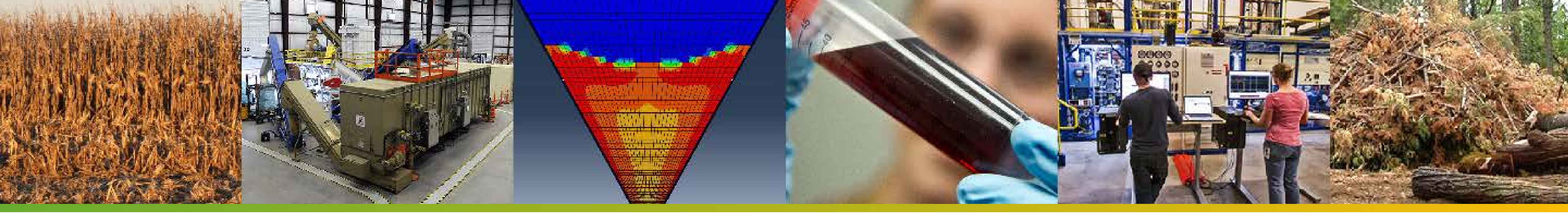

DOE Bioenergy Technologies Office (BETO) 2021 Project Peer Review

FCIC Task 5 - Preprocessing

March 15, 2021
Feedstock Conversion Interface Consortium (FCIC)

Lead - Vicki Thompson, INL
Co-Lead – Rick Elander, NREL

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FEEDSTOCK-CONVERSION
INTERFACE CONSORTIUM

FCIC Task Organization

Feedstock → Preprocessing → Conversion

Task 2: Feedstock Variability

Task 5: Preprocessing

Task 6: Conversion High-Temp

Task 1: Materials of Construction

Task 7: Conversion Low-Temp

Task 3: Materials Handling

Enabling Tasks

Task X: Project Management

Task 4: Data Integration

Task 8: TEA/LCA

Task X: Project Management: Provide scientific leadership and organizational project management

Task 1: Materials of Construction: Specify materials that do not corrode, wear, or break at unacceptable rates

Task 2: Feedstock Variability: Quantify & understand the sources of biomass resource and feedstock variability

Task 3: Materials Handling: Develop tools that enable continuous, steady, trouble free feed into reactors

Task 4: Data Integration: Ensure the data generated in the FCIC are curated and stored – FAIR guidelines

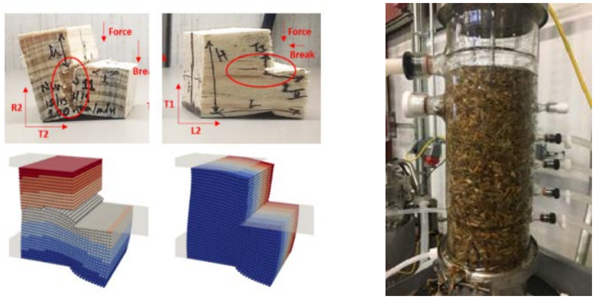
Task 5: Preprocessing: Enable well-defined and homogeneous feedstock from variable biomass resources

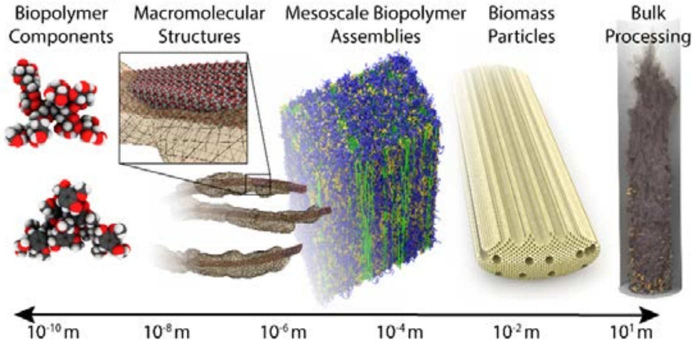
Task 6 & 7: Conversion (High- & Low-Temp Pathways): Produce homogeneous intermediates to convert into market-ready products

Task 8:Crosscutting Analyses TEA/LCA: Valuation of intermediate streams & quantify variability impact

Project Overview

- **Objective:** Develop science-based design and operation principles informed by TEA/LCA that result in predictable, reliable and scalable performance of preprocessing unit operations (comminution, fractionation, deconstruction, and real-time imaging)
- **Current Limitations:** Current rule-of-thumb/empirical methods are inadequate for real-world feedstocks – need to develop and implement experimentally-informed mechanistic models to enable reliable preprocessing that produce required quality attributes for conversion
- **Relevance:** Quantify, understand and manage variability in preprocessing operations through:
  - Experimentally-derived interactions of material attributes and process parameters
  - First principles models of comminution, fractionation, deconstruction, and imaging
  - Models to predict preprocessing performance and scalability
  - TEA/LCA Implications:
    - Impact of material attributes on comminution and fractionation
    - Impact of material attributes on equipment wear
    - Impact of material attributes on deconstruction reactor design/operation
- **Risks:** 1) Multi-dimensional, multi-scale problem; 2) Timely adaptation of preprocessing equipment for maximum impact; 3) Effective dissemination of tools and knowledge for maximum market impact





1 – Management

Subtask	Lead(s)	Major Responsibilities
5.1: Quality by Design for LT Preprocessing Unit Operations	Quang Nguyen, John Aston	Develop the relationship between MAs, PPs and QAs of corn stover for knife mills and fractionation
5.2: Quality by Design for HT Preprocessing Unit Operations	Neal Yancey, Jordan Klinger	Develop the relationship between MAs, PPs and QAs of woody biomass for multiple milling approaches and fractionation
5.3: Preprocessed Feedstock Bulk Transport Phenomena Modeling	David Sievers, Xiaowen Chen	Develop and validate physical/mechanistic models to predict consequences of MAs and PPs on bulk mass-transfer and kinetics in LT deconstruction (inform deacetylation TEA/LCA)
5.4: Integrated Experiment and Multiscale Simulation of Biomass Mechanics/ Fragmentation	Peter Ciesielski, Yidong Xia	Develop experimentally validated, multiscale computational framework that predicts the impacts of emergent feedstock variability on pre-processing for LT conversion processes.
5.5. Machine Vision for Feedstock Quality Identification	David Sievers	Develop automated machine vision and AI models to detect and quantify feedstock MAs in real-time to enable advanced process controls to manage feedstock MA variability

- **Risks:** Communication between experimentalists and modelers; communication between laboratories; communication with stakeholders and industry, complexity of the processes being studied
- **Mitigations:** Robust communication strategy developed with all partners and collaborators. Divided the project into manageable pieces (5 subtasks) to tackle the complexity

1 – Management (continued)

- **Communication strategy: FCIC cross-task collaborations:**
  - FCIC Pis, Task Leads, LRM and BETO TMs monthly meetings
  - Biweekly subtask meetings at each laboratory
  - Quarterly on-site meetings of entire Task 5 team (moved to virtual meetings in response to Covid-19)
  - Joint milestones with other Tasks
  - Coordination with Task 5 team members on other FCIC tasks
    - Task 1 – Vicki Thompson and Rick Elander
    - Task 2 – Amber Hoover and Luke Williams
    - Task 3 – Yidong Xia and Jordan Klinger
    - Task 6 – Jordan Klinger
    - Task 7 – Xiaowen Chen
    - Task 8 – David Thompson
  - Participation with lab-industry partnerships (DFO projects)
  - FCIC topical webinar presentations

2 – Approach

Technical Approach:

- Develop models guided with experimental data and utilize these models to predict process scaling.
- Collecting experimental data on the affect of material attributes and process parameters on quality attributes so that criticality can be assessed.
  - Knife mill – 9 MA, 5 PP, 10 QA related to flow and 6 QA related to conversion
  - Fractionation – 6 MA, 3 PP and 4 QA
  - Deacetylation – 6 MA, 7 PP and 5 QA related to conversion
- Generate process data (i.e., throughput, energy requirements, etc.) to inform TEAs/LCAs

Challenges:

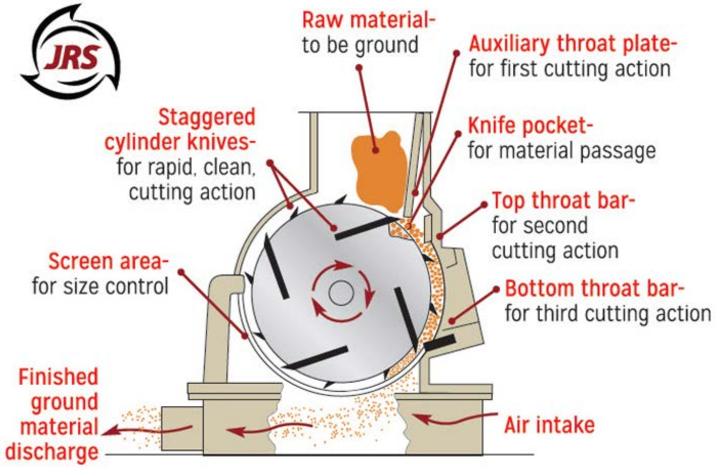
- Identifying and maintaining focus on the most critical attributes and process parameters
- Translating knowledge derived from first-principles models to novel equipment designs

Metrics:

- FY20 Go/No Go – Match model predictions for grain failure, deconstruction rate, and deconstruction energy within 80% (79-96%)of experimentally measured parameters. Demonstrate that the packed bed permeability model can predict pressure drop changes as a function of liquid feed rate per column cross section within 80% (82-100%) of predicted differences
- FY20/21 Corn stover preprocessing fractionation – demonstrated that anatomical fraction attributes have >10% (up to 29%) difference in packed bed flow permeability for initial conversion (deconstruction) unit operation

Impact:

- Preprocessing equipment designs based on first-principles knowledge of key material attributes will increase operational reliability and feedstock quality attributes
- Subsequent conversion unit operations will benefit from predictable material attributes and controllable process parameters that result in improved conversion performance and reliability



Dissemination:

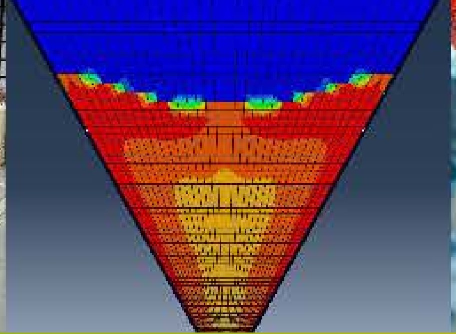
Near term:

- Peer-reviewed journals and trade journals
- Open-source model codes
- Conference presentations

Long term:

- Demonstrate applicability of knowledge and tools to industrial stakeholders
  - Equipment developers, process providers, etc.
- Incorporate design aspects and control capabilities to mitigate feedstock variability impacts to next-generation deacetylation equipment designs






4 – Progress and Outcomes

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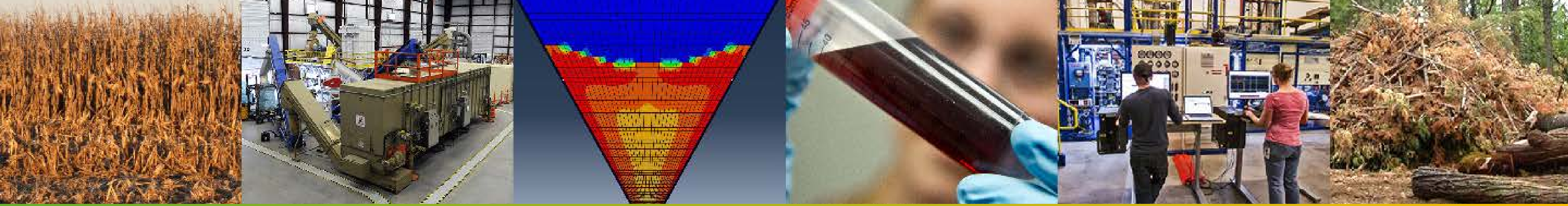

Milling – Experiments

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Shear Fracture Analysis of Pine

Knowledge

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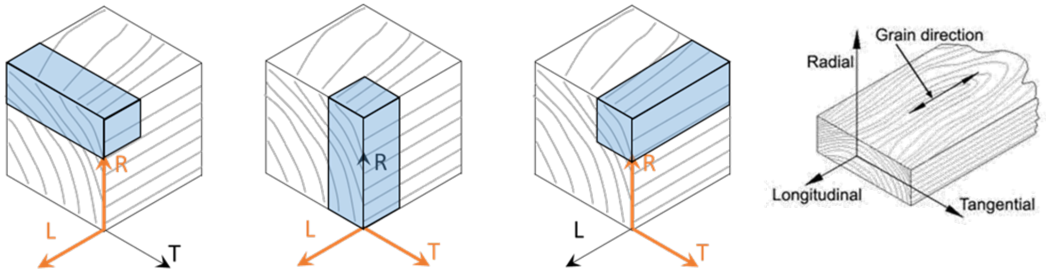
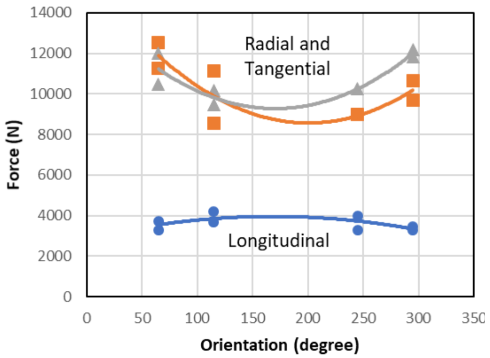
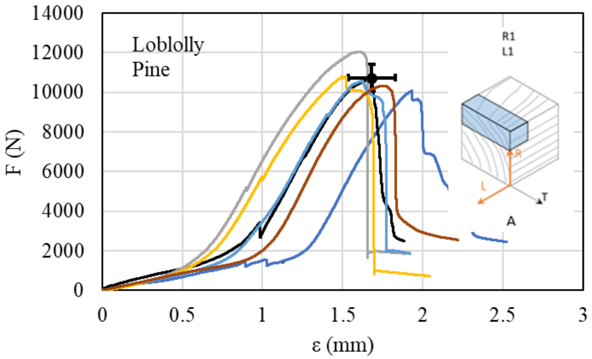
- **Shear failure of wood is the most efficient and preferred deconstruction mechanism**
- **Fundamental test quantify material shear and failure with respect to grain orientation and material attributes.**
- **Manuscript submitted to Powder Technology**







Current knowledge gap

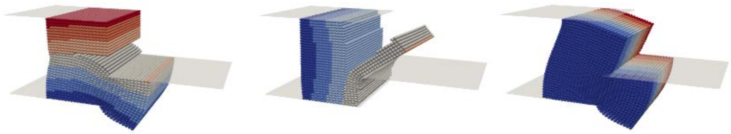
Fracture mechanisms for pine particles during milling are unknown and vary depending upon orientation of the grain to the impact.

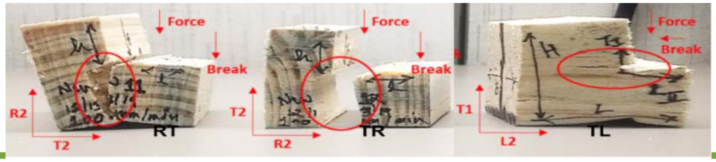
Achievement

Single particle tests to quantify the impact of material attributes (size, moisture content, grain orientation) on the force and fracture mechanisms needed to break particles. These data were utilized to parametrize Discrete Element Model (DEM) simulations and statistical models to predict milling performance.

Relevance

Biorefinery operators currently use “rule of thumb” and empiricism. Knowledge gained here contributes to milling models that will allow simulation at industrial scale to examine the impacts of changing CMAs and CPPs during size reduction. The coupled experimental-modeling strategy will be utilized to optimize mill design and operation





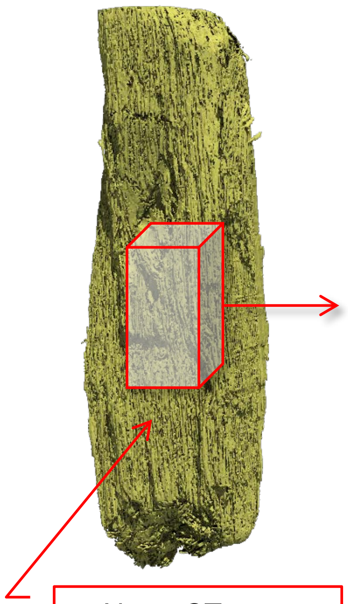
INL Idaho National Laboratory

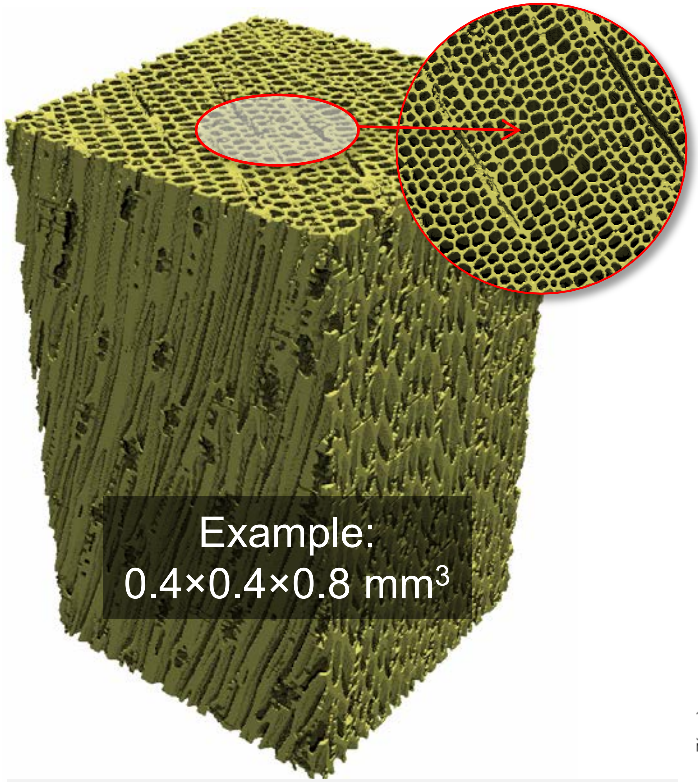
NREL NATIONAL RENEWABLE ENERGY LABORATORY

Enabling Microstructural Fracture Modeling

Tools

Developed XCT-informed loblolly pine 3D microstructural topology reconstruction workflow for fracture physics & models





Nano-CT scan

Reconstructed 3D microstructure

Description

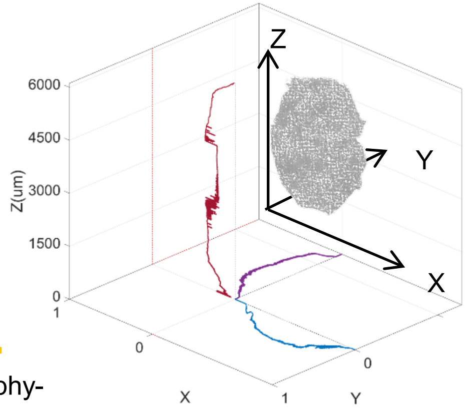
- Developed an XCT-informed microstructural topology reconstruction method for providing biomass microstructures

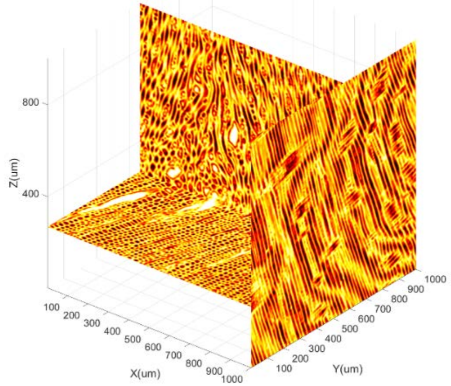
Value of new tool

- First-of-its-kind virtual laboratory for biomass micromechanics
- Microstructural mechanics DEM model will be open-source
- An intermediate-scale model links mesoscale model and macroscale model in the multis-scale model plan (NREL/INL)

Potential Customers & Outreach Plan

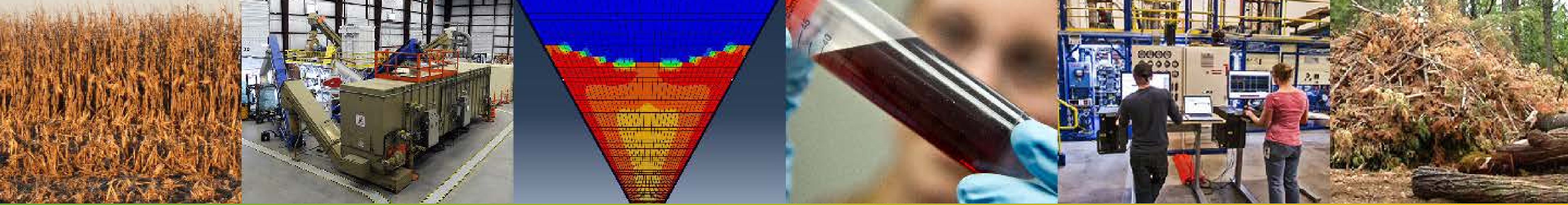
- Feedstock preprocessing industry partner, biorefinery designers
- Journals, conference presentations (ASABE, AIChE, etc.)





Advanced 3D analysis model reveals heterogeneous porosity distribution in pines (for the first time)

Q. Sun, Y. Xia, Q. Chen, J. Klinger, V. Thompson, “3D tomography-informed porosity analysis for woody biomass,” (in preparation)



Milling – Modeling

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Population Balance Model

Tools

- **Population balance of hammer-milled loblolly pine in ‘once-through’ and ‘fractional milling’ configurations**
- **Presented at AIChE Annual meeting in Feedstock Logistics for Biorefineries**
- **Manuscript in preparation**

Description

Model that predicts milled particle size distribution (PSD) as a function of tip speed, feed rate, initial PSD, screen size and moisture. Links single particle model to behavior observed (multiple particle impacts, etc) during milling operations. This provides extension of fundamental tests to prediction of mill performance.

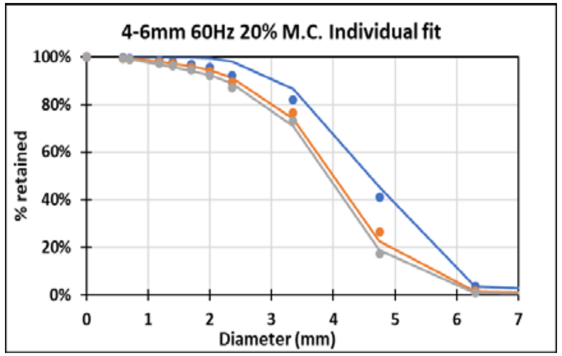
Value of new tool

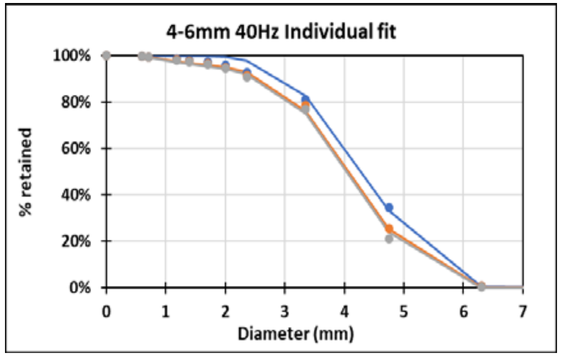
Biorefinery operators currently use “rule of thumb” and empiricism Simulation at industrial scale will identify CMAs and CPPs during size reduction and a tool to optimize mill design and to guide mill operation

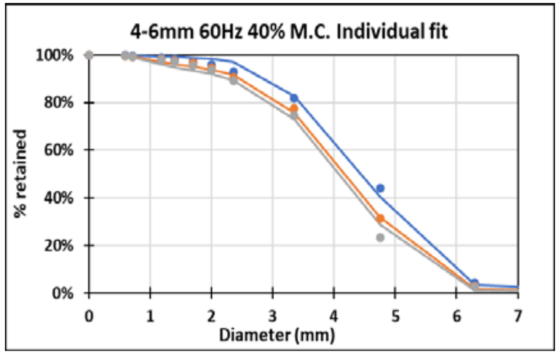
Potential Customers & Outreach Plan

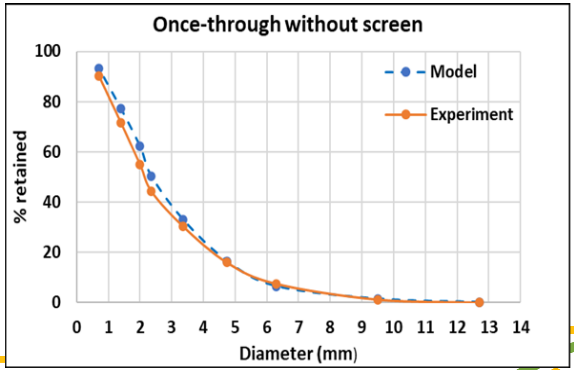
Mill operators and mill designers

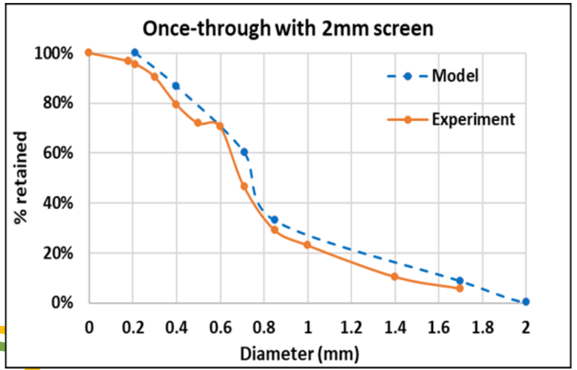
Publication, trade shows and public release of code

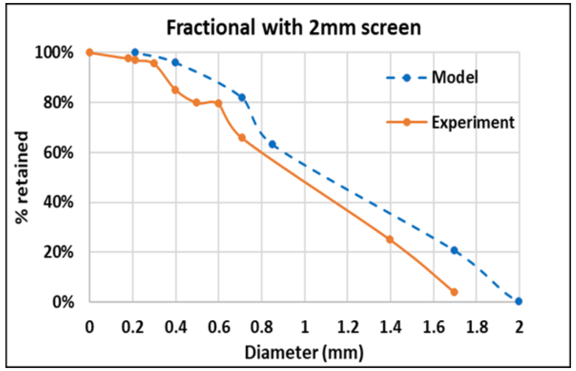










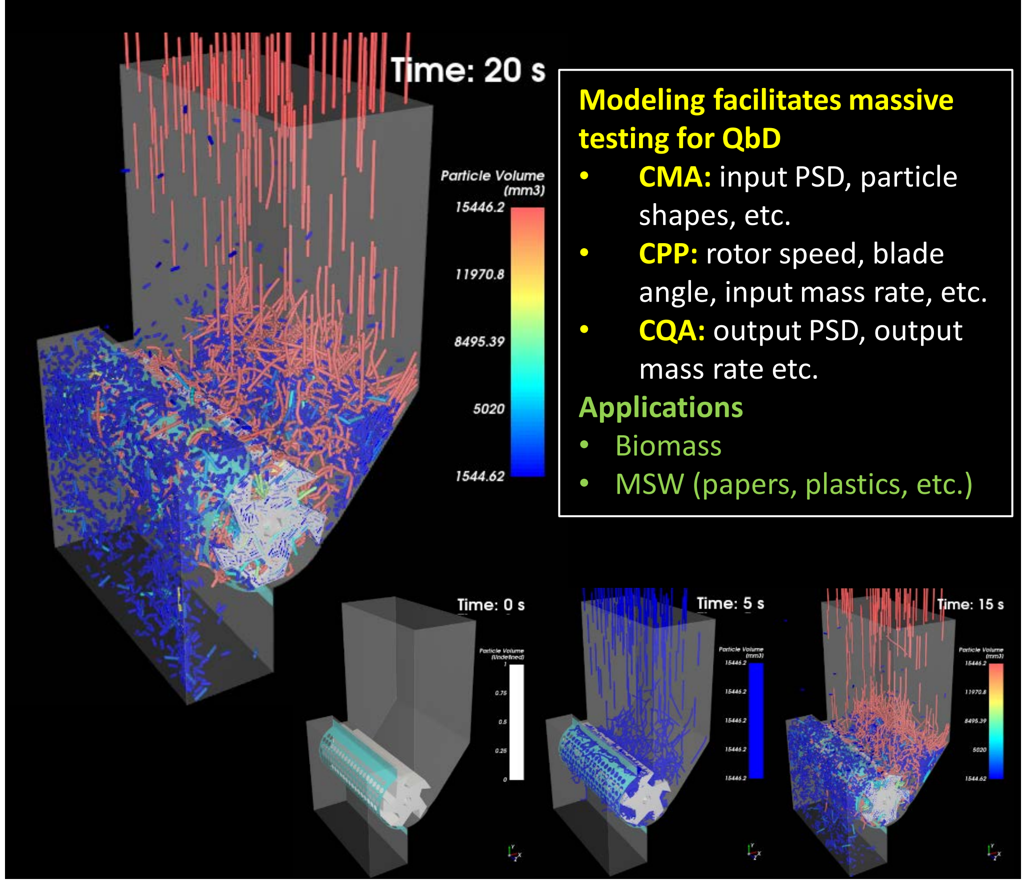


Pilot-scale Milling Deconstruction Model (1)

Tools

Developed experiment-validated pilot-scale knife milling model

Application example: JRS knife milling of corn stalks



Description

- Developed a pilot-scale knife milling model (DEM particle flow and deconstruction) for supporting Quality-by-Design (QbD)

Value of new tool

- First-of-its-kind virtual laboratory for knife milling process model
- Enabled fast massive testing and real-time diagnosis for design of optimal knife milling process for feedstocks of interest
- Rapid assessment of the impacts of material attributes and process parameters without having to conduct expensive experimentation

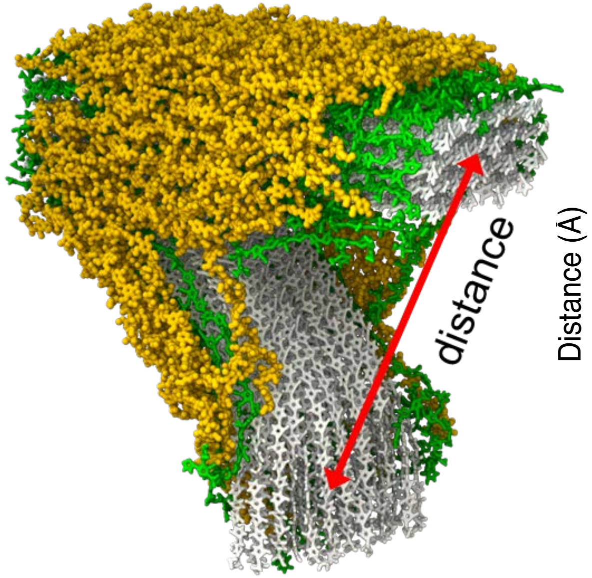
Potential Customers & Outreach Plan

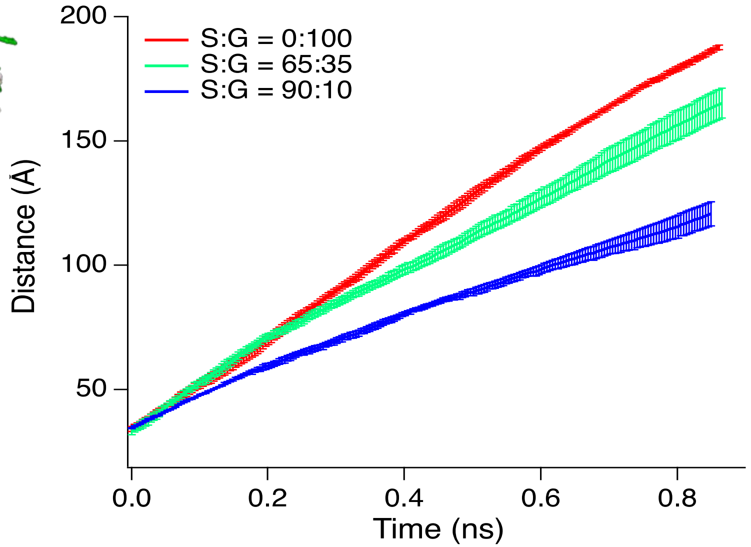
- Feedstock preprocessing industry partner, biorefinery designers
- Journals to attract industry and academic collaboration
- Conference presentations (ASABE, AIChE, etc.)

Coarse-Grained Modeling of Lignocellulose Mechanics

Knowledge

Understanding how forces propagate through lignocellulose at the molecular scale





L-J potential	xylan/s-lig	xylan/g-lig	xylan/h-lig	s-lig/s-lig	g-lig/g-lig	h-lig/h-lig
ε (kcal/mol)	24.27	20.91	15.35	24.10	22.00	17.31
r_m (Å)	4.13	4.53	3.95	4.76	3.43	4.49
r_0 (Å)	1.27	1.34	2.95	2.23	2.39	2.64

Current Knowledge Gap

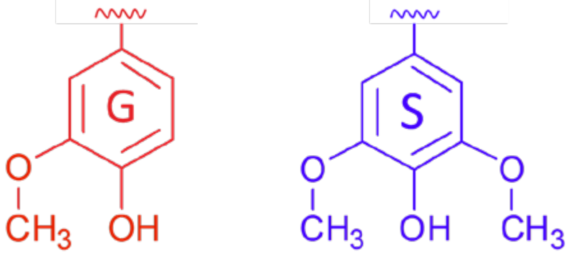
Lignocellulose is a complex intertwining of three biopolymers (cellulose, hemicellulose and lignin). Lignin is a random polymer of syringyl (S), guaiacyl (G) and p-hydroxyphenyl (H) subunits. The ratios of these subunits can vary markedly in different types of biomass and it is not known how these ratios impact physical properties of lignocellulose.

Achievement

- Increasing the number of methoxy groups on lignin subunits increases the interaction strength with other biopolymers.
- Molecular dynamics simulations clearly show that higher syringyl/guaiacyl (S/G) ratios require more mechanical energy to deconstruct

Relevance

This finding could potentially be exploited by genetic engineering strategies for designer biomass feedstocks that are designed to minimize energy inputs in conversion processes.



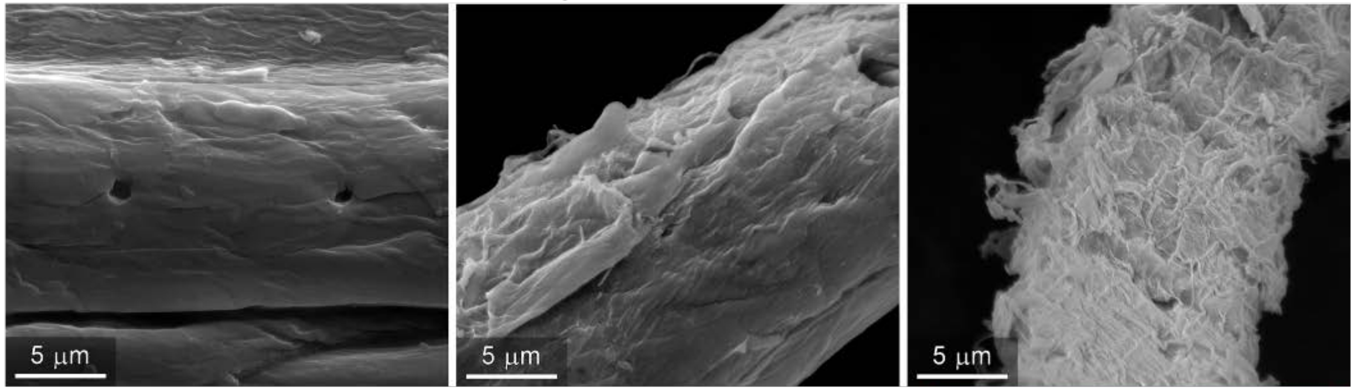
Coarse-Grained Modeling of Lignocellulose Mechanics

Knowledge

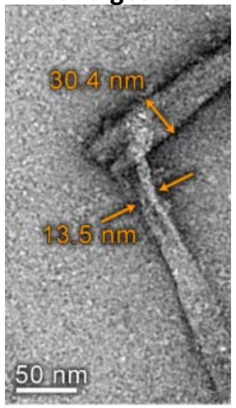
Biomass surfaces that exhibit kink defects are much more amenable to enzymatic hydrolysis

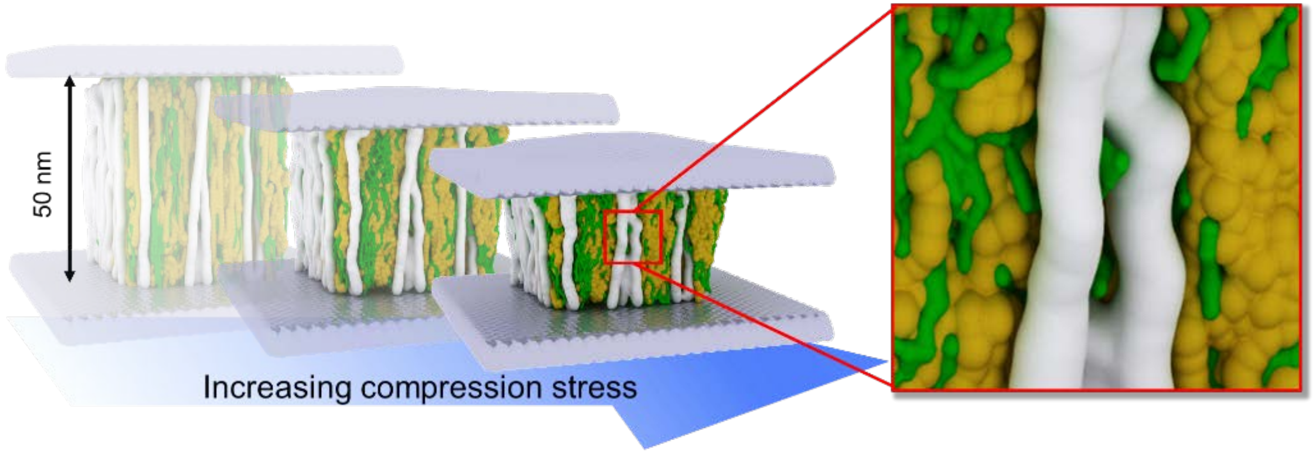
Before digestion

Deacylated | Deacylated + Disk Refined | Deacylated + Disk Refined + Szego Milled



After digestion





Current Knowledge Gap

Enzymes initiate hydrolysis at macromolecular kink defects[1]
Although mechanical treatments exist that are known to produce biomass surfaces with kinked cellulose and produce highly digestible biomass[2], it is not known what is occurring at the molecular scale during kink formation.

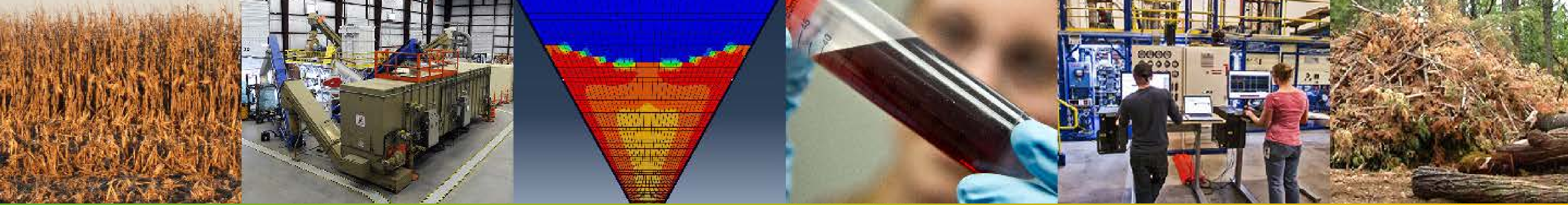
Achievement

Our coarse-grained simulation predicts formation of kink defects in lignocellulose when nano-mechanical stress exceeds a certain threshold

Relevance

Coarse-grained simulation can be utilized to identify modes of mechanical processing that maximize formation of "reactive defects" while minimizing energy requirements

[1]Ciesielski, et al. PNAS, 2019; [2]Chen, et al. ACS Sus. Chem. Eng., 2016

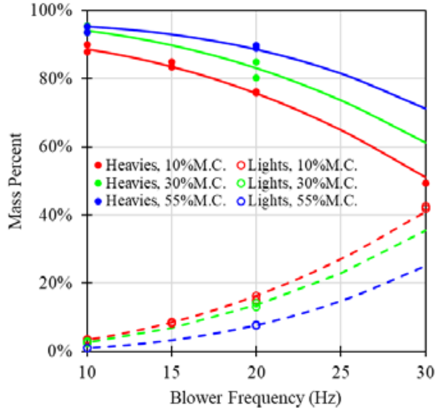


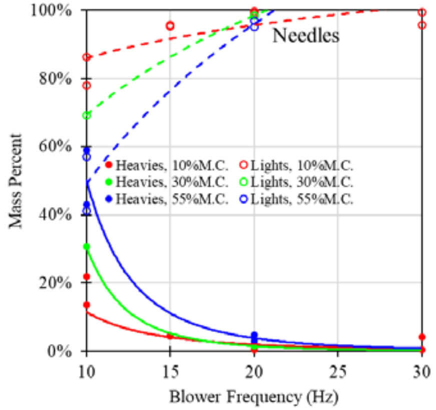
Fractionation – Air Classification

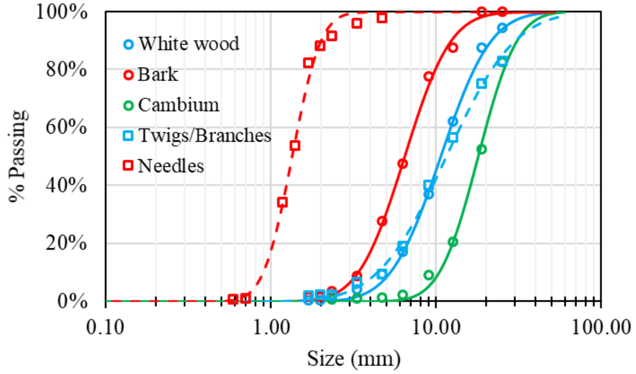
Air Fractionation for Anatomical Separation

Tools

- **Anatomical separation of corn stover and loblolly pine residues using air fractionation**
- **Manuscript in preparation**







Description

Air fractionation system to separate materials based on buoyancy which encompasses differential material properties such as density, shape, surface area, size and roughness found in plant anatomical fractions.

Value of new tool

Currently the entire plant is utilized and differences in properties result in variability during pretreatment, conversion and upgrading. This tool allows anatomical fractions to be enriched and processed differently if necessary.

Potential Customers & Outreach Plan

Next generation biorefineries

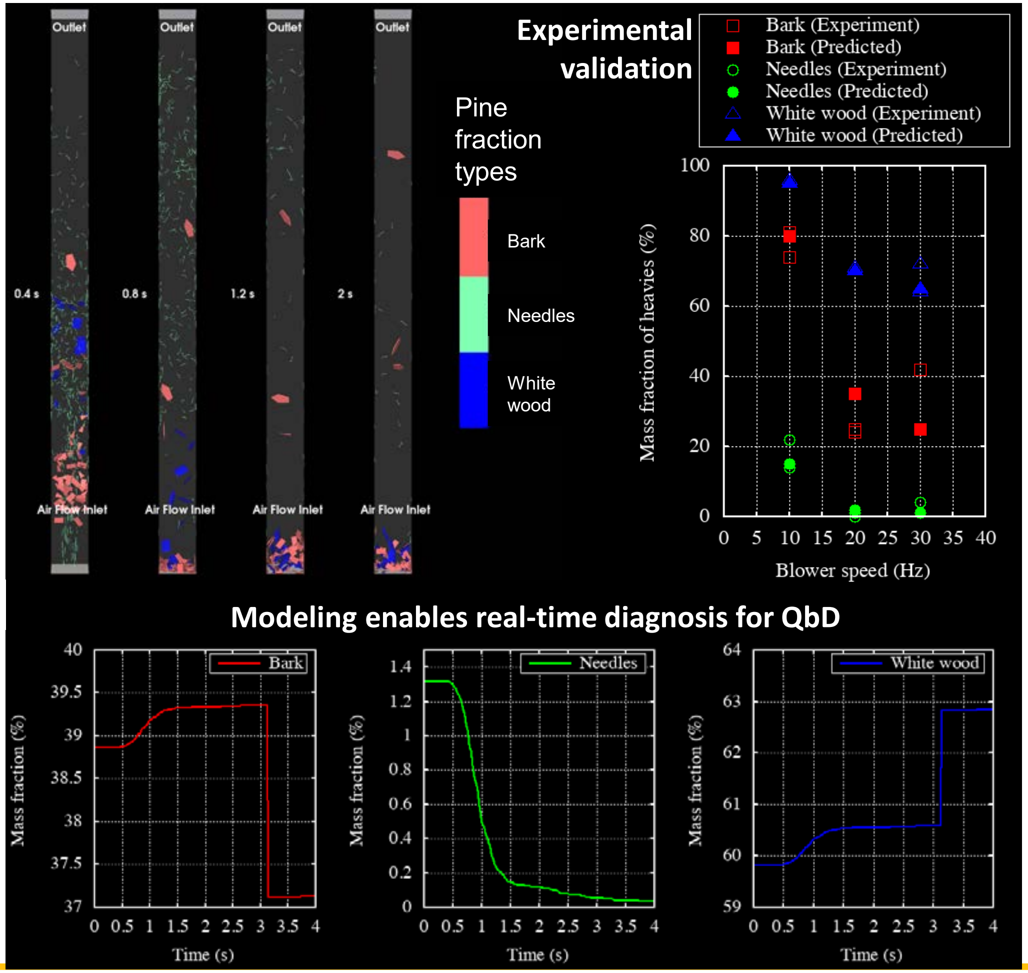
Publish the results of experimental studies and TEA/LCA studies to demonstrate the benefits of this approach.

Print version

Pilot-scale Air Classification Model

Tools

Developed experiment-validated particle-air model for predicting air classification



Description

- Developed a pilot-scale air classification model (DEM-CFD (particle-air) two-way coupling) for supporting QbD

Value of new tool

- First-of-its-kind virtual laboratory for air classification
- Enabled fast massive testing and real-time diagnosis for design of air classification

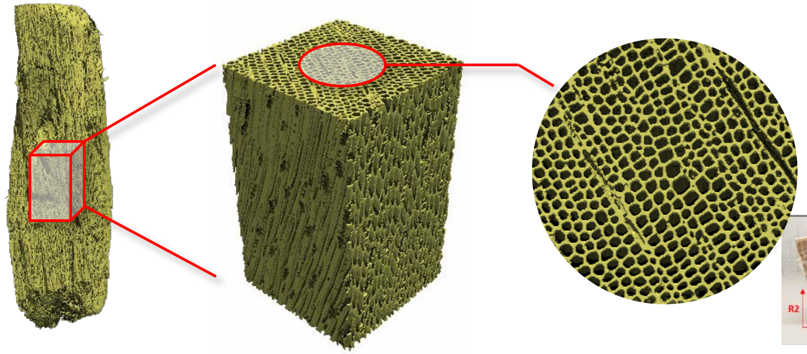
Potential Customers & Outreach Plan

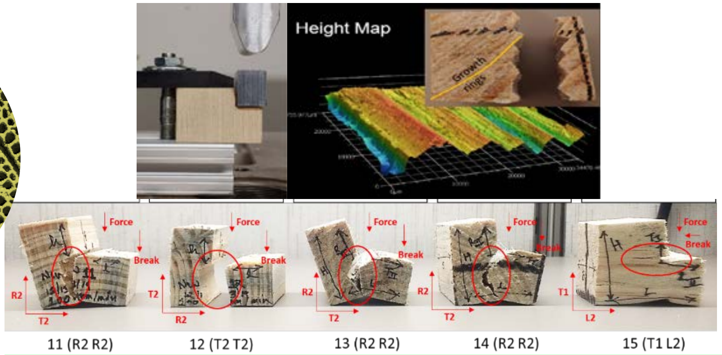
- Feedstock preprocessing industry partner, biorefinery designers
- Journals to attract industry and academic collaboration
- Conference presentations (ASABE, AIChE, etc.)

Virtual Laboratory for Preprocessing

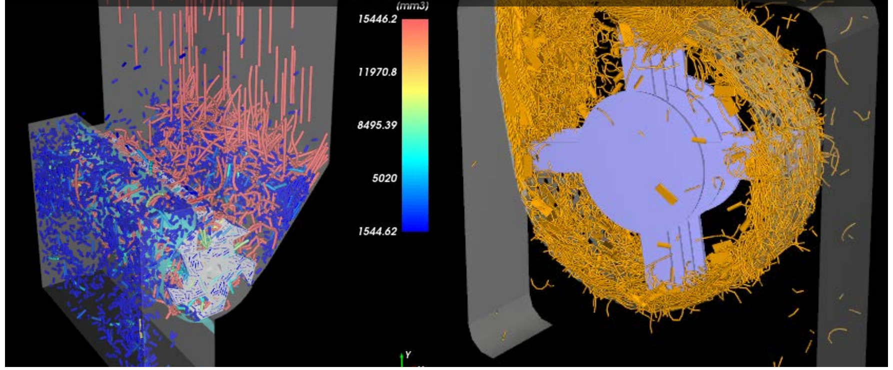
Tools

XCT-informed microstructural deconstruction model and upscaling



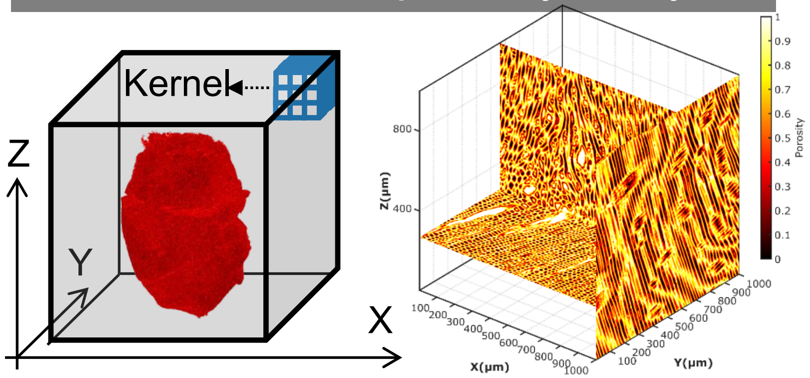


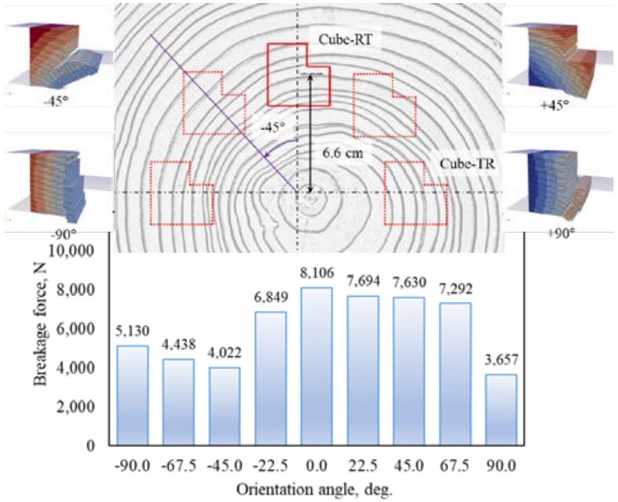
Modeling of various milling techniques



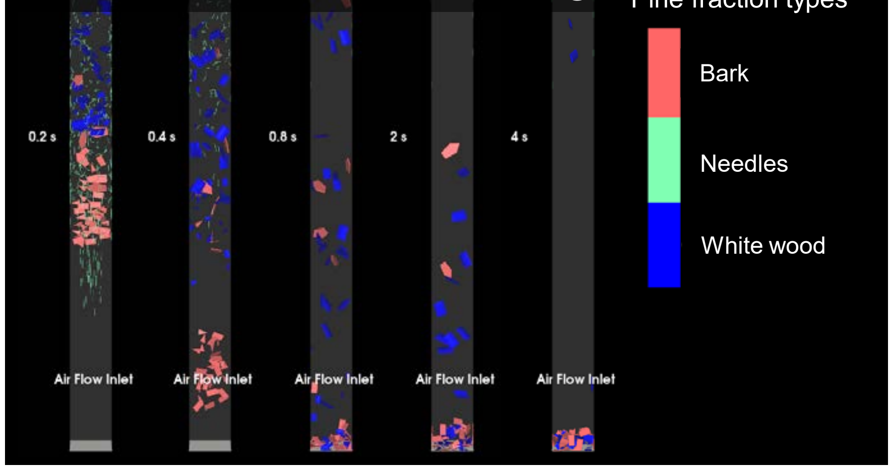
Microscale (um) → **Laboratory scale (cm)** → **Pilot scale (m)**

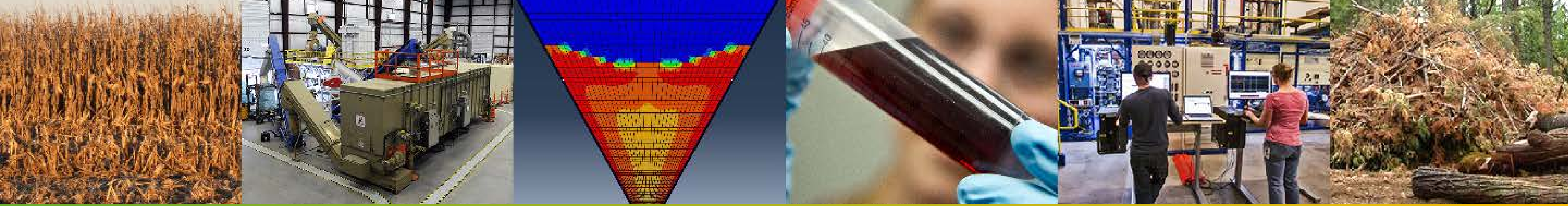
Advanced micro-porosity analysis





Air classification modeling



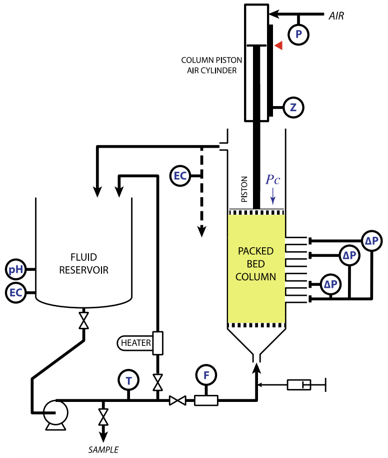


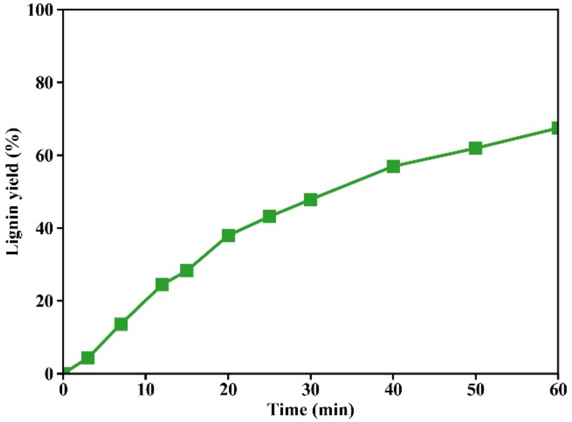
Deconstruction - Deacetylation

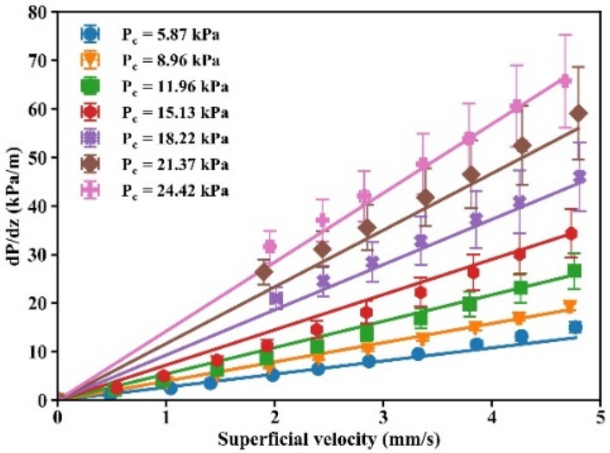
Differential-Compaction Packed-Bed Flow Column for Deacetylation CMAs

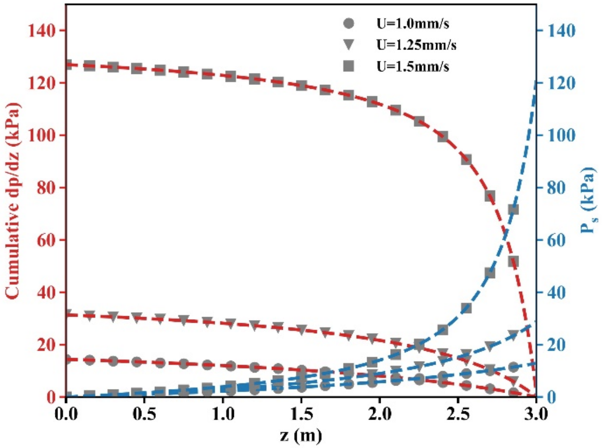
Tools

Packed bed column reactor test platform and models for deacetylation and similar processes









Description

- Commissioned a packed-bed flow column system with temperature control and pressure gradient measurement for flow-through deacetylation process.
- Developed a mechanistic model to predict permeability and flowability implications relevant to industrial processing
- Developed *compressible* packed-bed model for industrially relevant reactor designs and for TEA/LCA.

Value of new tool

- Integrated real-time instrumentation monitoring and recording of actual deacetylation process as a function of MAs and PPs
- A physics-based mechanistic model was developed and validated to predict the packed-bed behavior for scale-up of column reactor designs and optimization

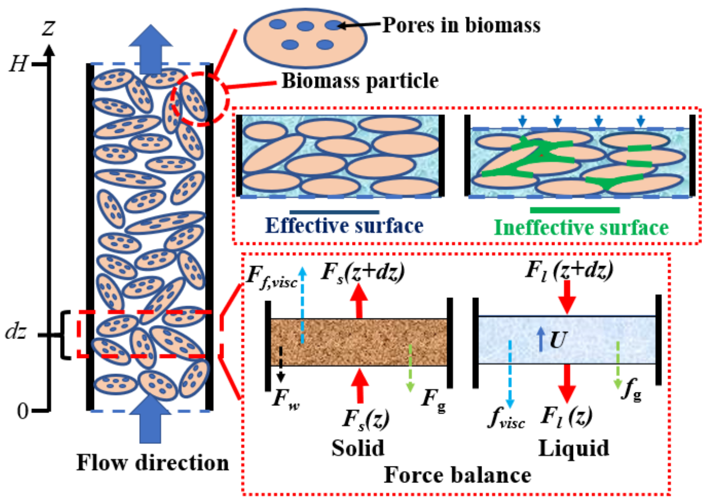
Potential Customers & Outreach Plan

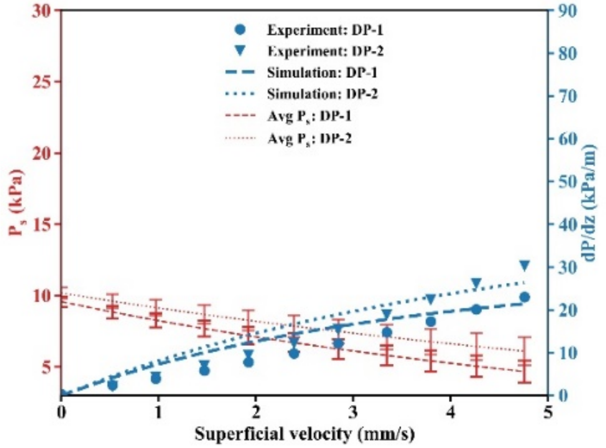
- Feedstock preprocessors (CMAs identified for specifications)
- Deacetylation reactor equipment engineers and plant operators
- Publications (2) expected in FY21

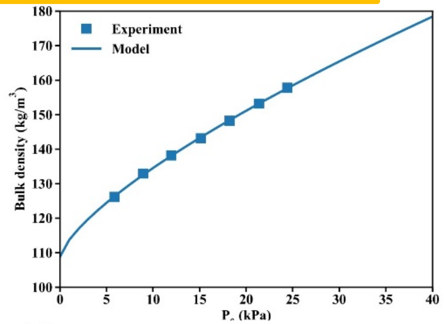
Compressible Packed-Bed Processes: from 1st-principles to Industrial Designs

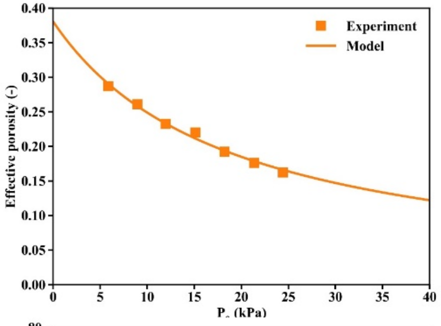
Knowledge

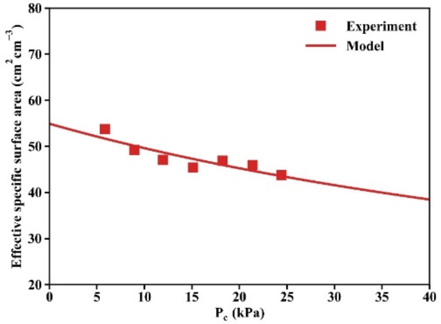
Compressible biomass packed bed flow phenomena and impacts on the process











Current Knowledge Gap

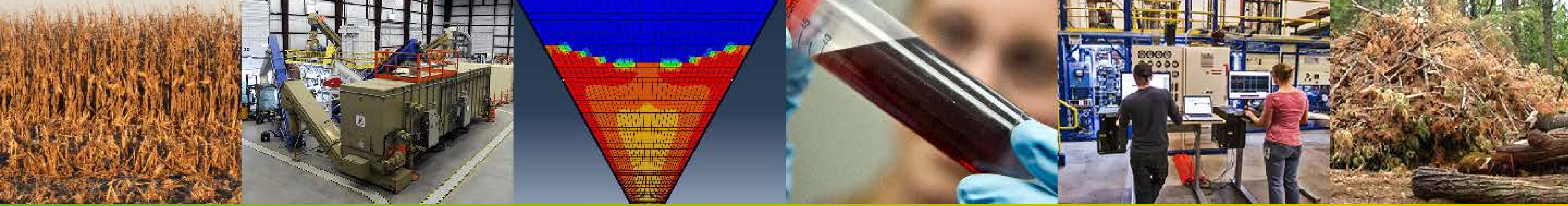
- Fundamentals of transport-controlling factors of compressible herbaceous biomass are not well understood.
- Relationships between MAs and herbaceous packed bed properties unknown.
- Effects of PPs on the dynamics of herbaceous packed bed unknown, but important for deacetylation and similar processes.

Achievement

- We proposed and validated an important modification to the classical Kozeny-Carman equation to account for biomass compressibility.
- A physics-based compressible packed bed model was developed and verified to predict system dynamics and is used to perform scale-up design optimizations.
- Air classification effect (preprocessing) on the packed bed permeability and the subsequent effect on the industrial relevant reactor design were studied.

Relevance

- De-risk deployment of deacetylation and washing reactors for herbaceous biomass processing in biorefinery industry.
- Scale-up design/optimization of industrially-relevant reactors for processing biomass feedstocks.

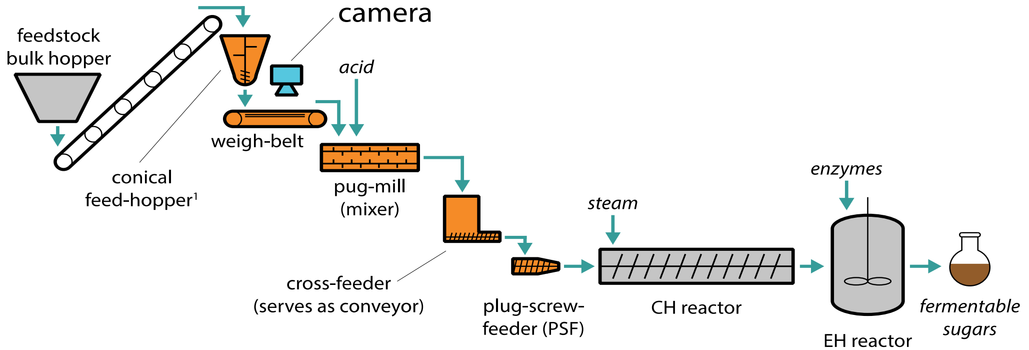


Process Control – Machine Learning

Real-Time Feedstock Image Analysis Model

Tools

Automated machine vision technique and models to detect and quantify corn stover feedstock particle quality in real-time and enable automatic decisions that can be made by advanced processing controls



[1]conical feed-hopper cyclically refilled every ~30 min

Description

- Utilized a 26,000 image dataset from processing corn stover in a pretreatment reactor captured using inexpensive digital cameras.
- Neural Network (NN) and Pixel Matrix Feature Parameterization (PMFP) approaches developed.

Value of new tool

- Neural Network (NN) model can detect anomalies (coarse-particle segregation that can cause feed interruption) even when camera lens obscured by dust.
- PMFP method reveals statistically significant image textural features such as surface roughness, shade variations, and particle angular direction variations that are proxies for particle size distribution variation.
- NN and PMFP approaches are complementary to one another and can describe why feedstock images are classified a certain way.

Potential Customers & Outreach Plan

- Broad applicability to other unit operations where continuous feeding images can be gathered (FCIC 2.4, 3.5, 5.1, and 5.2).
- INL/NREL collaboration on color channel model for predicting self-heating of feedstock based on photographic images.
- Journal Article: *Real-Time Biomass Feedstock Particle Quality Detection Using Image Analysis and Machine Vision.* http://dx.doi.org/10.1007/s13399-020-00904-w

Summary

Management:

- Met or exceeded all project milestones and Go/No-Go decision points
- Successful dissemination of results through presentations, publications and industrial communications
- Maintained coordination within Task 5 and between other FCIC tasks

Technical Approach:

- Develop first principles models guided with experimental data and utilize these models to predict process scaling.
- Collected data on material attributes and process parameters and how they impact quality attributes
- Generated process data (i.e., throughput, energy requirements, etc.) to inform TEAs/LCAs

Impact:

- Preprocessing equipment designs based on first-principles knowledge rather than rule of thumb
- Conversion unit operations will benefit from well controlled material attributes

Progress:

- Met all project milestones
- Published in high impact journals and conferences
- Successfully developed comminution, deconstruction and imaging models that predict experimental performance at greater than 80% accuracy.

Quad Chart Overview- FCIC, Task 5

Timeline

- 10/1/2018 - 9/30/2021

	FY20	Active Project
DOE Funding	$1760K	FY19- $2300 FY20- $1760 FY21- $1660 Total- $5720

Project Partners (N/A)

Barriers addressed

19Ft-J FSL Operational Reliability
19Ct-A CONV Defining Metrics around Feedstock Quality
19Ct-B CONV Efficient Preprocessing and Pretreatment

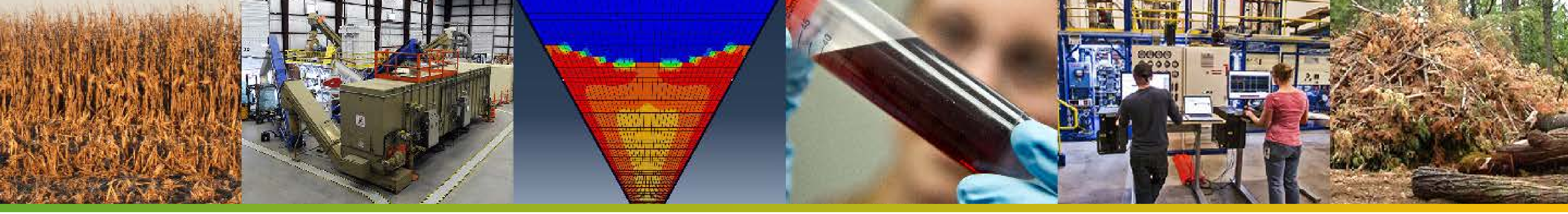
Project Goal

Develop science-based design and operation principles informed by TEA/LCA that result in predictable, reliable, and scalable performance of preprocessing unit operations. Develop a set of modeling tools that predict how material attributes of corn stover and pine residues and process parameters of milling, size classification and deacetylation interact to produce quality attributes required by high temperature and low temperature conversion.

End of Project Milestone

Demonstrate a multi-scale model (macro-scale and micro-scale) of fragmentation behavior in response to shear and compression from first principles. Construct a pine particle scale model with microstructure geometry obtained from loblolly pine XCT data. Verify that the simulation can predict the fracture stress within 15% of experimental measurements.

Funding Mechanism (N/A)



Thank you

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NREL/PR-5100-80649

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FEEDSTOCK-CONVERSION
INTERFACE CONSORTIUM

Publications, Patents, Presentations, Awards, and Commercialization

- Suna, Quan, Yidong Xia, Jordan Klinger, Robert Seifert, Joshua Kane, Vicki Thompson and Qiushi Chen. 2021. X-ray computer tomography-based porosity analysis: Algorithms and application for porous woody biomass. Submitted to Bioresource Technology Feb. 18, 2021.
- Guo, Yuan, Q. Chen, Y. Xia, J. Klinger, V.S. Thompson. 2021. A nonlinear eastoplastic bond model for DEM modeling of biomass comminution. Powder Technology Accepted March 4.
- J. E. Jakes, S. L. Zelinka, C. G. Hunt, P. N. Ciesielski, C. Frihart, D. Yelle, L. Passarini, S. C. Gleber, D. Vine, S. Vogt. "Measurement of moisture-dependent ion diffusion constants in wood cell wall layers using time-lapse micro X-ray fluorescence microscopy." *Under review*.
- Sievers, D., E. Kuhn, V. Thompson, N. Yancey, A. Hoover, M. Resch, and E. Wolfrum. 2020. Throughput, reliability and yields of a pilot-scale conversion process for production of fermentable sugars from lignocellulosic biomass: A study on feedstock ash and moisture. Accepted to *ACS Sustainable Chemistry & Engineering*, January 16, 2020.
- Klinger, J., D. Carpenter, V. Thompson, N. Yancey, R. Emerson, K. Gaston, K. Smith, M. Thorson, H. Wang, D. Santosa and I. Kutnyakov. 2020. Pilot plant reliability metrics for grinding and fast pyrolysis of woody residues. Accepted to *ACS Sustainable Chemistry & Engineering*, January 16, 2020.
- Yuan Guo, Qiushi Chen, Yidong Xia (corresponding author), Jordan Klinger, Vicki Thompson. "A nonlinear elastoplastic bond model for DEM modeling of biomass comminution" (in preparation)
- Sinquefield, Scott, Peter N. Ciesielski, Kai Li, Douglas J. Gardner, and Soydan Ozcan. "Nanocellulose Dewatering and Drying: Current State and Future Perspectives." *ACS Sustainable Chemistry & Engineering* (2020).
- Gudavalli, C., Bose, E., Donohoe, B. S., Sievers, D. A. (2020). Real-Time Biomass Feedstock Particle Quality Detection Using Image Analysis and Machine Vision. *Biomass Conversion & Biorefinery*. http://dx.doi.org/10.1007/s13399-020-00904-w
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- Lintao Bu, Peter N. Ciesielski, Vivek Bharadwaj, Meagan F. Crowley, Bryon S. Donohoe, Michael E. Himmel, Paul Dupree, Munir Skaf, Carole Simoes, and Michael F. Crowley. Effect of Degree of Polymerization on Xylan Binding to Cellulose. *In preparation.*
- Quang Nguyen, How artificial intelligence and machine learning will lower the barriers to growing a bioeconomy? ABLC 2020.
- Yidong Xia, Yuan Guo, Jordan Klinger, Vicki Thompson, Qiushi Chen, "Understanding Pine Fracturing Mechanism: An Experimental and Discrete Element Modeling Study", presented at the ASABE 2020 Annual International Meeting, July 13-15.
- Tiasha Bhattarcharjee, Jordan Klinger, Neal Yancey and Vicki Thompson. "Population Balance of Hammer Milled Loblolly Pine in 'Once-through' and 'Fractional Milling' Configurations" AIChE 2020 Annual meeting.
- Yidong Xia. "Limitations and Best Practice for DEM Modeling of Biomass." International Powder & Bulk Solids Conference & Exhibition, The Powder Show Digital Flow (virtual), October 1, 2020.
- Yuan Guo, Qiushi Chen, Yidong Xia, Feiyang Chen, and Jordan Klinger, "A Nonlinear Elasto-Plastic Bonded-Sphere Model for the Discrete Element Simulation of Biomass Particle Breakage," 2020 Virtual AIChE Annual Meeting, November 16-20, 2020.