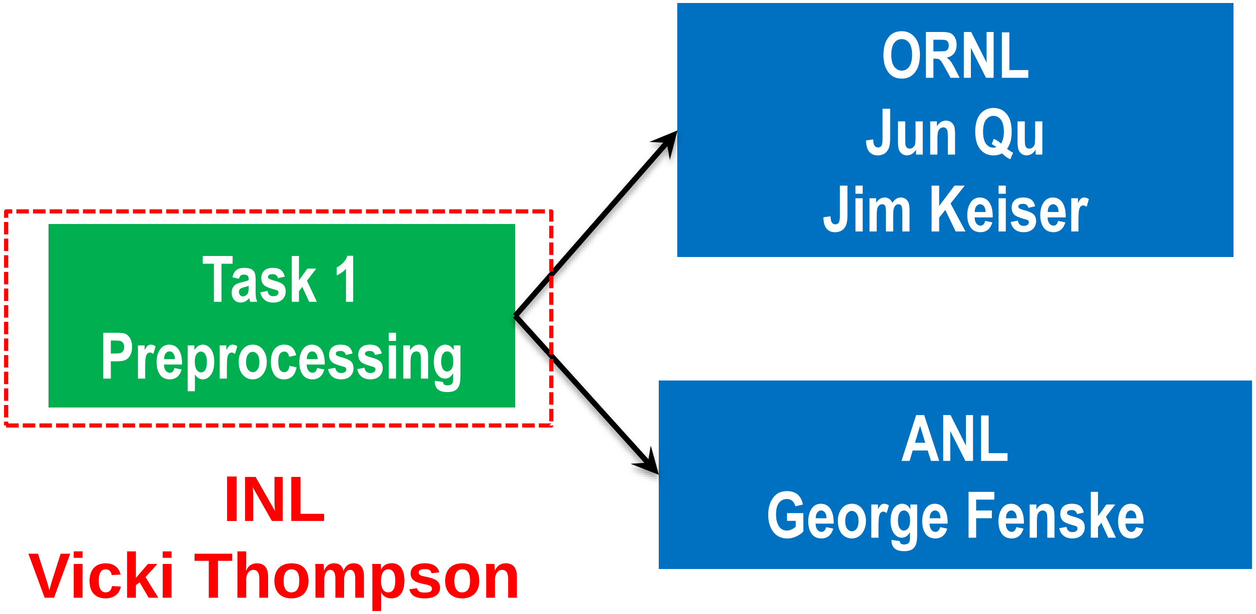
Experimental Baseline Runs



2 - Management Approach (3)

Experimental Wear Baseline

- Organizationally part of Task 1

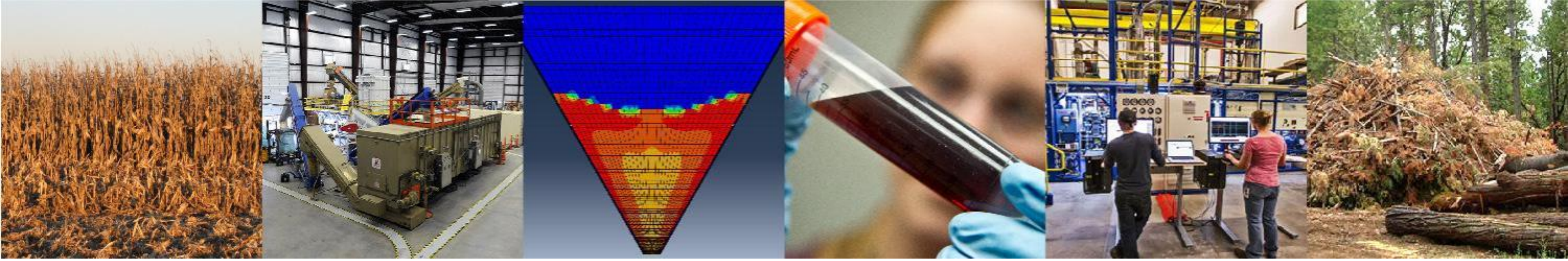


INL coordinated the work investigating equipment wear in preprocessing & low-T conversion

INL & NREL provided samples to ORNL for chemical analysis

ORNL & ANL served as SME's for wear

Detailed reporting on wear work included in FY18 Q3 and Q4 Quarterly Reports



2 – Technical Approach

2 – Technical Approach

Technical Approach

- Design & execute the Experimental Baseline Runs for both low-T and high-T pathways, including collecting process and observational data, securing physical samples, and subsequent sample analysis

Challenges

- Helping “stand up” the FCIC; participating in a Consortium with colleagues with diverse backgrounds and technical skills
- Operating the three experimental PDUs differently
  - Integrated runs between INL and NREL PDUs
  - Documenting “throughput” in addition to “conversion performance”

Critical Success Factors

- Quantifying preprocessing and conversion sensitivity to feedstock variability
- Changing how we normally operate the PDUs – collecting intervention and process data and smoothly integrating INL and NREL PDUs
- Identifying biomass properties impacting “throughput”

2 – Technical Approach (cont'd)

FY18 Goal

Document the current state of **throughput** and **conversion performance** with feedstocks with widely variable properties by executing **Experimental Baseline Runs** for preprocessing, low-temperature conversion, and high-temperature conversion

Feedstock Variability

- Variable ash and moisture content

Throughput

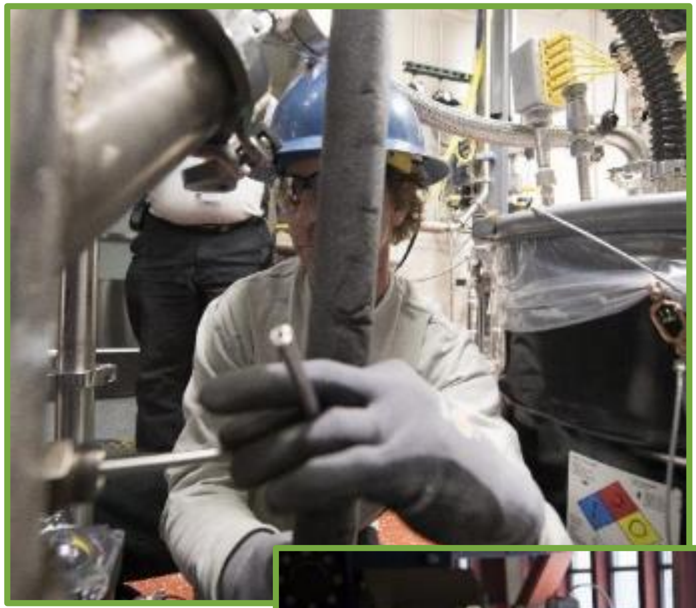
- Records of process upsets & required interventions
- Expressed in consistent and reasonable ways

Conversion Performance

- Described using well-accepted conversion expressions for both pathways

Experimental Baseline Runs

Low-Temperature – corn stover
High-Temperature – clean pine & forest residues



LALM – low ash, low moisture
LAHM – low ash, high moisture
HALM – high ash, low moisture
HAHM – high ash, high moisture

Facilities Used for Experimental Baseline Runs

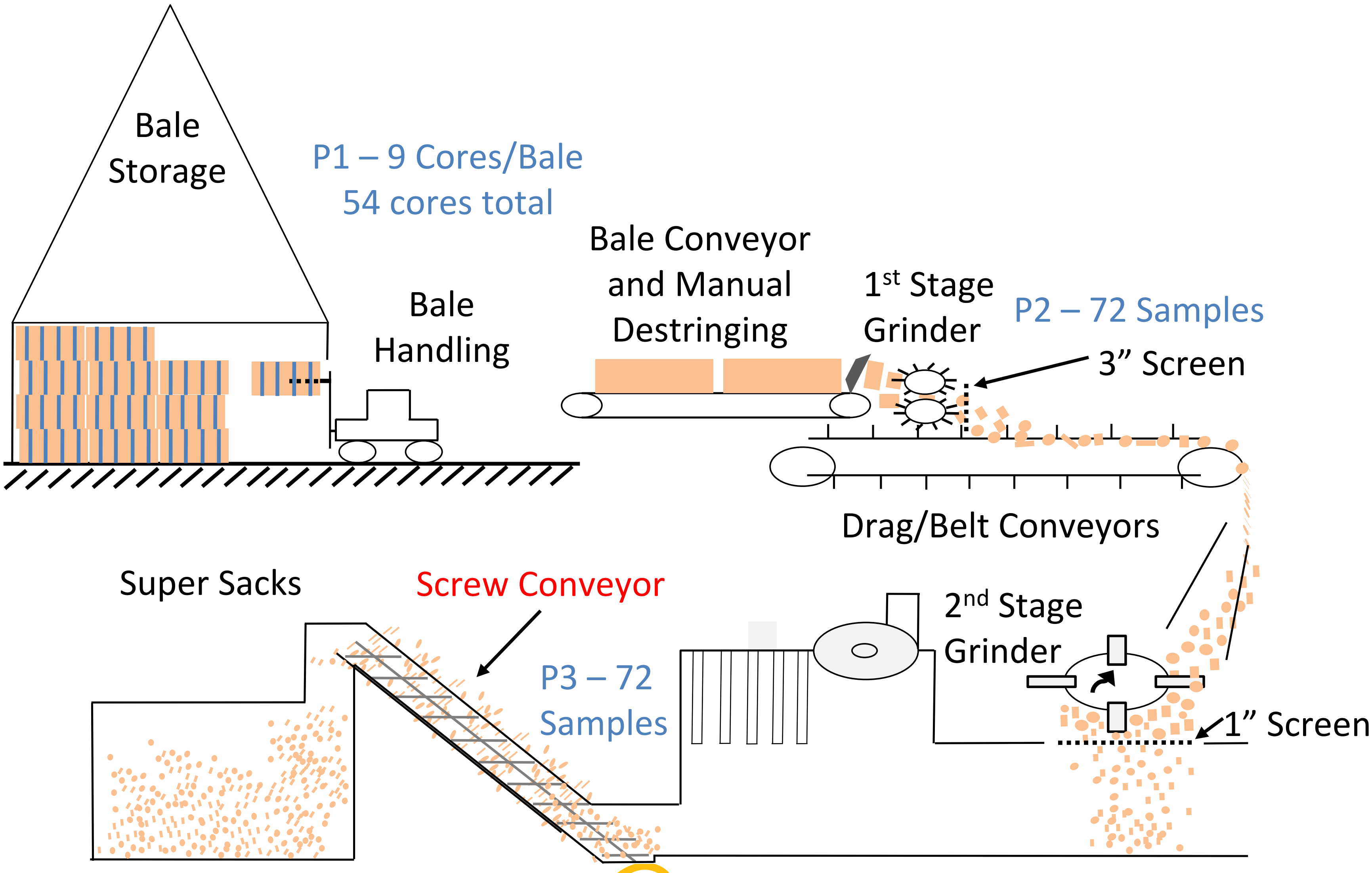
- Preprocessing (both pathways)
  - **INL BFNUF**
- Low-Temperature Conversion
  - **NREL IBRF**
- High-Temperature Conversion
  - **NREL TCPDU**
- Equipment Wear Baseline Study
  - **INL, NREL, ORNL, ANL**

TCPDU (NREL)

BFNUF (INL)

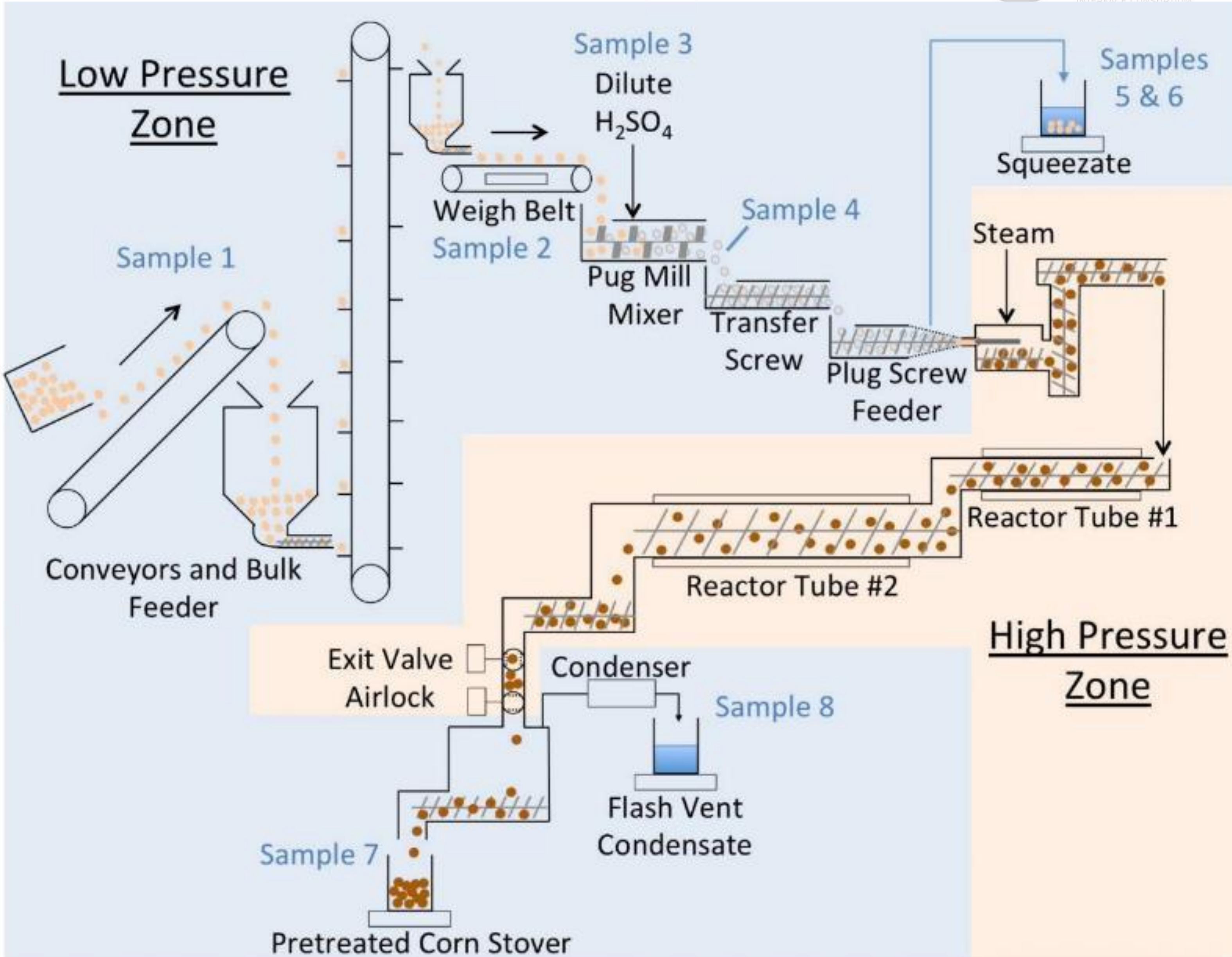
IBRF (NREL)

Low-Temperature Preprocessing

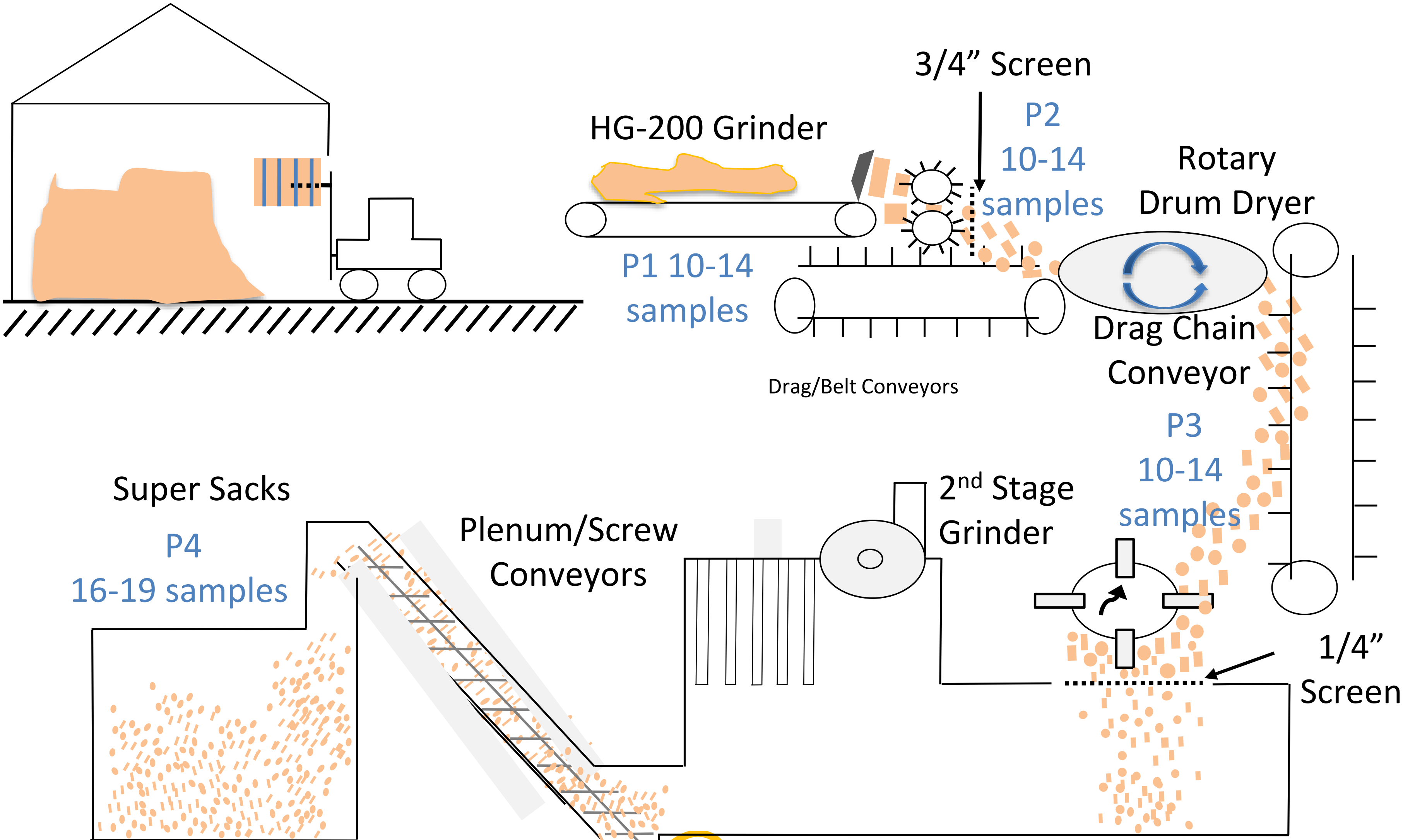


Low Temperature Conversion

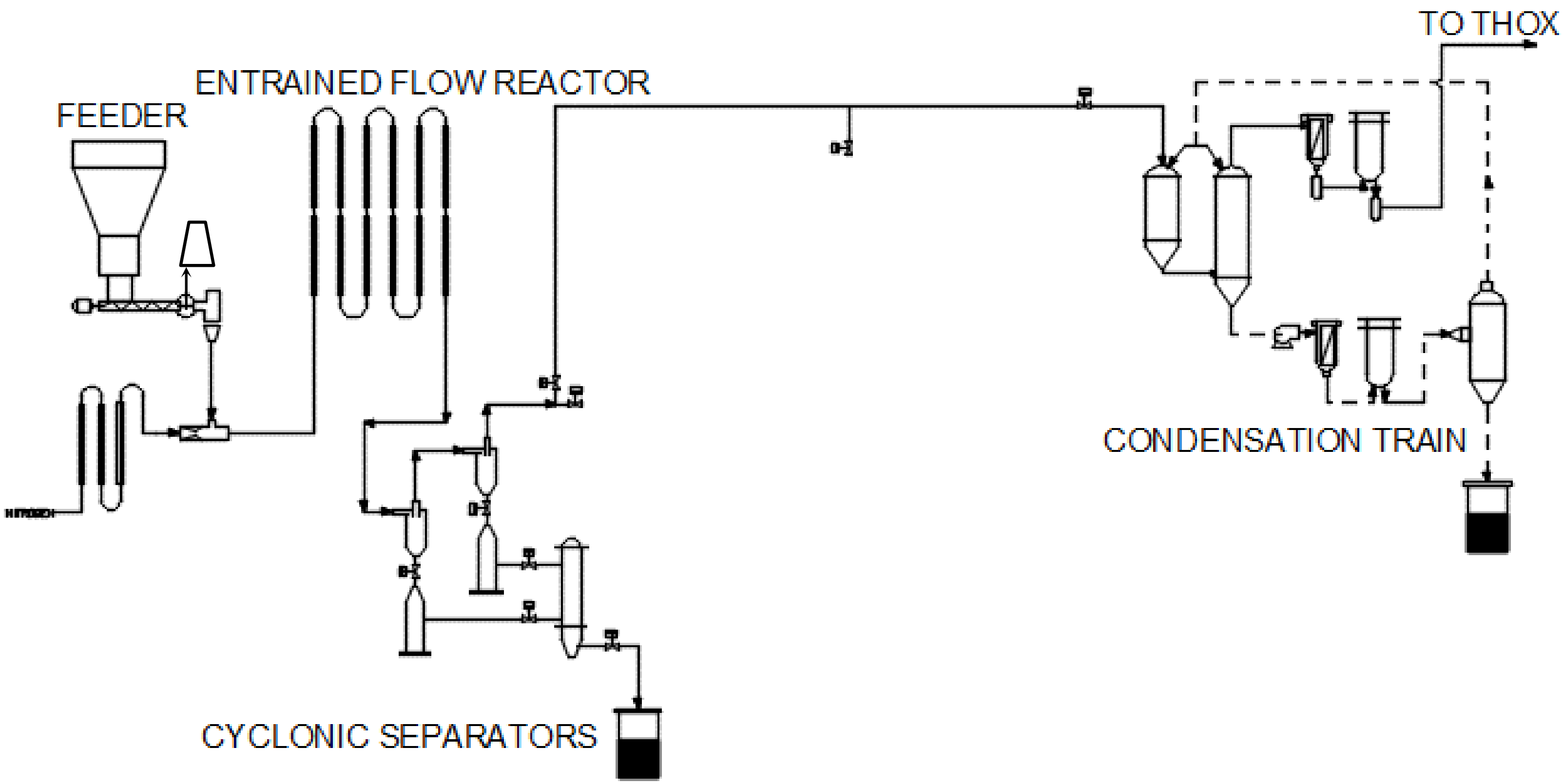
- 0.5 MT/d
- Direct steam injected
- Corrosion resistant Hastelloy in high pressure zone
- Reactor tubes 1 and 2 steam jacketed



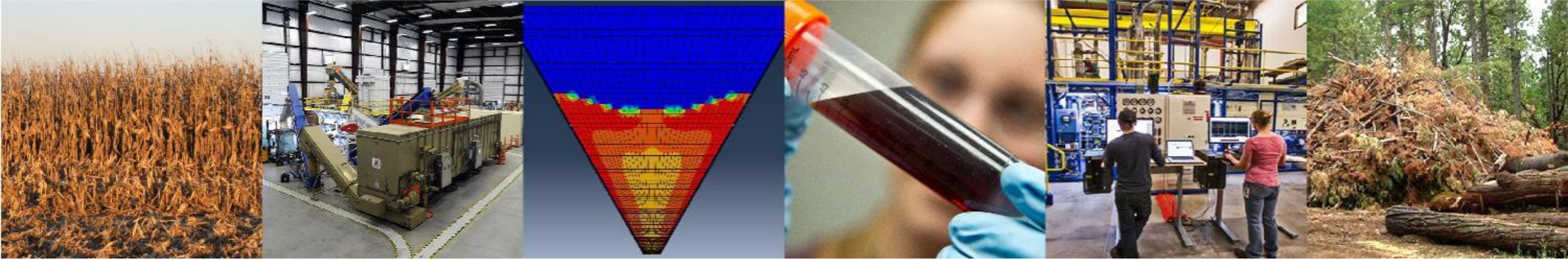
High-Temperature Preprocessing



High-Temperature Conversion



- **Throughput: 0.5 ton/day biomass**
- **Pyrolysis Temperature: 500°C**
- **Residence time: < 3 s**
- **Condensation: spray quench**



Accomplishments

Experimental Baseline Runs

- Execution of the Experimental Baseline Runs was the major activity for the Process Integration AOP in FY18 (along with Wear Baseline work)
- FY18 Milestones Provide Details:
  - Q2 – Detailed Experimental Plan/Schedule for Review
  - Q3 – Preliminary Results from Initial Runs
  - Q4 – Summary Report on all Runs

We will only show highlights of this work here

Key Findings – Throughput (Low-T)

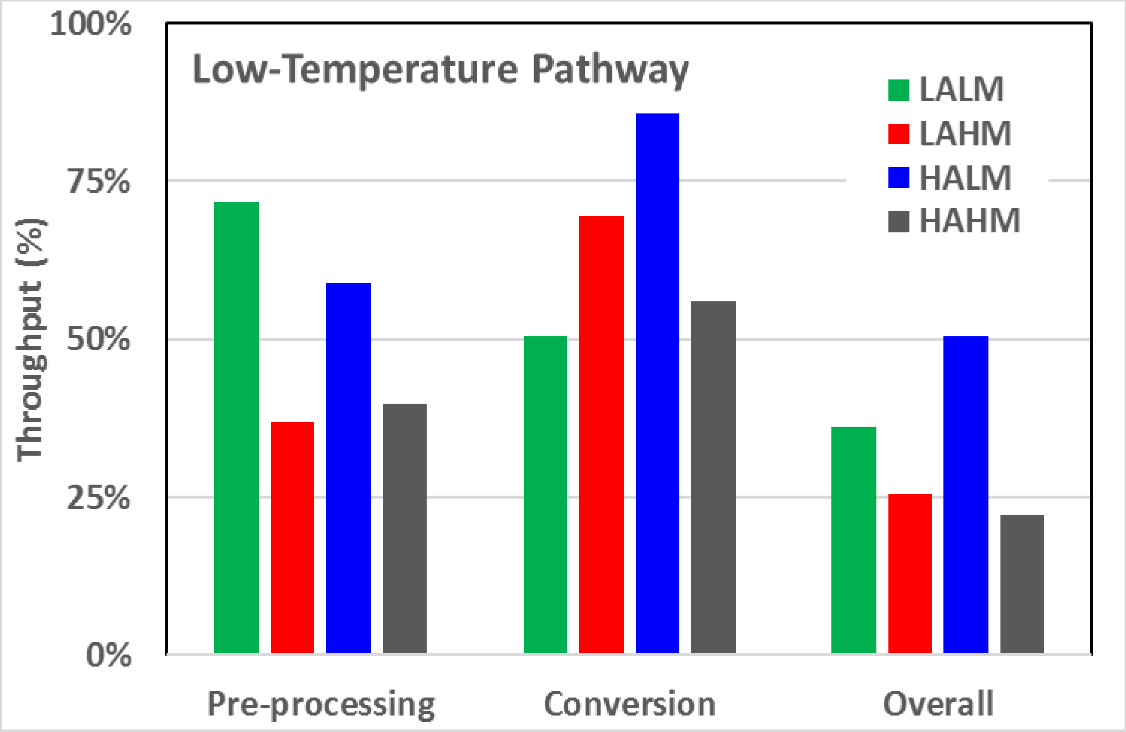
Throughput - Actual input mass flowrate divided by PDU nameplate capacity
Overall Throughput - Product of pre-processing and conversion throughput

LALM Corn Stover

- best in preprocessing but worst in conversion
- more long, stringy material than any other material
- most plugging of pretreatment plug screw feeder

Fundamental Questions

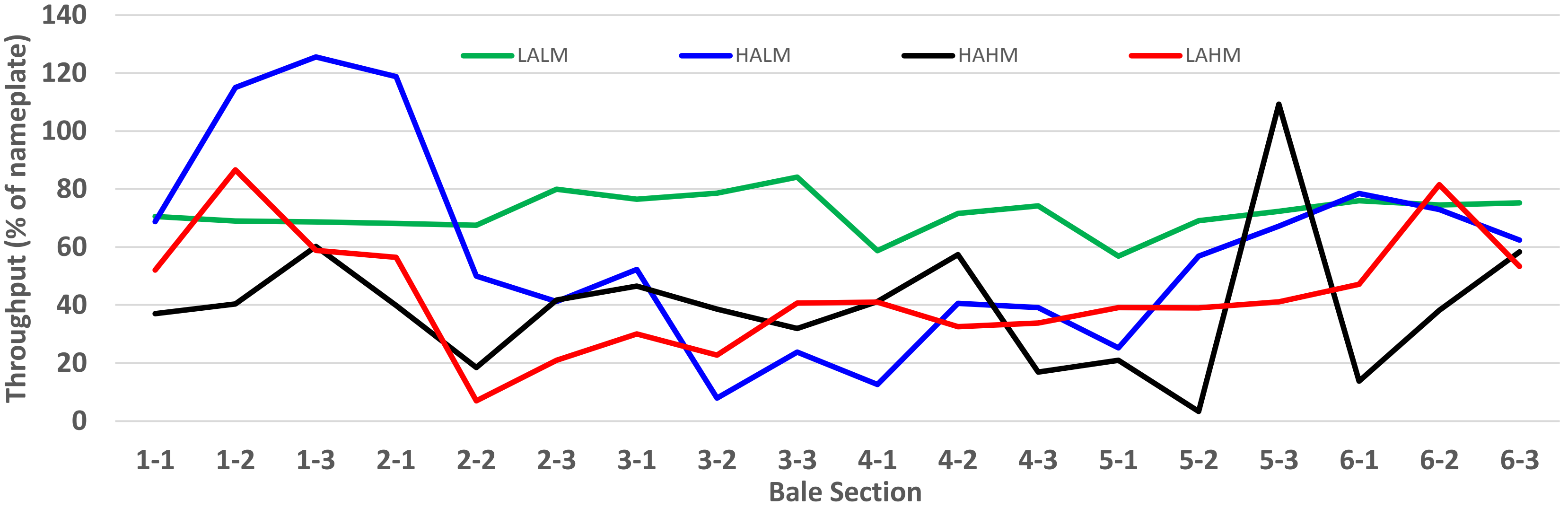
- How can we quantify storage impacts on biomass?
- Can we predict biomass-moisture interactions?
- How do different tissue types deconstruct?



Throughput Varied Within a Single Run

Low-Temperature Preprocessing

Throughput data averaged by bale section



- Some materials processed consistently (**LALM**), others less so (**HALM**, **HAHM** and **LAHM**). Processability varied widely within a single bale **HAHM**)
- Feedstock variability due to variability in harvest, collection, storage, transport

Mitigation Options

- Homogenize bales prior to grinding
- Better methods to predict bale properties prior to preprocessing

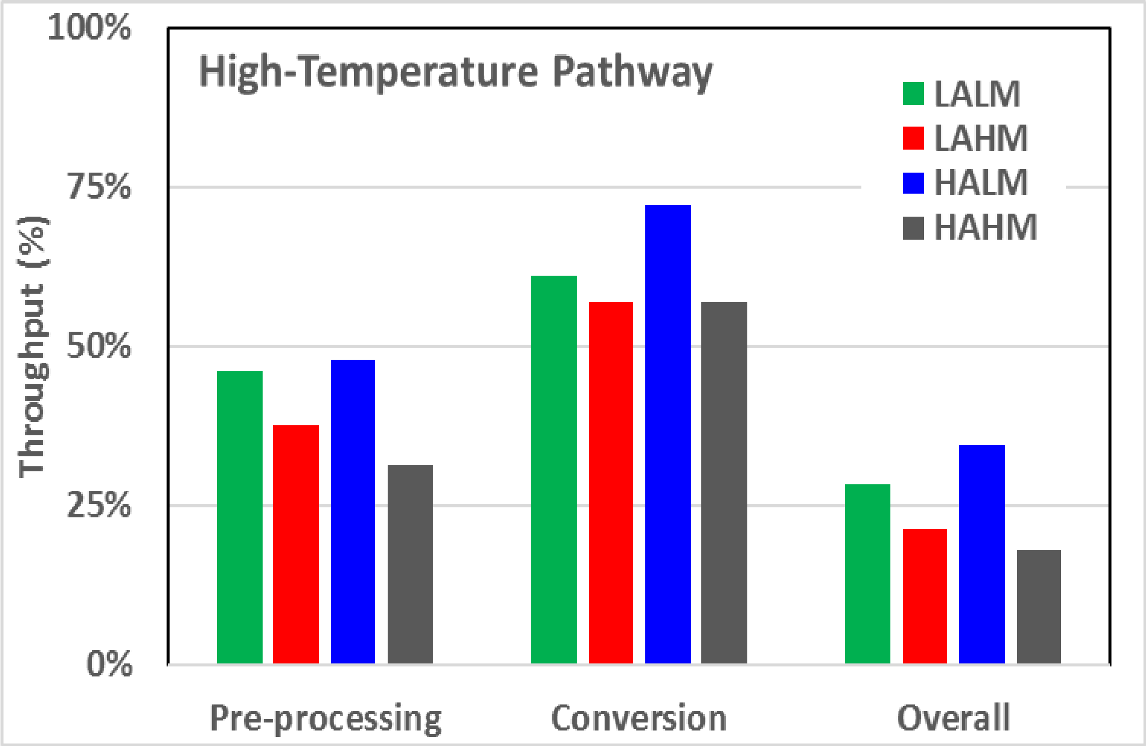
Key Findings – Throughput (High-T)

Throughput - Actual input mass flowrate divided by PDU nameplate capacity
Overall Throughput - Product of pre-processing times conversion throughput

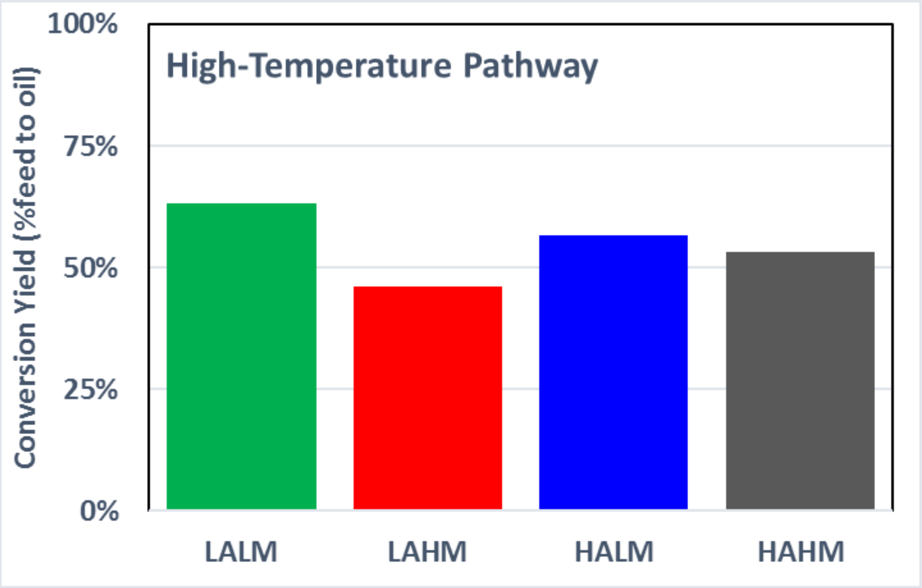
- All materials showed same trends for preprocessing and conversion
- High moisture decreased throughput
- Forest residues had higher throughput than clean pine

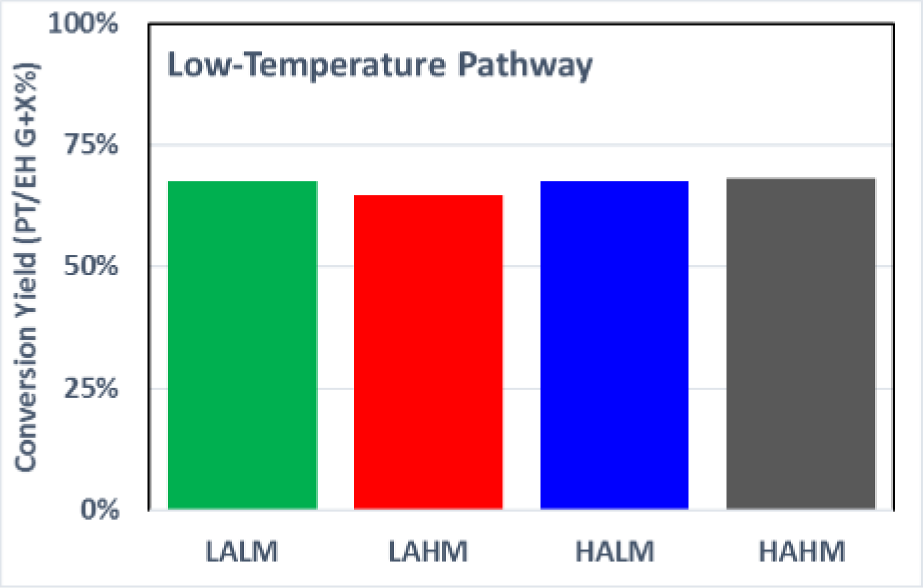
Fundamental Questions

- Why do forest residues flow better?
- How do bark, needles, branches deconstruct?
- Can we predict biomass-moisture interactions?



Key Findings – Conversion Yield

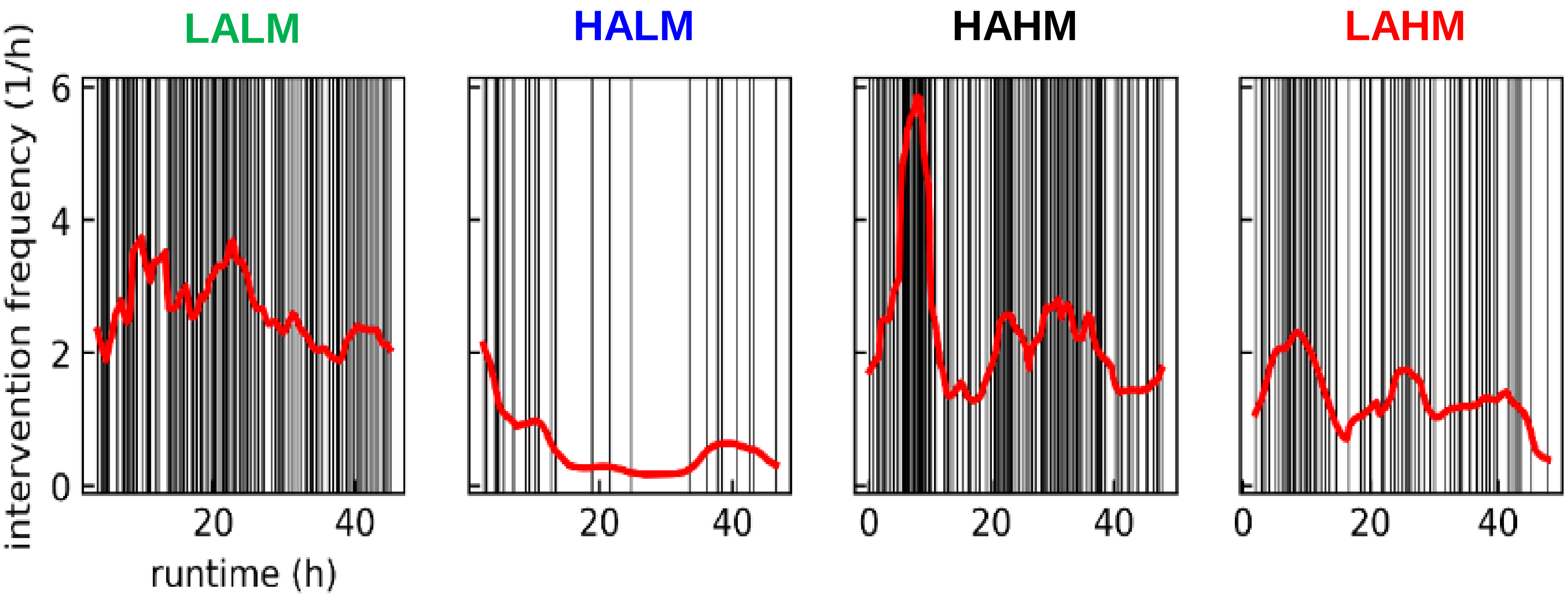




- Some variability in high-temperature conversion yields due to higher ash (**HALM**, **HAHM**) or significant interventions leading to product loss (**LAHM**)
- Less variability in low-temperature conversion – we hypothesize this is the result of mass transfer limitations during acid addition
- Downstream effects (after enzymatic hydrolysis) <u>are still unknown</u> – <u>outside the boundary limits of this work</u>

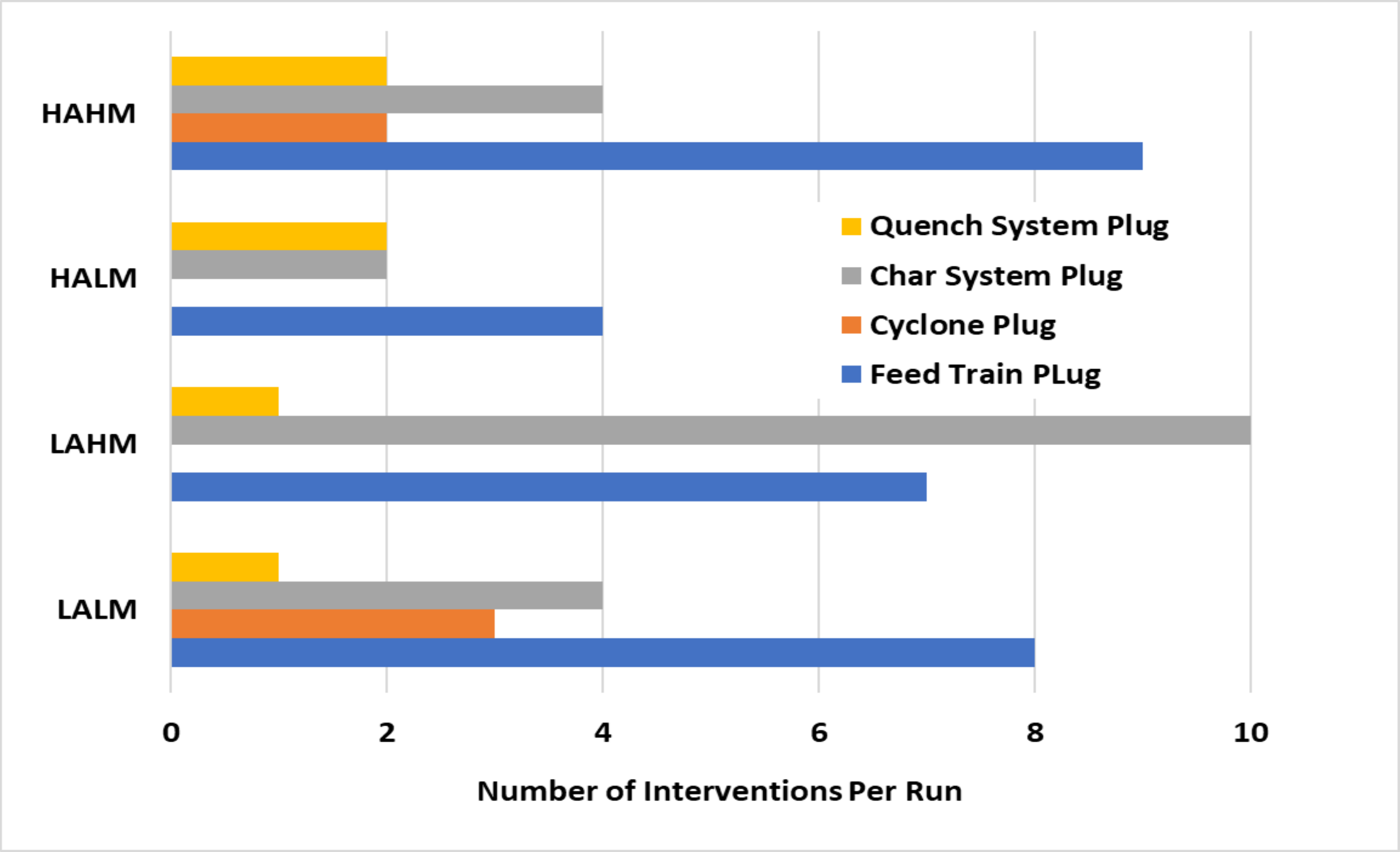
Low-Temperature Interventions

- Low-Temperature Conversion Operator Interventions
- Each black line is a single intervention



- Greater human intervention frequency for **LALM** and **HAHM** runs compared to **HALM** and **LAHM**
- Lowest human intervention frequency for **HALM,** may be due to particle size and density differences

High-Temperature Interventions

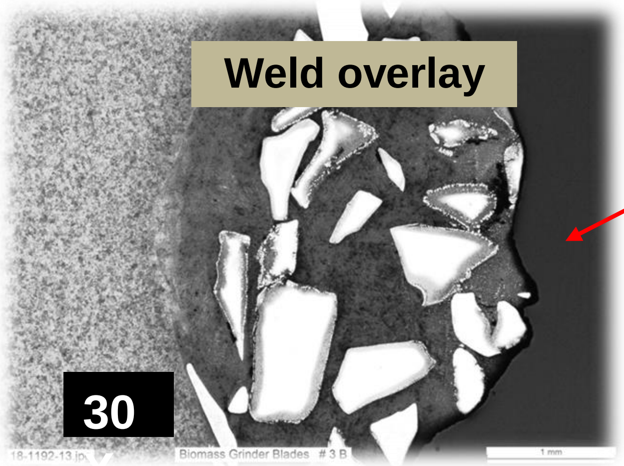


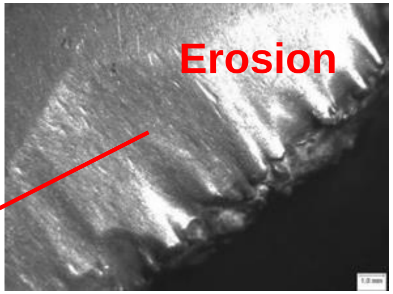
- Large variability in type/frequency of operator interventions among feedstocks
- Lowest intervention frequency for **HALM,** may be due to heterogeneous physical of particles (included wood, bark, needles)

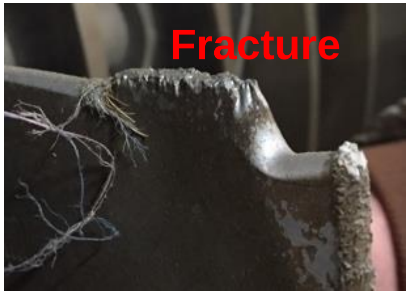
Stage 2 Hammer Mill Wear

Problem: The current carbide weld overlay on the steel hammers experiences significant wear due to its very coarse composite structure and incompatible matrix material.

Proposed mitigation: Wear-resistant, fine-structured coatings possessing higher hardness and fracture toughness.







Blade	Steel substrate	WC-Co particles
Hardness (HV)	$430^{\pm 13}$	$1257^{\pm 110}$

Wear modes: erosion and fracture

Erosion and plastic deformation, as a result of impact and sliding against organic biomass and inorganic extrinsic ash;

Microcracking and fracture, as a result of impact against inorganic extrinsic ash.

Plug Screw Feeder Wear

PSF Disassembly

Unused Screw

Worn Screw

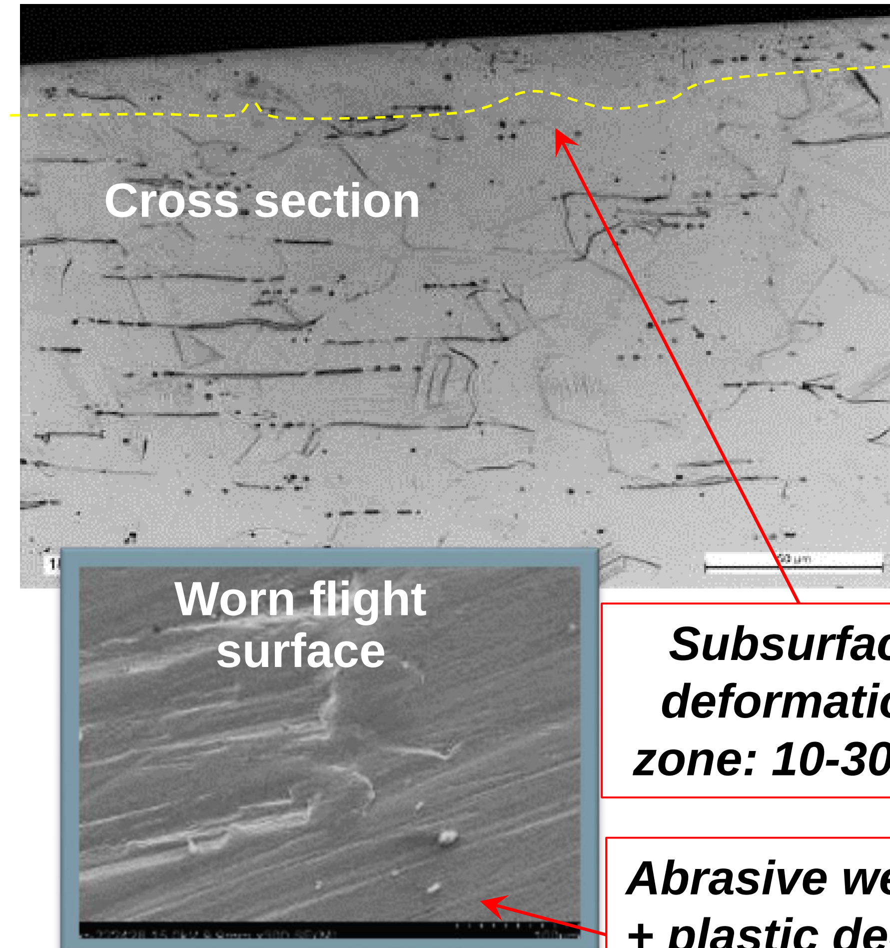
Run ID	Mass Loss (g)
LALM	140
HALM	225
HAHM	283
LAHM	167

- 100% greater wear in **HAHM** versus **LALM**
- Both high-ash runs had larger mass loss than corresponding low-ash runs
- Wear observed in the screw compression zone

Plug Screw Feeder Wear (2)

- Plug screw feeder made of 316 Stainless (316 SS)
- 316 SS is known to have poor wear resistance because of its relatively low hardness and high tendency to scuffing
- Candidate wear-resistant alloys, coatings, and surface treatments surface treatments will likely reduce this wear substantially.

Wear modes
3-body (dominant)
2-body abrasion
plastic deformation



Subsurface deformation zone: 10-30um

Abrasive wear + plastic de-formation

Key Learnings – Preprocessing & Conversion

Biomass Preprocessing

- Biomass is variable and many sources of variability cannot be controlled
  - Large variability in measured properties within a single batch of material causing variability in throughput among tested materials – up to 10x difference in throughput for a single material
- We need fundamental studies of biomass deconstruction and flow
- We need to understand the biorefinery at the unit operations level to understand how feedstock variability propagates through biorefinery
- Active process control could be a valuable mitigation tool

Biomass Conversion

- Feedstock Variability affected throughput and Conversion Performance – specific differences depended on material and pathway
- Need to extend our investigations downstream of the initial project boundaries of initial deconstruction step (i.e. product quality attributes that impact downstream steps)

Key Learnings - Equipment Wear

Preprocessing

- Wear mechanisms in selected preprocessing equipment identified, and varied with unit operation
- Accelerated wear testing producing similar wear modes to pilot-plant runs

Conversion (Low-Temperature)

- Significant wear losses of the plug screw feeder in baseline runs with strong correlations to total ash content
- Materials approaches likely to reduce wear (coatings/surface treatments)

Implications for Future Work

- There is little literature addressing the effects of feedstock properties (and property variability) on mechanisms and rates of wear in biomass preprocessing and conversion equipment
- Work needed to identify critical biomass properties and connect them with equipment wear rates; both fundamental and applied approaches useful

Relevance

FCIC FEEDSTOCK-CONVERSION INTERFACE CONSORTIUM

FCIC Overall Goal

The overall goal of the Feedstock Conversion Interface Consortium (FCIC) was to identify and address the impacts of feedstock variability – chemical, physical, and mechanical – on biomass preprocessing and conversion equipment and system performance, to move towards 90% operational reliability

FCIC Process Integration Project FY18 Goal

Document the current state of throughput and conversion performance with feedstocks having widely variable properties by executing Experimental Baseline Runs for preprocessing, low-temperature conversion, and high-temperature conversion

Relevance (cont'd)

This project addressed Feedstock Variability

- Variability among different feedstocks
- Temporal variability with a given feedstock
- This variability strongly affected performance

Robust Data Sets from Three Pilot Plants

- Throughput in addition to conversion performance
- Data will be made available to the public through the INL Bioenergy Feedstock Library (BFL)
- Key learnings will guide future work in this area
  - Unit-operations-level data on operation reliability
  - Examining effects of feedstock & process variability on downstream (after 1st-stage deconstruction) unit operations

Process Integration - Summary

Overview - A key **experimental project** of the FCIC in FY18, but worked closely with colleagues from other FCIC projects

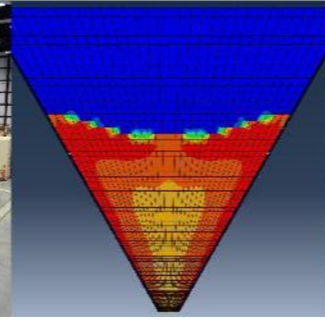
Challenges – (1) coordination of work across multiple National Laboratories and execution of very aggressive experimental plans, (2) operation of the PDUs in a fundamentally different way

Approach - **Execute robust, industrially-relevant Experimental Baseline Runs** that document throughput and conversion performance across biomass preprocessing and both low- and high-temperature conversion process operations

Accomplishments - **Robust datasets** showing the effects of feedstock variability on throughput and conversion performance, and physical samples available for future work

Relevance – Develops **fundamental knowledge** that will solve industry issues



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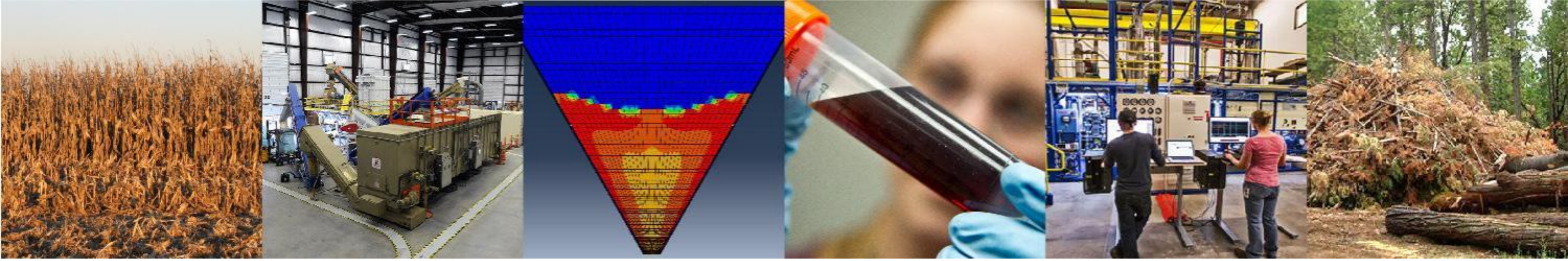


Additional Material

Process Integration Task Structure

Task #	Name	Lead Lab	PI	Key FY18 Activity
1	Upstream/Preprocessing	INL	Vicki Thompson	Baseline Experiments
2	Low-Temperature Conversion Process Integration	NREL	Erik Kuhn	Baseline Experiments
3	High-Temperature Conversion Process Integration	NREL	Dan Carpenter	Baseline Experiments
4	Fundamental Modeling to Predict Conversion Yield	NREL	Peter Ciesielski	NA
5	Validation of Fundamental Modeling Results	INL	Tyler Westover	NA

FY18 FCIC Process Integration Project Milestone Table

Milestone Name/Description	Date	Type
Demonstrate at least one substantial effect (variation in operational reliability with associated conversion performance>30% compared to base case) of feedstock variability on process robustness and/or conversion performance through primary deconstruction for one low-temperature and one high-temperature process using industrially-relevant equipment. Identify at least two experimental approaches to mitigate these effects for the affected unit operations in the pathway.	6/30/18	Quarterly (Regular)
Complete baseline experiments for one low-temperature and one high-temperature preprocessing and conversion pathway at industry-relevant scales to measure process robustness (operational reliability with associated conversion performance) according to plan developed in FY18 Q2.	9/30/18	Annual (Regular)
Demonstrate one low-temperature and one high-temperature conversion modeling approach that is validated with experimental data and predicts variation in conversion performance due to variability in at least three feedstock properties.	9/30/19	Annual (Regular)
Demonstrate a 50% improvement in operational reliability while maintaining conversion performance over baseline data generated in FY18 for both low-temperature and high-temperature conversion pathways in using industrially-relevant experiments	9/30/20	End-of-Project

Material Sourcing – Loblolly Pine

Single species – Loblolly pine

- Most forests have multiple species
- Difficult to find locations with only loblolly

Ash

- Clean pine (low ash)
  - Debarked stems of pulpwood sized trees, aged 11-25 years
  - Ash content typically less than 1%
- Forest residues (high ash)
  - Branches, needles and bark from trees, aged 11-25 years
  - Often left on ground and susceptible to soil contamination
  - Ash content can range widely but typically 3-5%

Moisture

- At harvest, moisture is 40-50%
- Industry practice is to dry to 10% then grind
- Chose to dry to 10% and 30% to alter PSD

Woody Biomass Harvesting

①

Pine Plantation

②

Feller-buncher

③

Grapple skidder

④

Whole tree disc knife chipper with chain flail

Clean Pine Chips

⑤

Residue pile

⑥

Drum knife chipper

Residue Chips

Source and Preprocessing

	Clean Pine	Forest Residues
Sample:	Clean Loblolly Pine	Loblolly Pine Residues
Harvest Site:	Screven, GA; 3/27/18	Edgefield County, SC; 3/26/18
Moisture Content:	49.3% at harvest	50.9% at harvest
Anatomical Fraction:	Chips of de-barked stem / bole, 11-25 years of age	Loblolly in-woods tops, ~7-in. dib at large end, 11-25 years of age
Harvest Equipment:	TIGERCAT 724G Feller buncher, TIGERCAT 630E grapple skidder, Peterson Pacific 5000H Disc Chipper with flail chains	CAT 563D Feller buncher, CAT 535D grapple skidder, CAT 559C knuckleboom loader, MORBARK 40/36 drum knife chipper
Harvest Operations:	Debarked, chipped, 2-in. nominal, ~23 tons (wet) loaded directly to trailer	Tops removed and placed in a pile, chipped 2-in. nominal, ~22 tons (wet) loaded directly into trailer
Screening:	NO	NO

Material Sourcing – Corn Stover

Condition	Ash	Moisture
LALM	5-10%	10-20%
HALM	10-20%	10-20%
HAHM	10-20%	25-40%
LAHM	5-10%	25-40%

Sourcing Challenges

- Harvest already completed prior to FCIC start
- After merging with Dow, Dupont shut down Project Liberty during sourcing
- Iowa farmers rarely square bale unless contracted ahead of time
- Bales stored up to 6 months

Condition	Source	County	Equipment	Harvested	Baled
LALM	Dupont	Story	Agco 2270XD Large Square Baler	10/27/17	10/27/18
HALM	Dupont	Hardin	Agco 2270XD Large Square Baler	10/21/17	10/21/17
HAHM	Dupont	Hamilton	Agco 2270XD Large Square Baler	10/18/17	10/18/17
	Heishman	Poweshiek	Hesston 2270XD Large Square Baler	9/18/17	10/12/17
LAHM	Dupont	Hamilton	Agco 2270XD Large Square Baler	10/18/17	10/18/17
	Heishman	Poweshiek	Hesston 2270XD Large Square Baler	9/18/17	10/12/17

Summary Data – Throughput

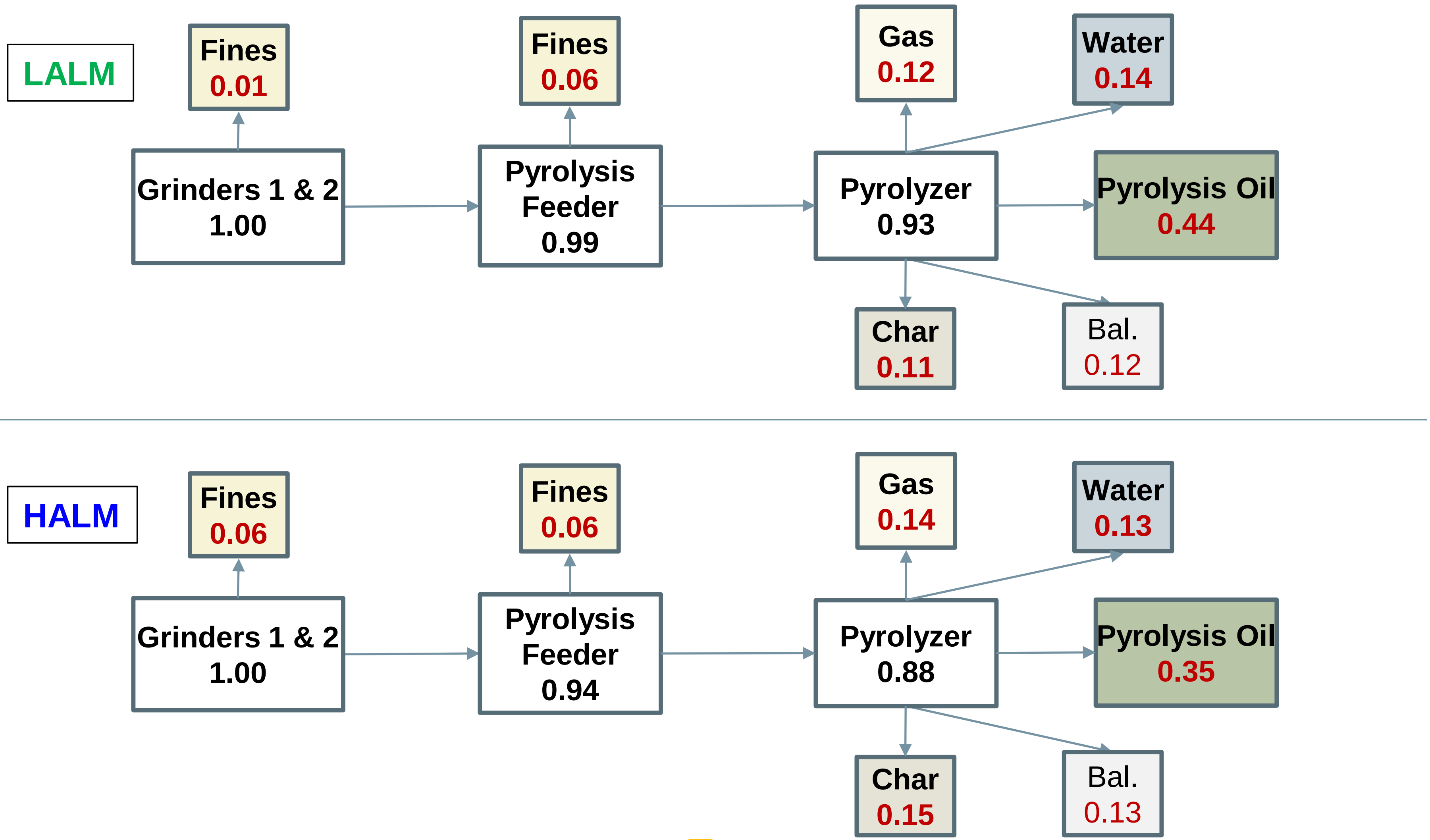
Throughput - Actual input mass flowrate divided by PDU nameplate capacity
Overall Throughput - Product of pre-processing times conversion throughput

High-Temperature Pathway			
	Pre-processing	Conversion	**Overall**
LALM	46%	61%	**28%**
LAHM	38%	57%	**21%**
HALM	48%	72%	**35%**
HAHM	31%	57%	**18%**

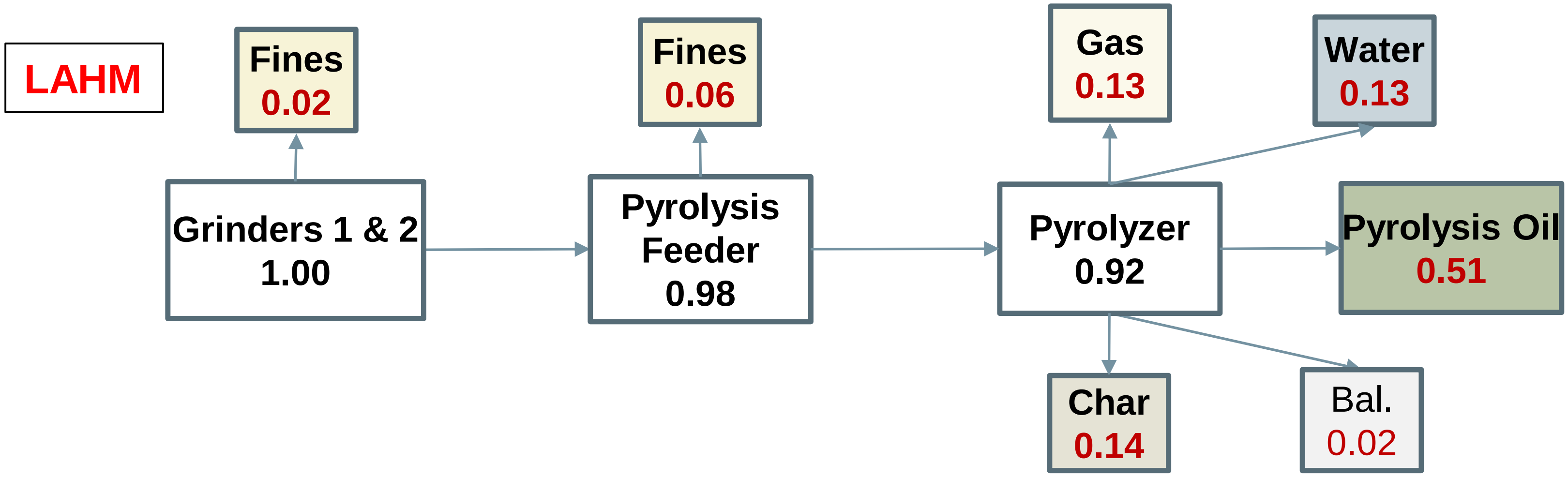
Low-Temperature Pathway			
	Pre-processing	Conversion	**Overall**
LALM	72%	50%	**36%**
LAHM	37%	70%	**26%**
HALM	59%	86%	**50%**
HAHM	40%	56%	**22%**

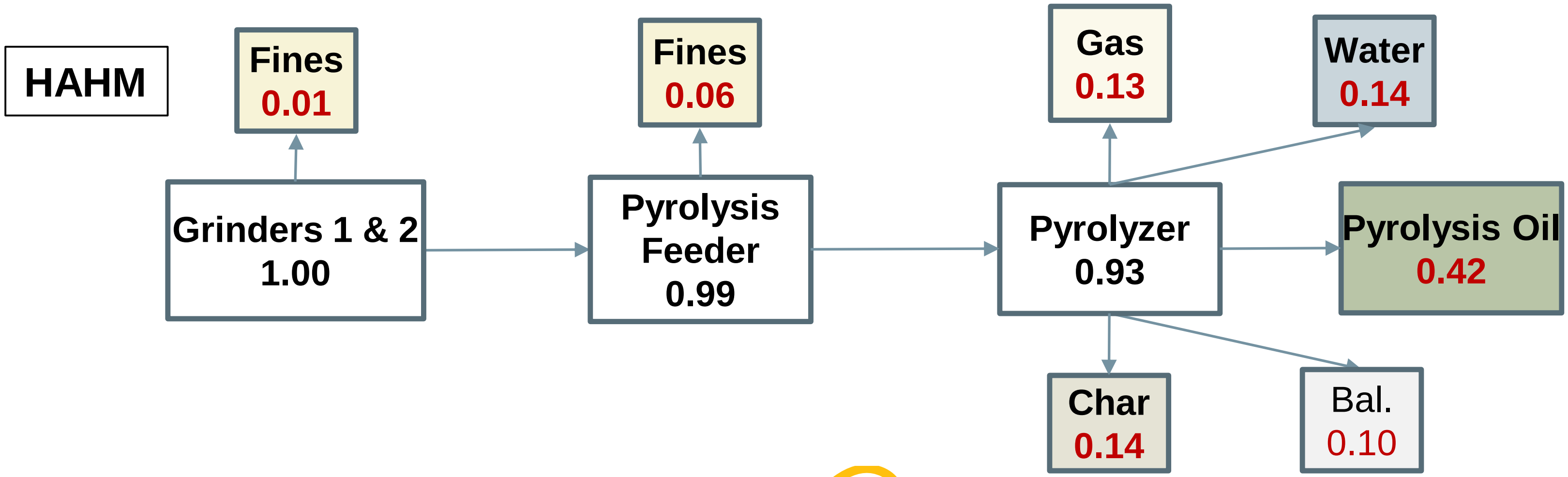
C

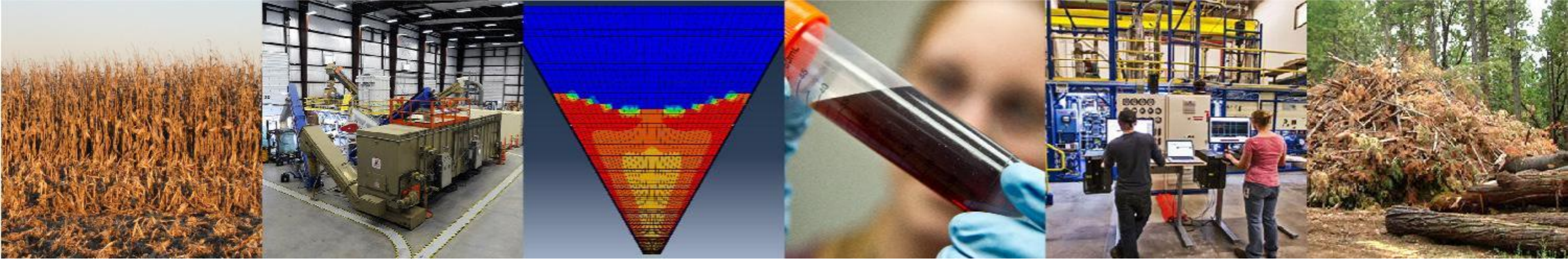
High-T Results (Mass Balances)



High-T Results (Mass Balances)



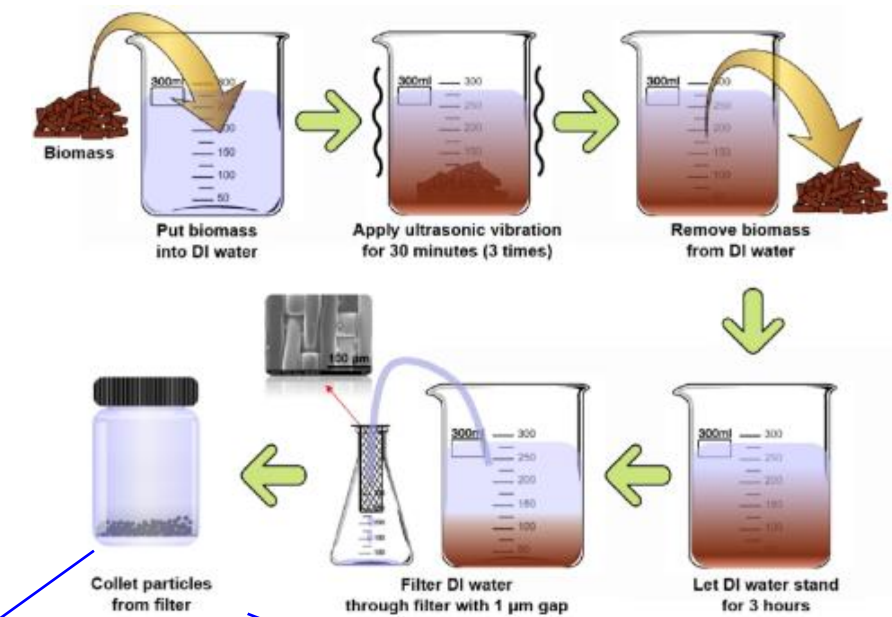


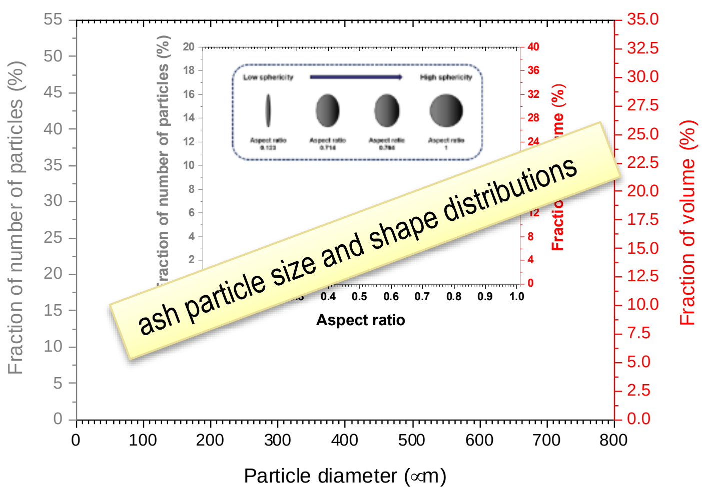


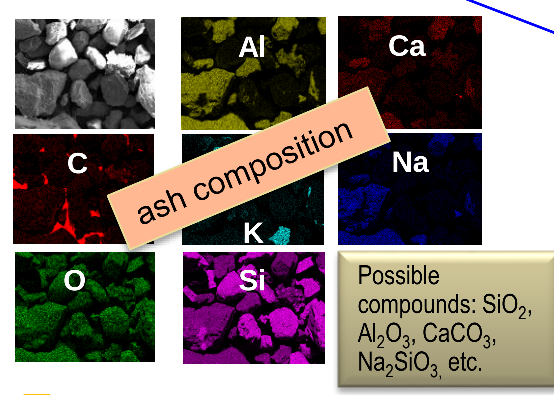
Additional Wear Investigation Data

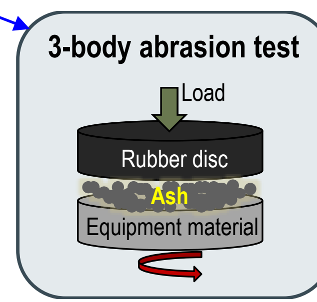
Characterization of the extrinsic ash of corn stover

- **Conventional ash extraction is 'furnace burning' based, which severely alters the original ash compounds as a result of oxidation and/or decomposition.**
- **This study used sonication+filtering to extract extrinsic ash to preserve the original ash compounds.**
- **Analyzing ash particle size and shape distributions (SEM+Image analysis)**
- **Revealing ash species (EDS, XRD, and XPS)**
- **Measuring the ash mechanical properties (nanoindentation)**
- **Evaluating ash abrasiveness (3-body abrasion test)**
- **Correlating the ash attributes with the wear behavior of equipment components in preprocessing**



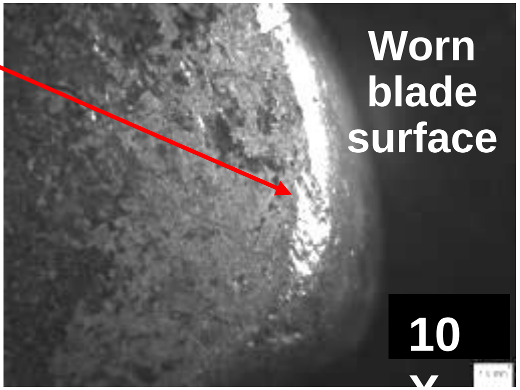






Investigation of the wear of bale grinder (stage 1)

- **Stage 1. Bale Grinder (**400 HP, 800-1500 rpm)
  - **The current carbon steel hammers experience significant wear due to its low hardness.**
  - **Candidate wear-resistant alloys or case hardening treatments are to be identified in FY19-20.**



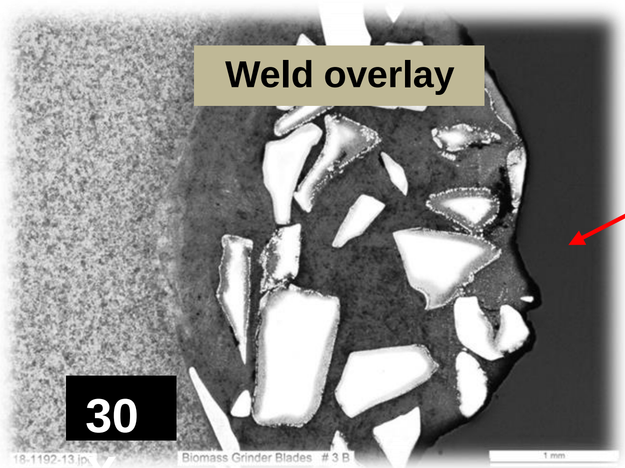
Hardness of current carbon steel hammers: 400-500 HV

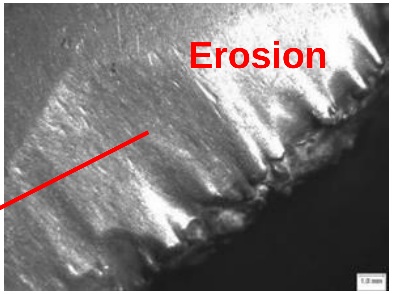
Wear mode: erosion and abrasion

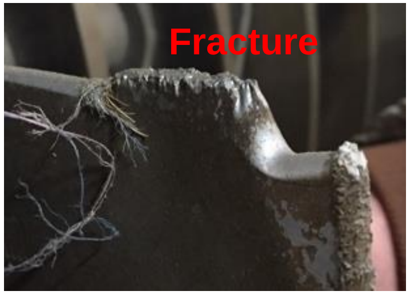
- **Abrasion/polishing: impact and slide against soft biomass particles and hard inorganic contaminant particles at low incident angles**
- **Chipping/indentation: impact and slide against hard inorganic contaminant particles at high incident angles**

Investigation of the wear of hammer mill (stage 2)

- **Stage 2. Hammer Mill (150 HP, 1750 rpm)**
  - **The current weld overlay on the steel hammers experiences significant wear due to its very coarse composite structure and incompatible matrix material.**
  - **Candidate wear-resistant fine-structured coatings with both higher hardness and toughness are to be identified in FY19-20.**







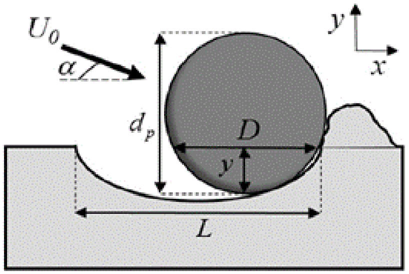
Blade	Steel substrate	WC-Co particles
Hardness (HV)	$430^{\pm 13}$	$1257^{\pm 110}$

Wear modes: erosion and fracture

- **Erosion and plastic deformation,** as a result of impact and sliding against organic biomass and inorganic extrinsic ash;
- **Microcracking and fracture,** as a result of impact against inorganic extrinsic ash.

Mechanics of Wear

- Project start – September 2018
- Develop semi-mechanistic analytical tools to predict wear of feedstock components as functions of feedstock properties, material properties, and operating conditions
  - Abrasion/erosion, adhesion, fatigue, corrosion
  - Hammermills, screw feeders, conveyors, bins, hoppers
- Model input parameters include:
  - Impingement angle, speed, restitution, particle size & density, hardness, toughness, & fatigue
- Model replicated & implemented
- Future Plans –
  - Validation of model against INL ‘corn-stover’ wear data
  - Material property requirements for specific operating conditions



Vertical component – deformation
Horizontal component – cutting

$$\Delta Q_T = \Delta Q_D + \Delta Q_C$$

ΔQ – volume of material worn per unit mass of ash