DOE Bioenergy Technologies Office (BETO) 2023 Project Peer Review

Enhanced Feedstock Characterization and Modeling to Facilitate Optimal Preprocessing and Deconstruction of Corn Stover

DE-EE0008907 FY19 BETO MultiTopic FOA

04-05-2023

Feedstock Technologies Program

David Hodge
Montana State University

Project Overview

- **Challenges**
  - Chemical and physical heterogeneity in herbaceous biomass feedstocks due to substantial differences in tissue types can contribute to challenges to handling, preprocessing, and conversion in biorefining processes
  - How to quantify this heterogeneity and potentially exploit these differences to facilitate more streamlined processing/preprocessing?

- **Project Approach**
  - Develop/adapt technologies for physical fractionation of corn stover
  - Develop/adapt new tools for characterization of fractionated corn stover

Project Overview

Corn Stover → Primary Fractionation → Stem-Rich Fraction → Secondary Fractionation → Pith-Rich Fraction

Primary Fractionation → Other Fractions, *e.g.*:
- Leaves, leaf sheaths, and husks
- Cobs
- Fines and ash

Secondary Fractionation → Other Fractions, *e.g.*:
- Nodes
- "Fibers"
- Rind
- Fines and ash

- **Develop Novel Heterogeneity Reducing Fractionation Technologies for Corn Stover**
- **Understand Relationship Between Preprocessing Conditions and Fraction Properties**

Idaho National Laboratory

Develop and Apply Novel Characterization Tools

Low-Field ^{1}H NMR Relaxometry
- Different pools of sorbed water

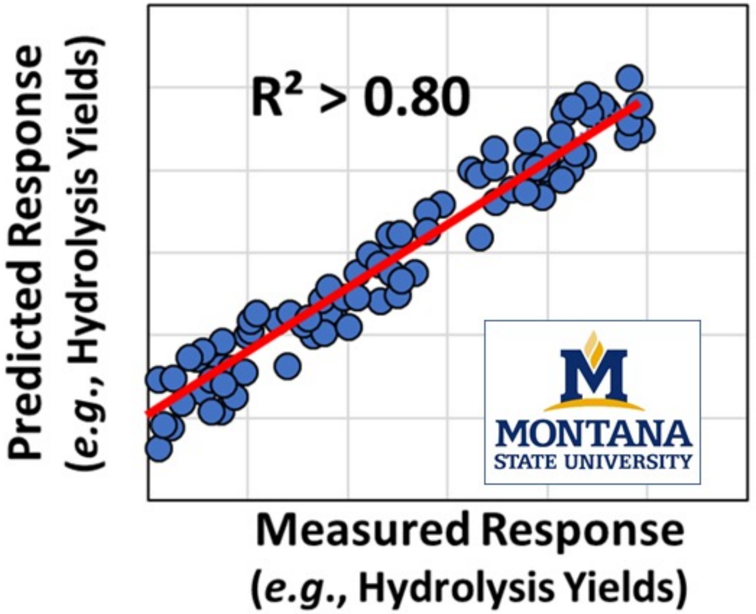
Water Retention Value
- Feedstock hygroscopicity

Dynamic Image Analysis
- Distributions of particle dimensions, classification of tissue types

MONTANA STATE UNIVERSITY

Develop and Validate Robust Models to Assess and Predict Processing Performance

- Preprocessing: Classification of Tissue Type
- Deconstruction: Prediction of Hydrolysis Yields



Project Overview

OBJECTIVE 1: Identify conditions for optimal fractionation using a two-stage physical fractionation

OBJECTIVE 2: Assess how physical fractionation impacts properties, partitioning of biomass, and response to processing

OBJECTIVE 3: Develop and validate advanced characterization tools for assessing biomass properties

OBJECTIVE 4: Develop and validate predictive models based on measurements to relate chemical and physical properties to processing behavior (preprocessing and deconstruction)

TASK 1. Initial Verification
- Work with DOE verification team to define baseline technology readiness level

TASKS 2, 4. Physical Fractionation of Corn Stover
- Fractionation by cell type for model development
- Shredding-comminution coupled to fractionation by sieving, air classification

TASKS 3, 5. Characterization of Fraction Properties
- Chemical composition (polysaccharides, lignin, ash)
- Water-biomass interactions (WRV, TD-NMR)
- Distribution of particle size, morphology, cell types
- Response to pretreatment and enzymatic hydrolysis

TASK 6. Model Development and Validation
- Correlation of characterized properties
- Development of models to assess preprocessing performance and predict responses to deconstruction
- Model validation in a relevant environment

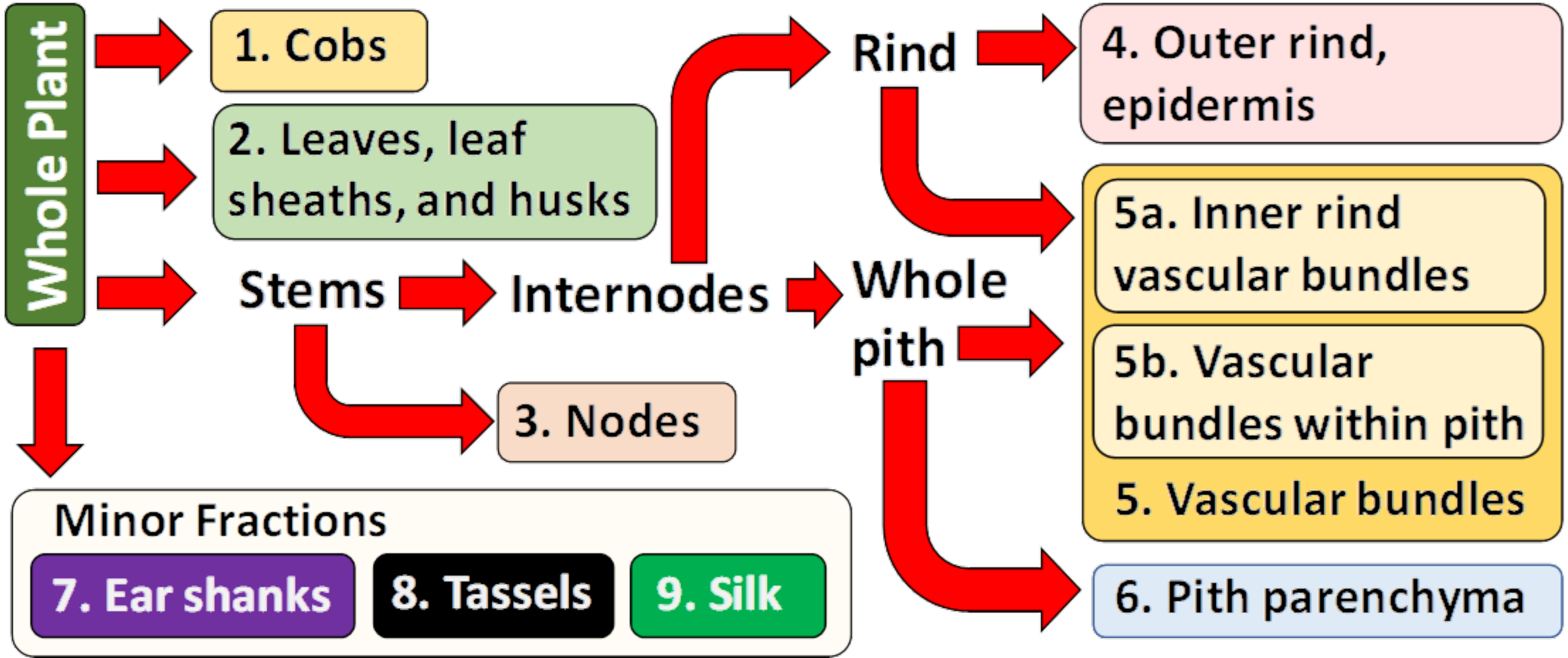
TASK 7. Project Management and Reporting
- Reporting submitted
- Participation in FY21 PEER Review

1 - Approach

- **Task 1**: Initial Verification → • Work with DOE verification team to define baseline technology readiness level

Go/No-Go Decision Point DP 1.1
✓ Complete

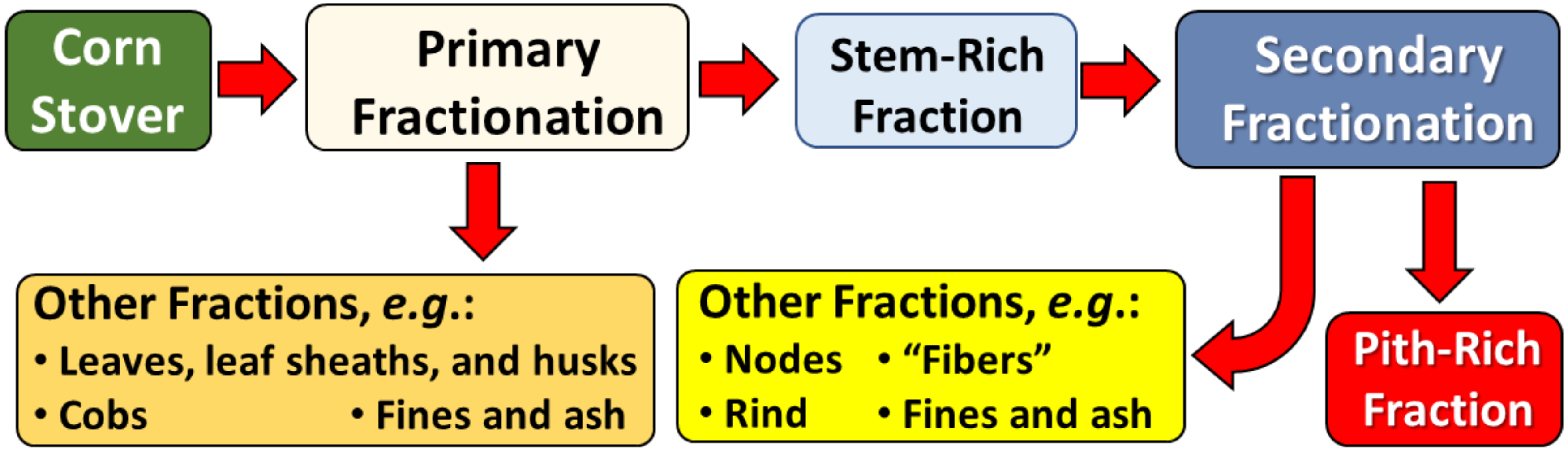
- **Tasks 2, 4** : Physical Fractionation of Corn Stover
  – Fractionation by cell type as reference set for model development



Milestone M2.1.1 : Manual Fractionation
✓ Complete

1 - Approach

- **Tasks 2, 4**: Physical Fractionation of Corn Stover
  - Shredding-comminution coupled to fractionation by sieving, air classification

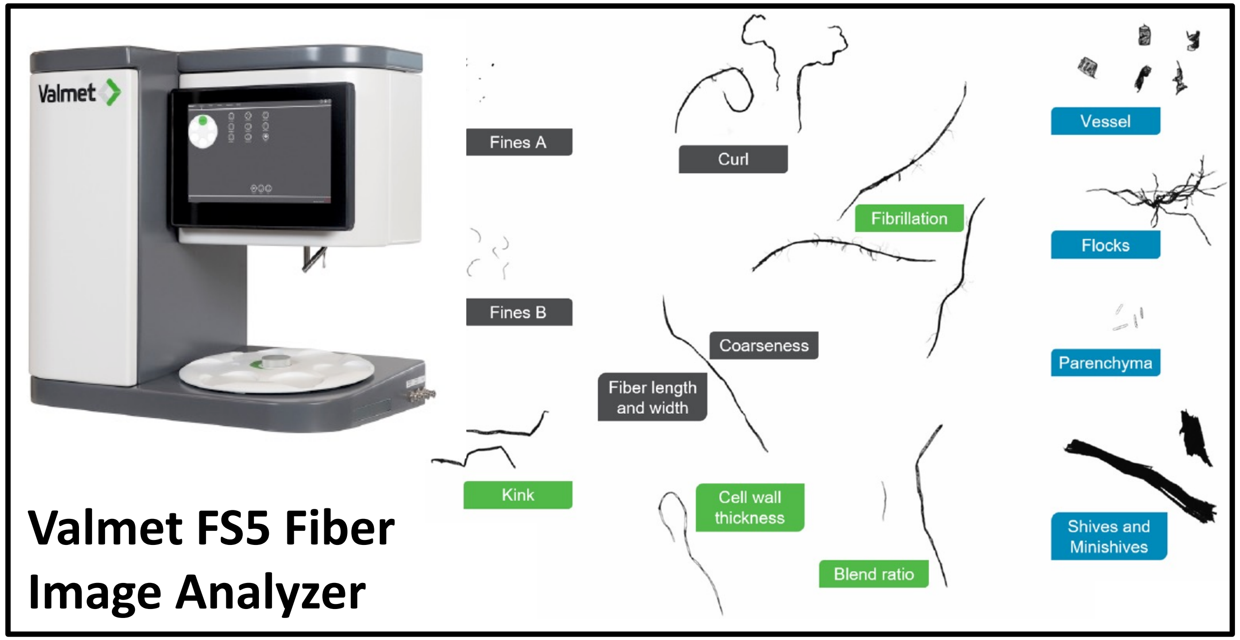


  - Primary Classification: Stem separation → **Stem Recovery at >75% yield and purity**
  - Secondary Classification: Pith separation → **Pith Recovery at >75% yield and purity**

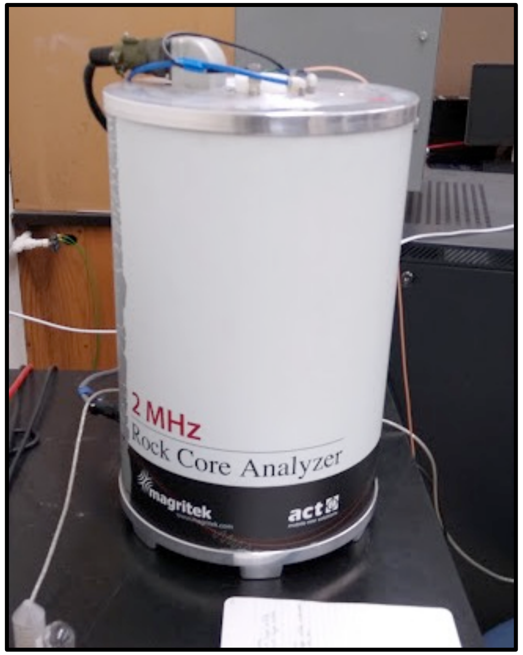
Go/No-Go Decision Point DP 2.2.1
✓ **Complete**

1 - Approach

- **Tasks 3, 5**: Property characterization of physically fractionated corn stover
  - Mass composition and component partitioning
  - Particle size, aspect ratio, and tissue type
  - Characterization of sorbed water in corn stover fractions
  - Assessing response of fractionated corn stover to processing

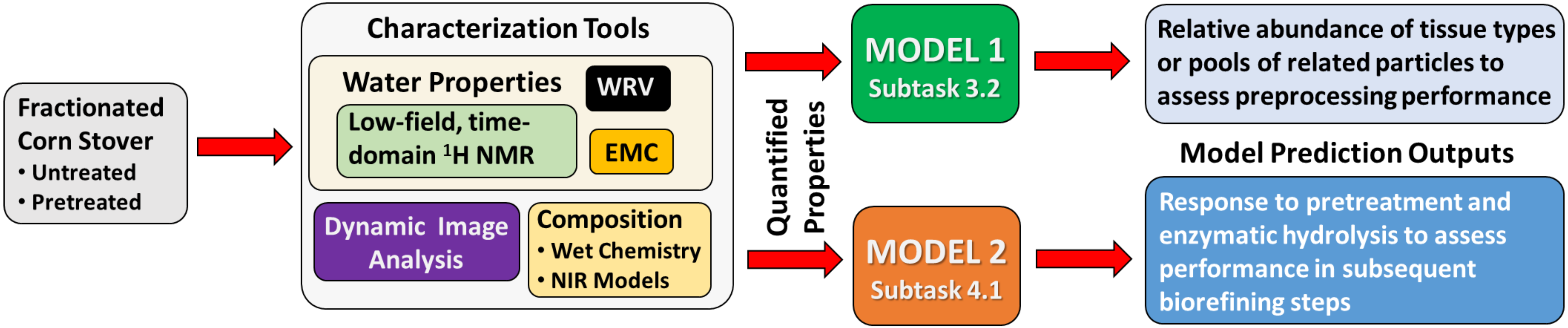


Valmet FS5 Fiber Image Analyzer



1 - Approach

- **Task 6**: Formulate and validate predictive models



- Empirical models to be tested:
  - Artificial neural network (ANN)
  - Partial least squares (PLS)
  - Mixed linear regression (MLR)

Li et al. *Bioenerg Res*, 2017, 10, 329

Karim and Hodge, *Biotechnol Prog*, 2003, 19, 1591

- Develop predictive models with $R^2 > 0.80$ on an independent test dataset
- End of Project Goal

- Achieve target separations
- Develop and validate robust predictive models

2 – Progress and Outcomes

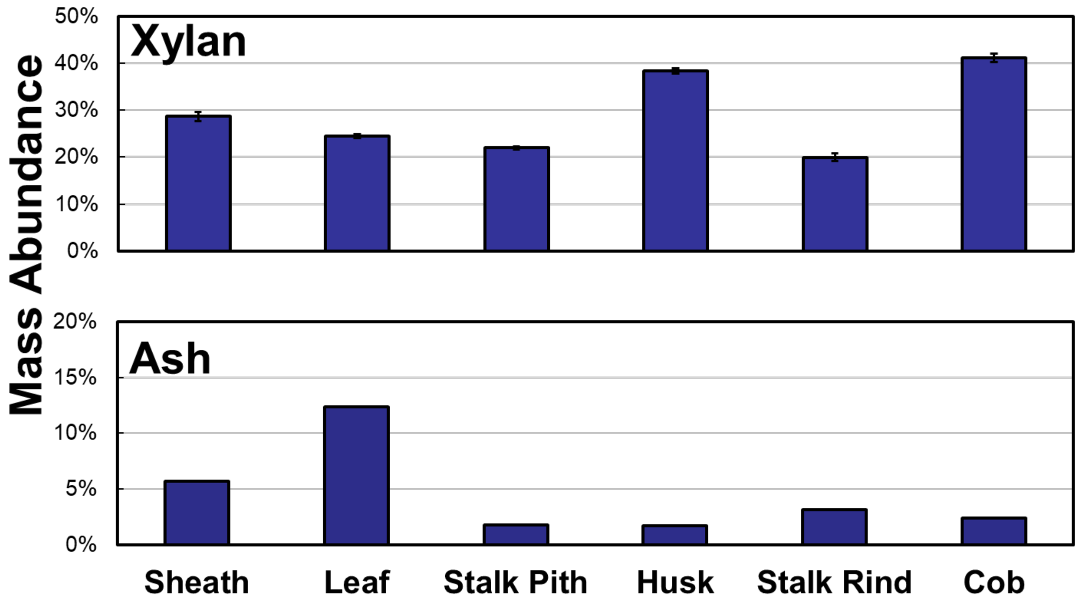
Task 2: Physical Fractionation of Corn Stover	
Milestone 2.1.1	Complete manual classification to yield 30-100 g samples of anatomical fractions from corn stover
Milestone 2.2.1	Generate at least one clean stem fraction that contains a minimum of 75% stems in terms of purity and yield
Go/No-Go Decision Point DP3.3.1	Demonstrate ability to achieve target separations: (1) removal of >30% of the ash in a high-ash leaf fraction with less than 10% loss of biomass (2) generation of low-ash (<7%) leaf/husk/sheath fraction (3) generation of stem-rich fraction at >75% purity and yield (4) generation of cob-rich fraction >75% purity and yield

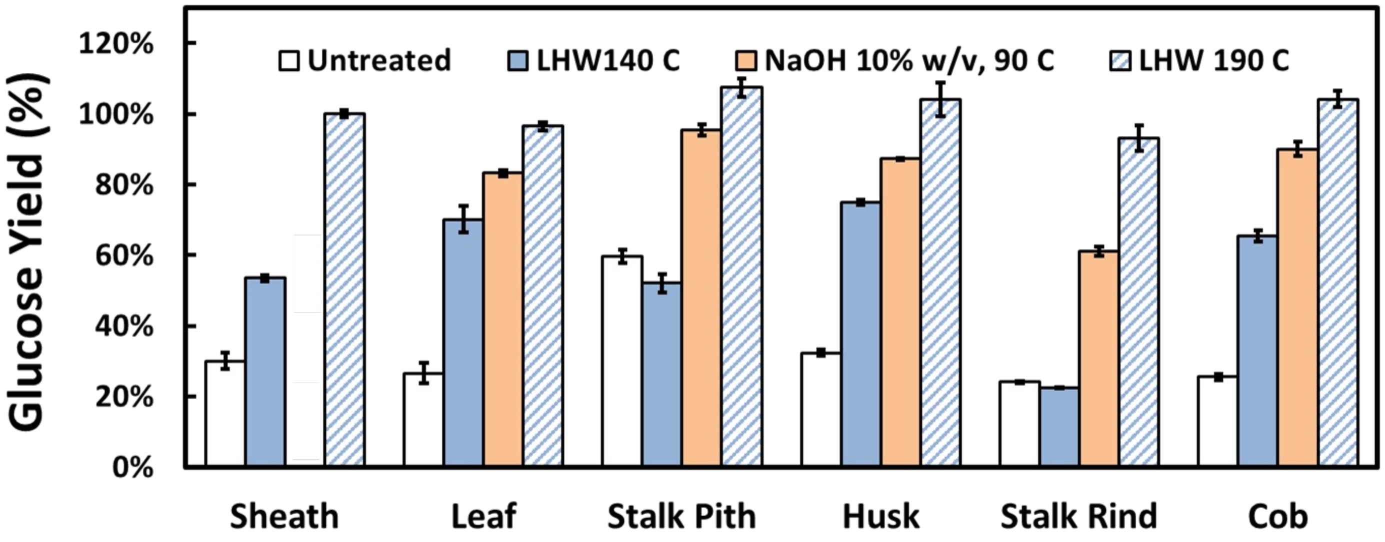
Task 2: Physical Fractionation of Corn Stover by Anatomy and First-Stage Fractionation

Subtask 2.1: Manual classification of biomass for use as a reference set

Significant differences in anatomical fractions with respect to:

- **Composition**
- **Response to pretreatment and hydrolysis**
  - **Increasing pretreatment "severity" increases hydrolysis yields**





M2.1.1 – Complete manual classification

Completed

Task 2: Physical Fractionation of Corn Stover by Anatomy and First-Stage Fractionation

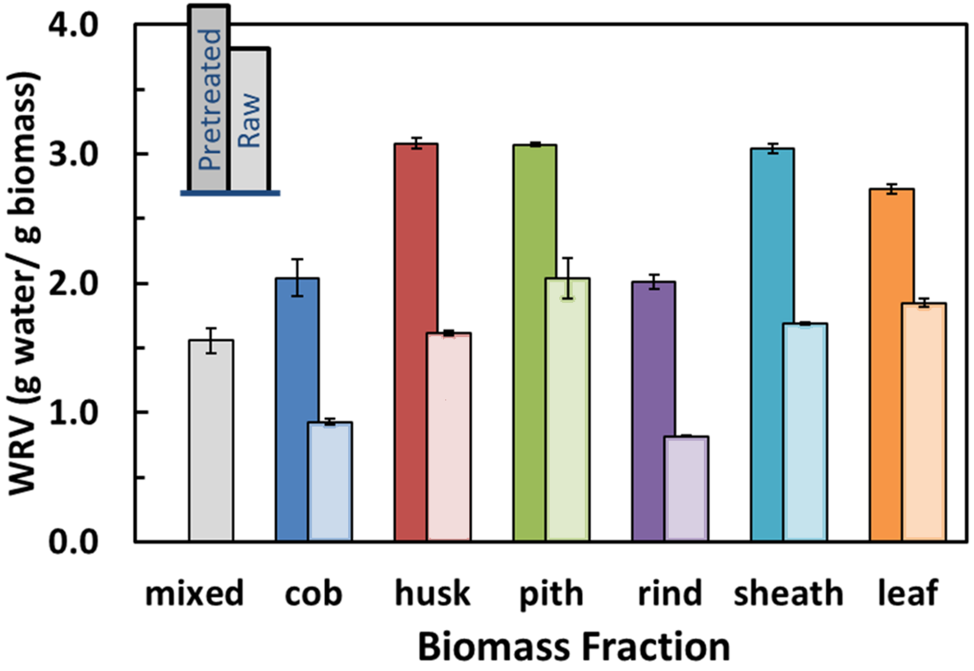
Subtask 2.1: Manual classification of biomass for use as a reference set

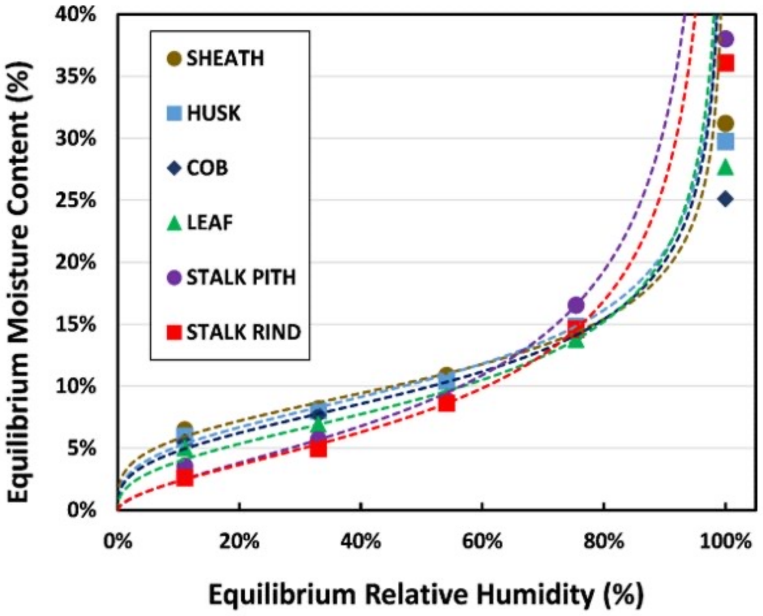
Significant differences in anatomical fractions with respect to:

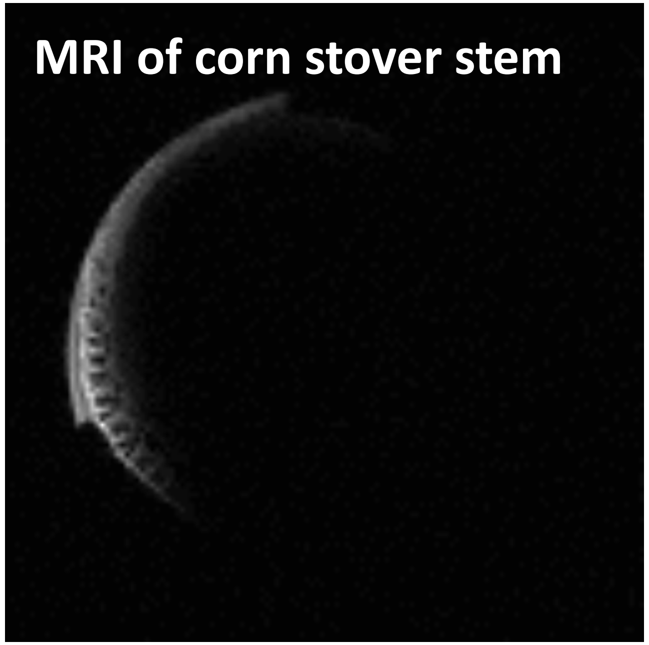
- **Water sorption**
  - **Increasing water sorption with pretreatment**
  - **Water sorption generally corrected to hydrolysis yields**

M2.1.1 – Complete manual classification

Completed

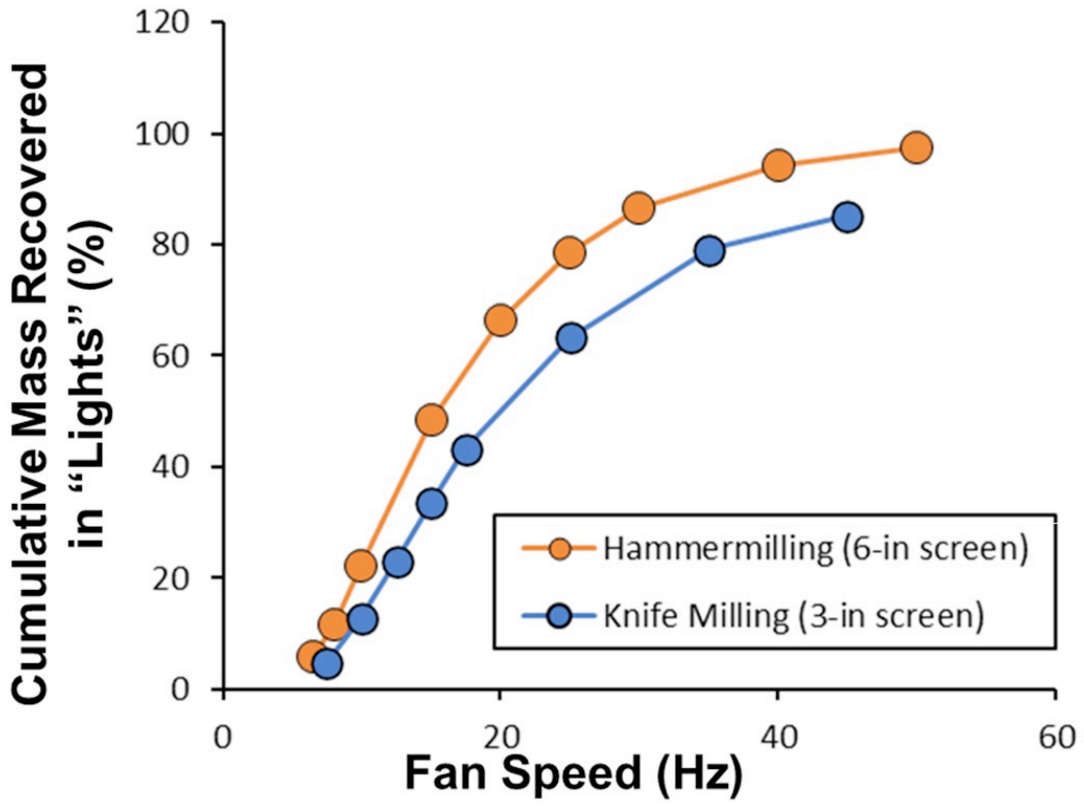


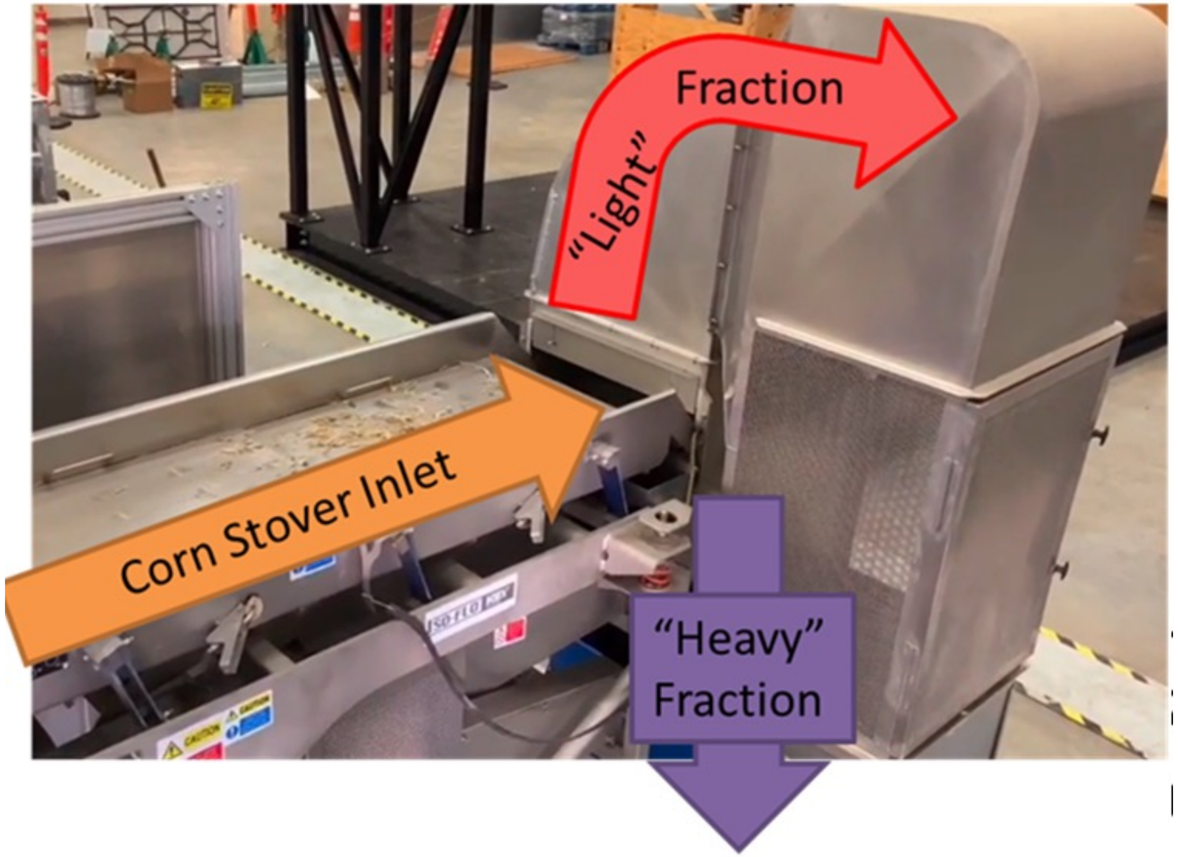




Task 2: Physical Fractionation of Corn Stover by Anatomy and First-Stage Fractionation

Subtask 2.2: Air classification for first stage of air classification



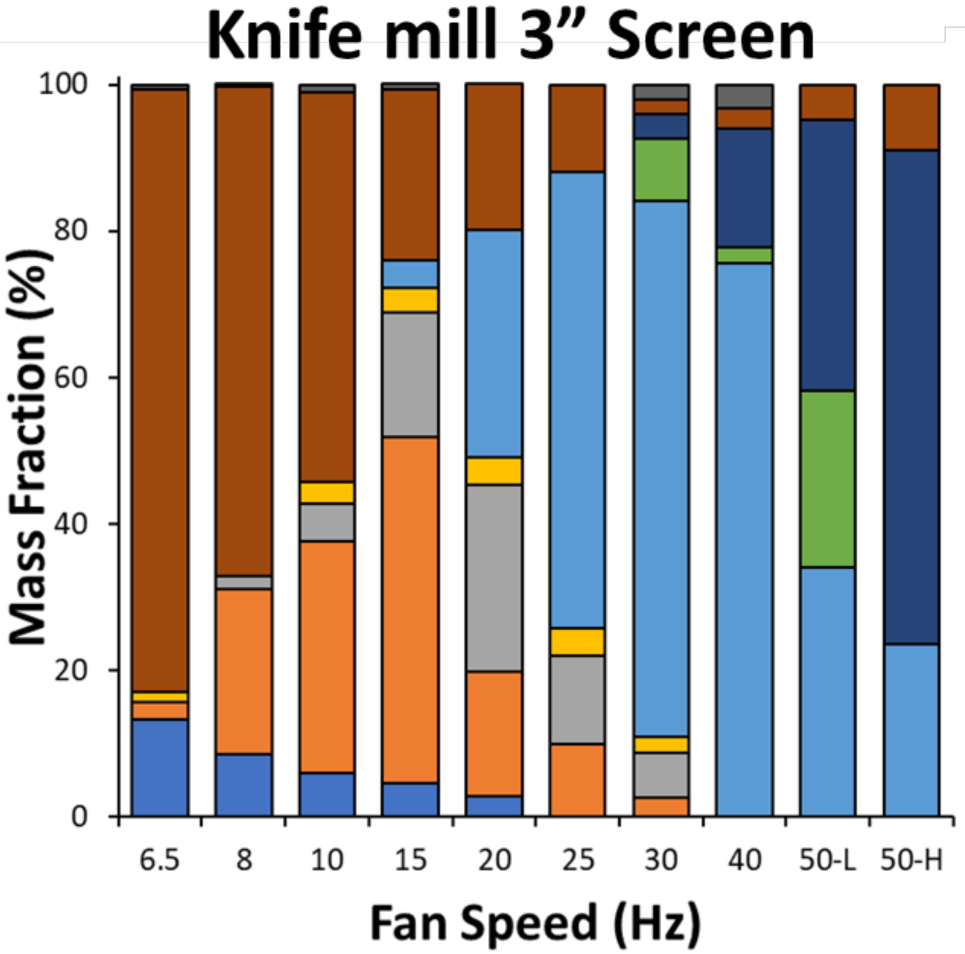


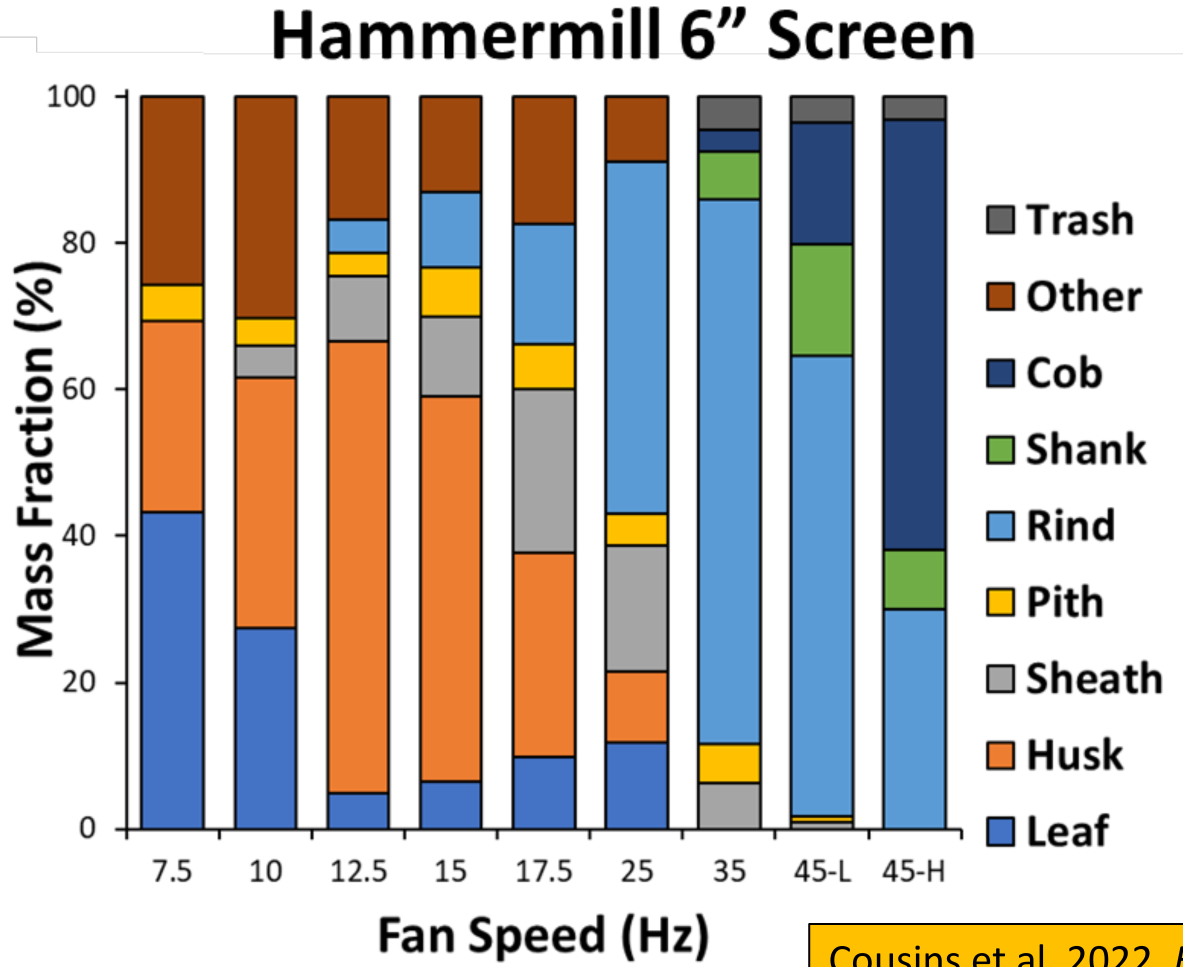
- Corn stover bale subjected to "breaking" followed by hammermilling or knife milling
- Classified by 2X Air Cleaner (Key Technology)
- Manual identification of components recovered

Cousins et al. 2022. *Biomass Conv Bioref*. DOI: 10.1007/s13399-022-03307-1

Task 2: Physical Fractionation of Corn Stover by Anatomy and First-Stage Fractionation

Subtask 2.2: Air classification for first stage of air classification





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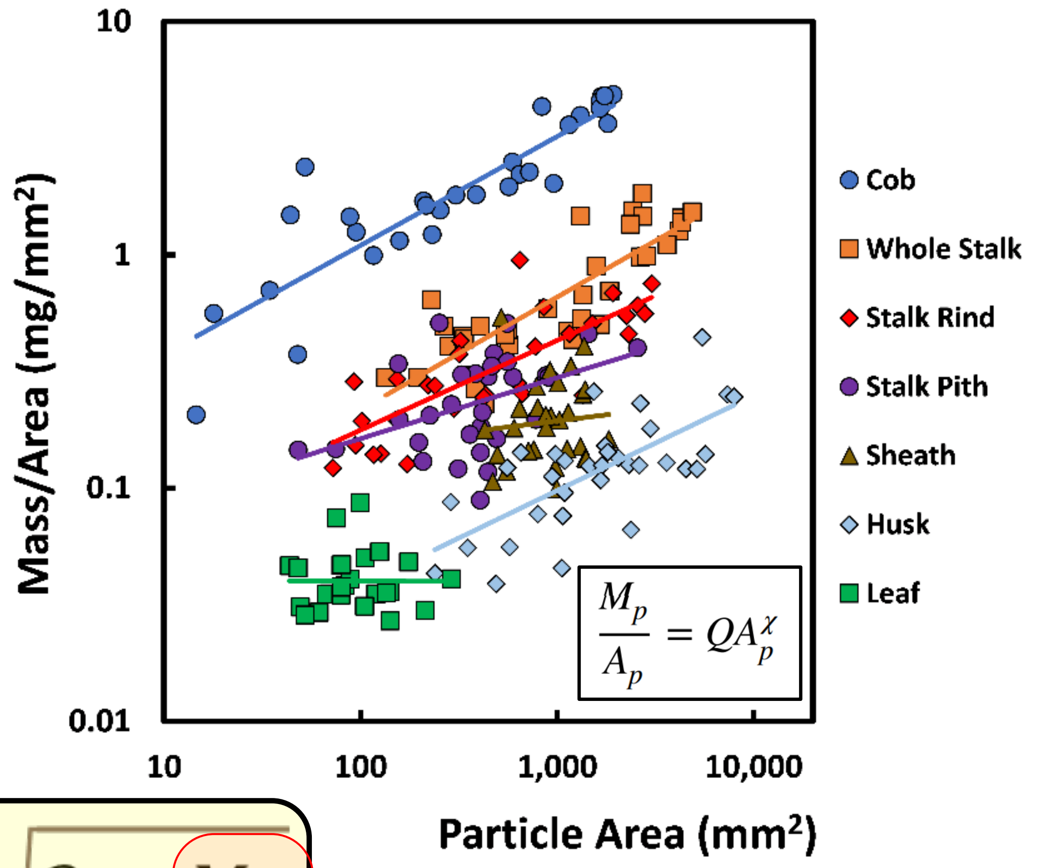
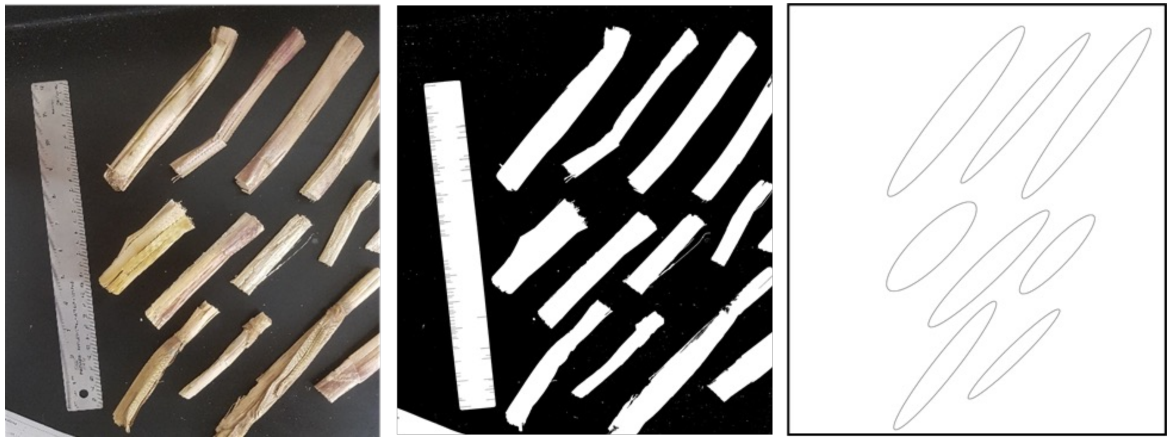
Task 2: Physical Fractionation of Corn Stover by Anatomy and First-Stage Fractionation

Subtask 2.2: Air classification for first stage of air classification

Development of air classification model. Goal:
- **Develop tool for prediction of air classification performance**
- **Potential to adjust operating conditions and particle properties (prior comminution) to yield optimal separations**



- Fluidization velocity (u_0) is a function of mass/area ratio (M_p/A_p), drag coefficient (C_D)
- Can use automated image analysis to determine particle area, estimate M_p/A_p



$$u_0 = \sqrt{\frac{2g}{C_D \rho_f} \frac{M_p}{A_p}}$$

Cousins et al. 2022. *Biomass Conv Bioref*. DOI: 10.1007/s13399-022-03307-1

Task 2: Physical Fractionation of Corn Stover by Anatomy and First-Stage Fractionation

Subtask 2.2: Air classification for first stage of air classification

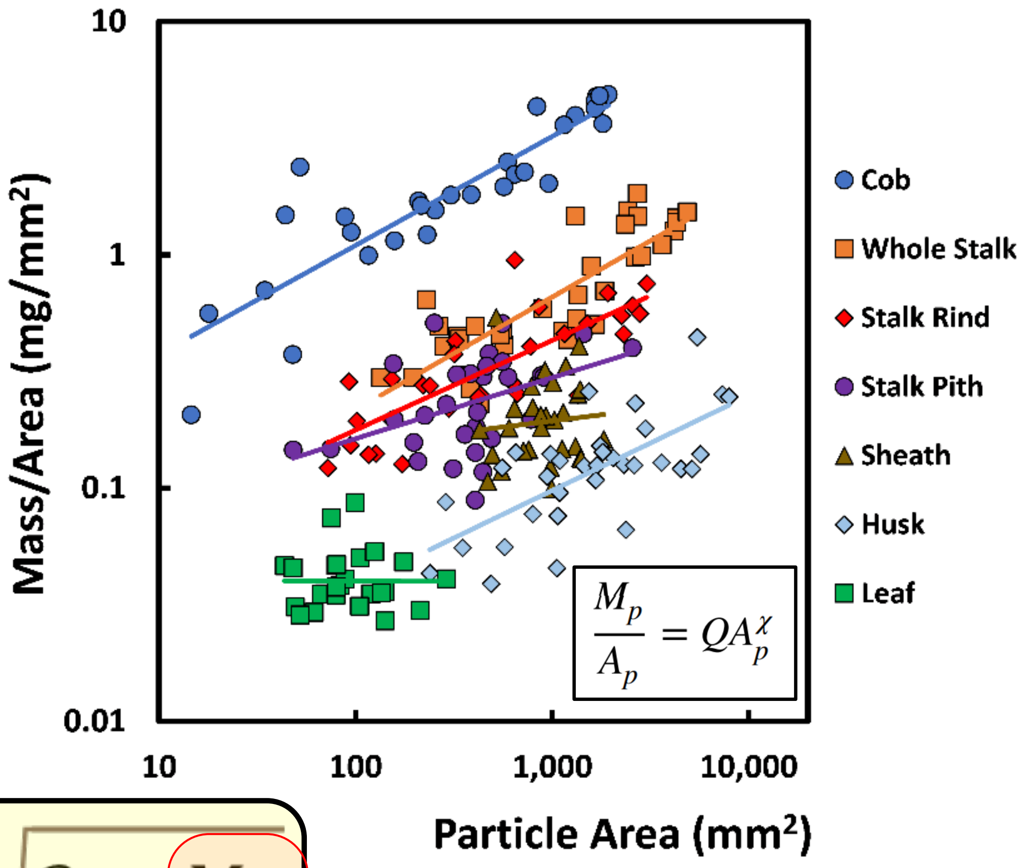
Development of air classification model. Goal:

- **Develop tool for prediction of air classification performance**
- **Potential to adjust operating conditions and particle properties (prior comminution) to yield optimal separations**

- Fluidization velocity (u_t) is a function of
-

Tissue	Prefactor (Q)	Exponent (χ)	R^2
Cob	0.13	0.47	0.85**
Whole stalk	0.024	0.48	0.70**
Sheath	0.094	0.10	0.001
Husk	0.0059	0.41	0.40**
Leaf	0.041	−0.0046	0.0001
Stalk rind	0.030	0.38	0.53**
Stalk pith	0.049	0.26	0.21*

$$\sqrt{\frac{2g}{C_D\rho_f}\frac{M_p}{A_p}}$$



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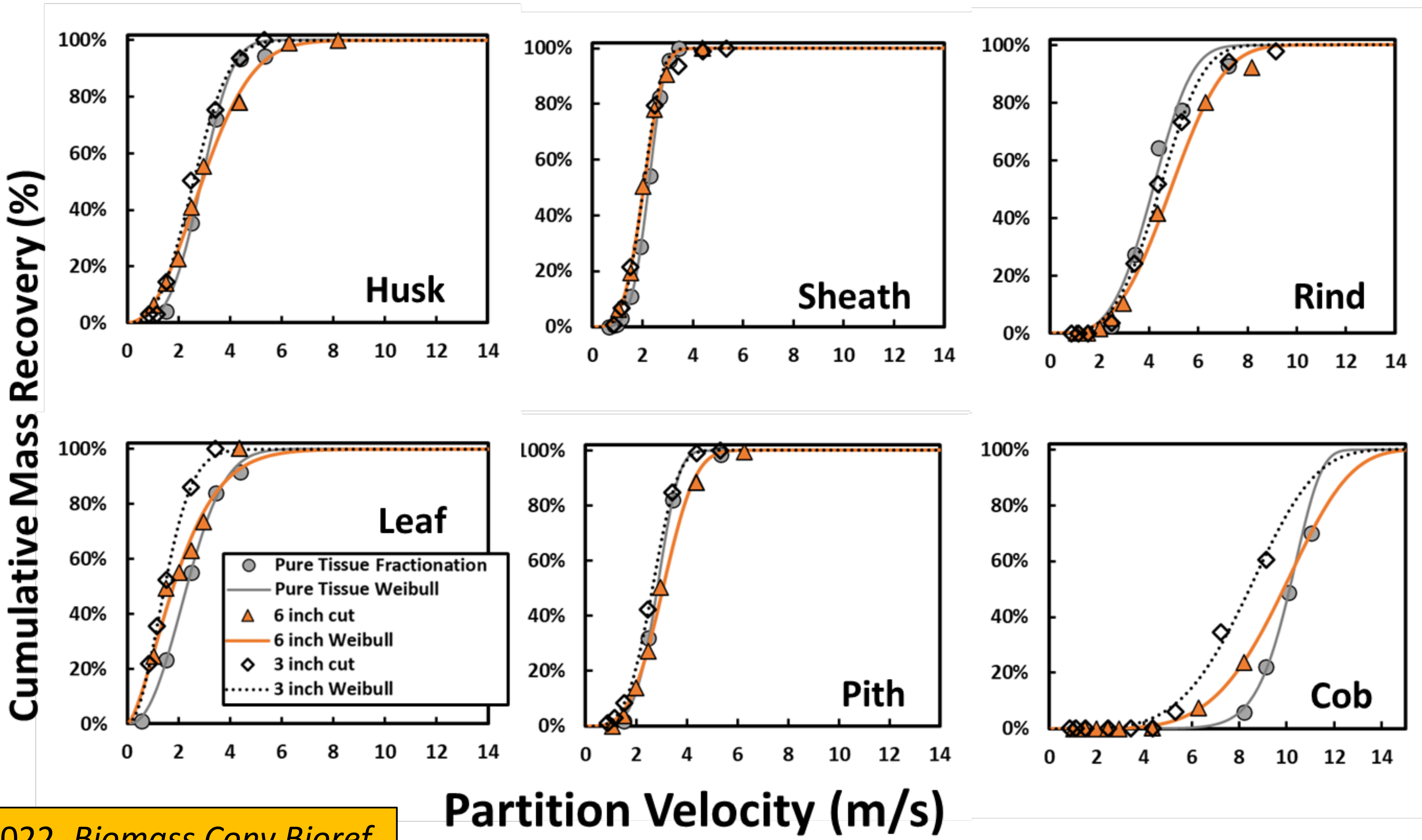
Task 2: Physical Fractionation of Corn Stover by Anatomy and First-Stage Fractionation

Subtask 2.2: Air classification for first stage of air classification

- Three experimental data sets
  - Significant differences in particle size distributions
  - Parameters fitted for models for predicting mass partitioning during air classification
  - Differences in partition velocity indicate potential for separation



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DOI: 10.1007/s13399-022-03307-1

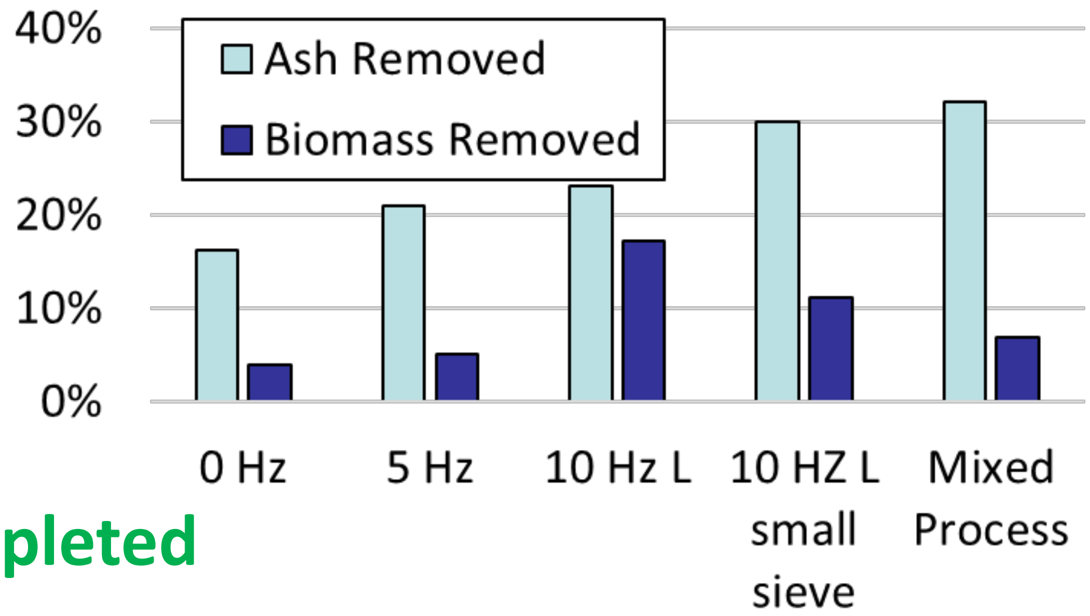
Task 2: Physical Fractionation of Corn Stover by Anatomy and First-Stage Fractionation

Subtask 2.2: Air classification for first stage of air classification

Ash Reduction Optimization with a Dirty Bale

- 10.9% ash content in **dirty bale**

- 0 Hz air speed but 60 Hz vibration using Key Technologies air classifier
- At 5 Hz and 10 Hz the lights are removed
- The lights at 10 Hz are sieved using 6 mm screen
- The lights from 5 Hz and lower were removed. The heavies at 10 Hz were sieved with 30 mesh screen and the fines (72% ash) were removed.

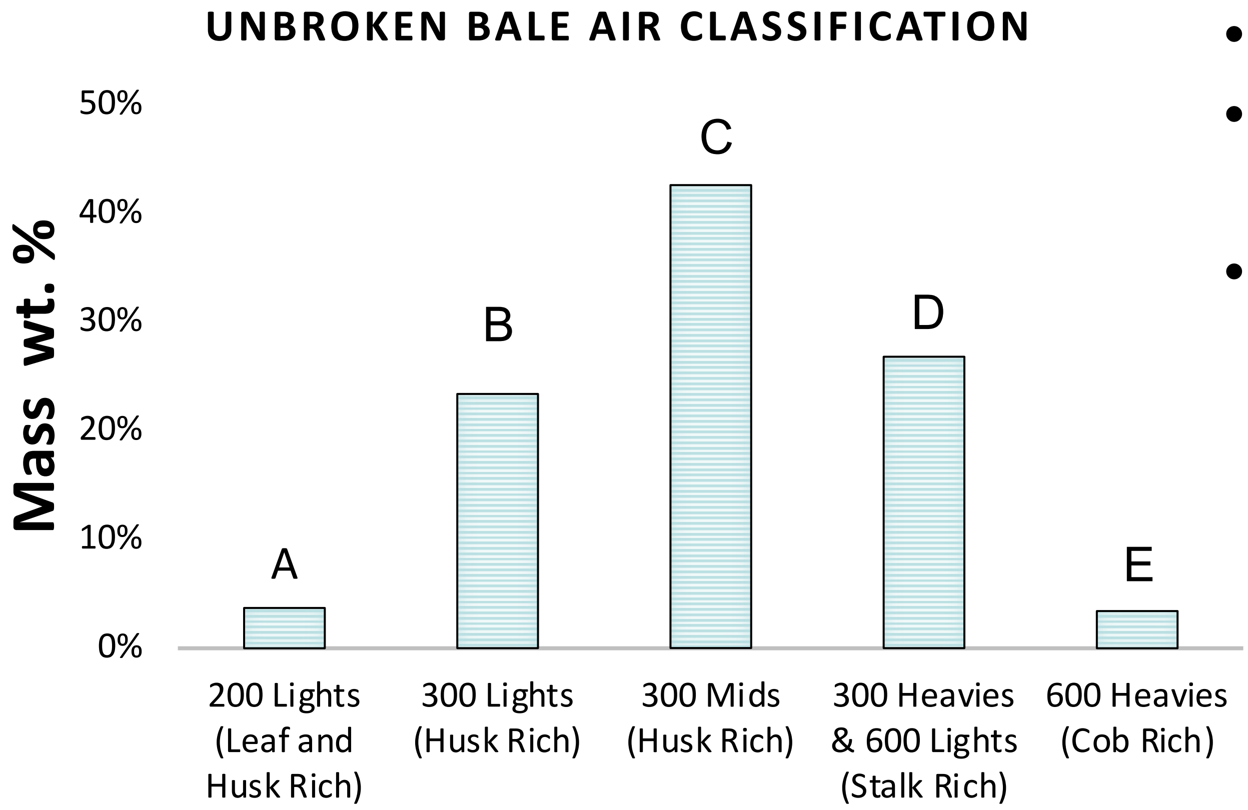


DP2.2.1.1 – Demonstrate ability to achieve target separations: removal of >30% of the ash in a high-ash leaf fraction with less than 10% loss of biomass

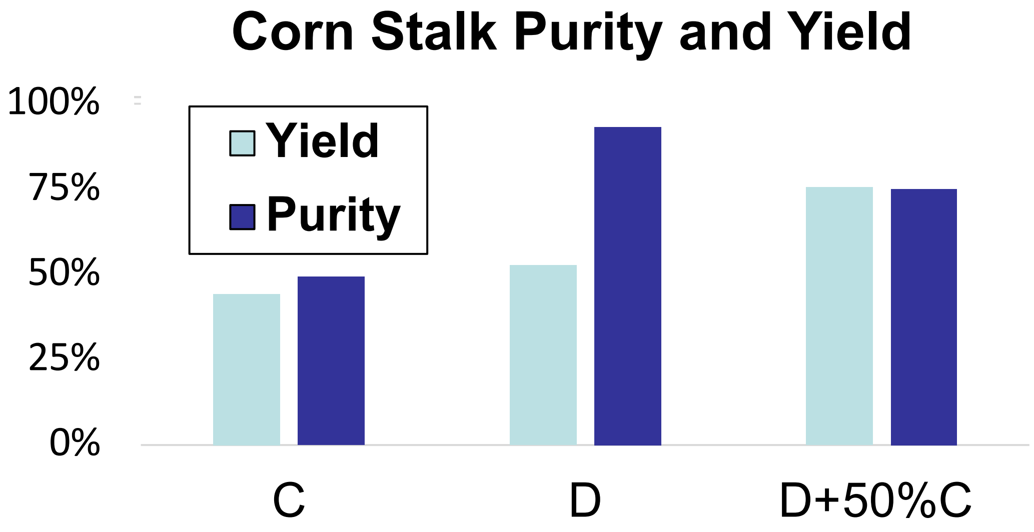
Completed

Task 2: Physical Fractionation of Corn Stover by Anatomy and First-Stage Fractionation

Subtask 2.2: Air classification for first stage of air classification



- Total recovery was 98.6 wt%
- Fractions defined by fan speed (rpm) of SPUDNIK and nature of resulting anatomical content
- Half of medium from 300 rpm were mixed with lights from 600 rpm

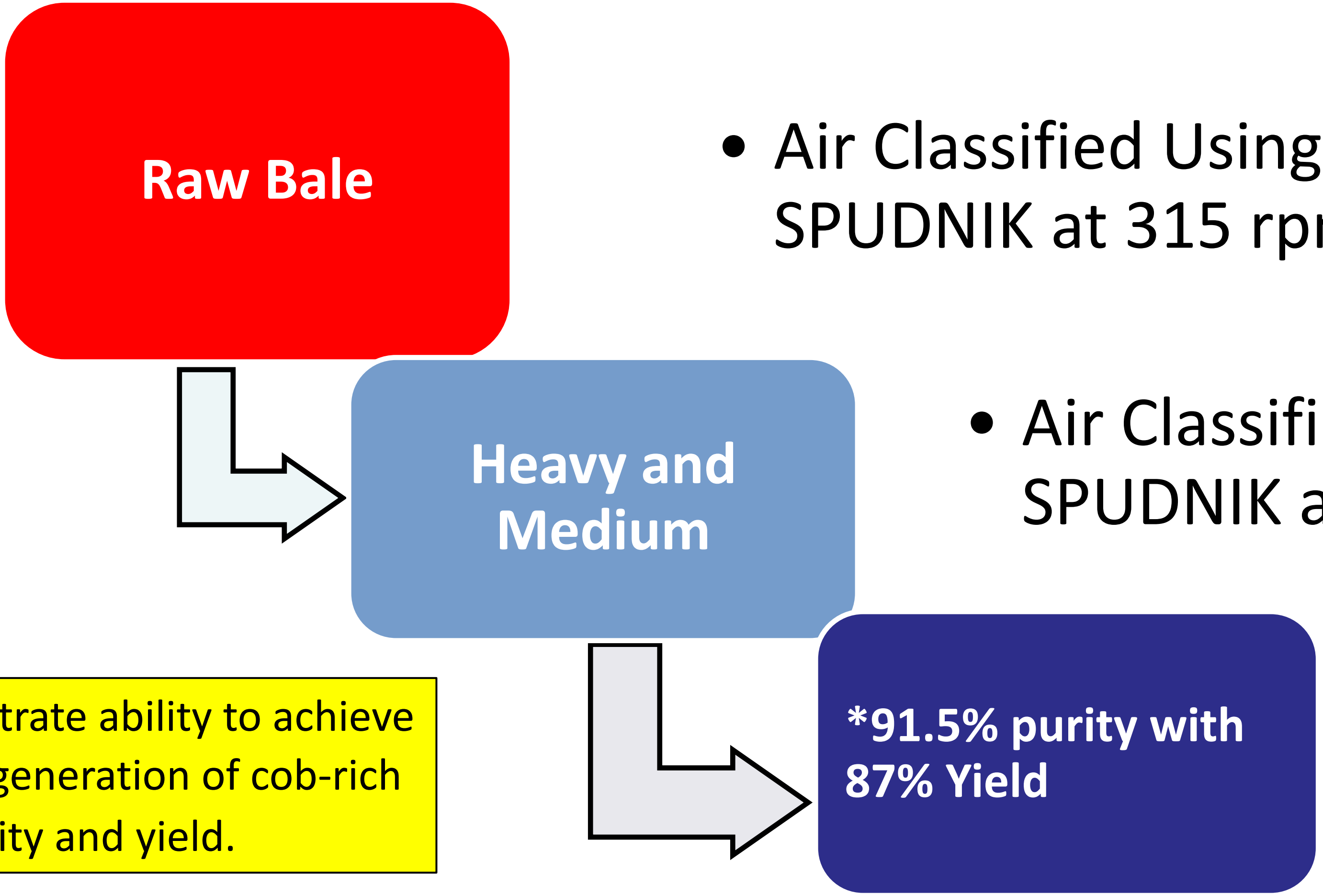


DP2.2.1.3 – Demonstrate ability to achieve target separations: generation of stem-rich fraction at >75% purity and yield

Completed

Task 2: Physical Fractionation of Corn Stover by Anatomy and First-Stage Fractionation

Subtask 2.2: Air classification for first stage of air classification



- Air Classified Using SPUDNIK at 315 rpm
- Air Classified Using SPUDNIK at 600 rpm
- Heavy

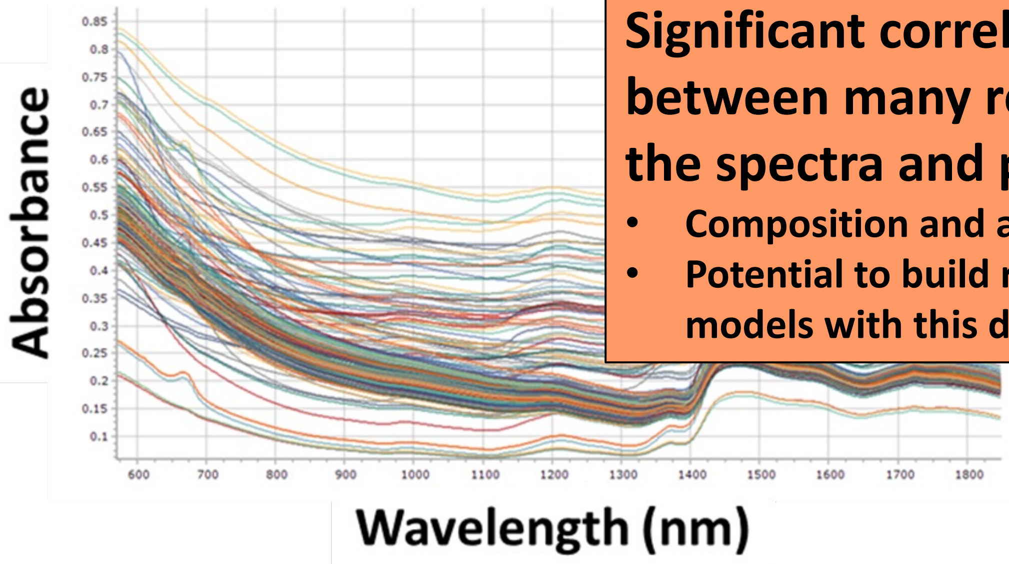
DP2.2.1.4 – Demonstrate ability to achieve target separations: generation of cob-rich fraction at >75% purity and yield.

Completed

Task 3: Development and Assessment of Biomass Characterization Protocols

Subtask 3.1: Development of tools for assessing composition and mass partitioning

M3.1.1 – Determine feasibility of utilizing NIR characterization as supplement or replace wet chemistry compositional analysis

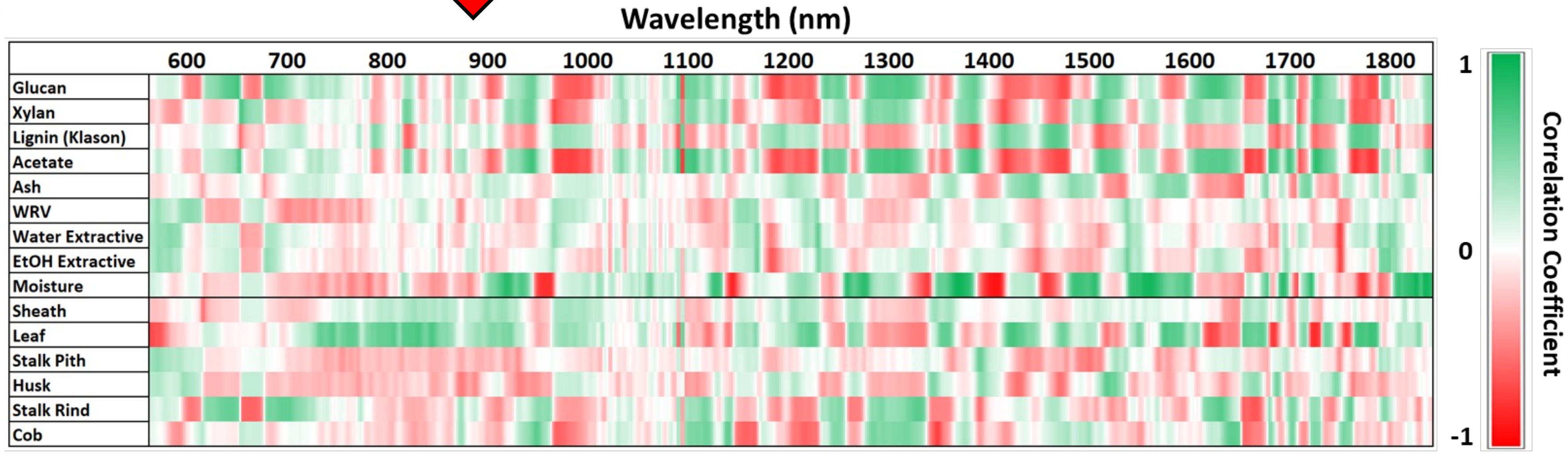


Significant correlations apparent between many regions within the spectra and properties:

- **Composition and anatomical fraction**
- **Potential to build robust predictive models with this data set**

- **Developed empirical NIR models utilizing:**
  - Partial Least Squares (**PLS**)
  - Gaussian Process Regression (**GPR**)
  - Neural Networks (**NN**)
- **Prediction of:**
  - Composition
  - Relative abundance of anatomical fraction
- **Assessed model performance**

Cousins et al. 2022. *Front Energ Res* 10, 836690



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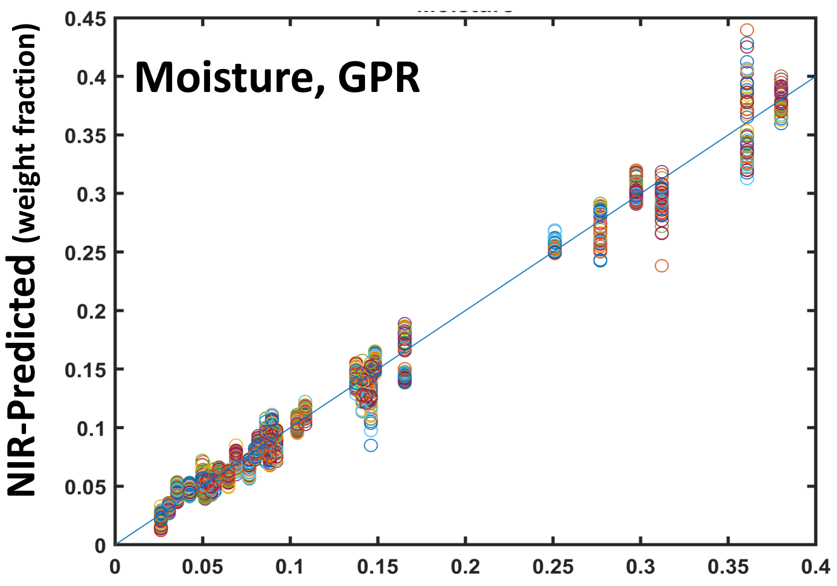

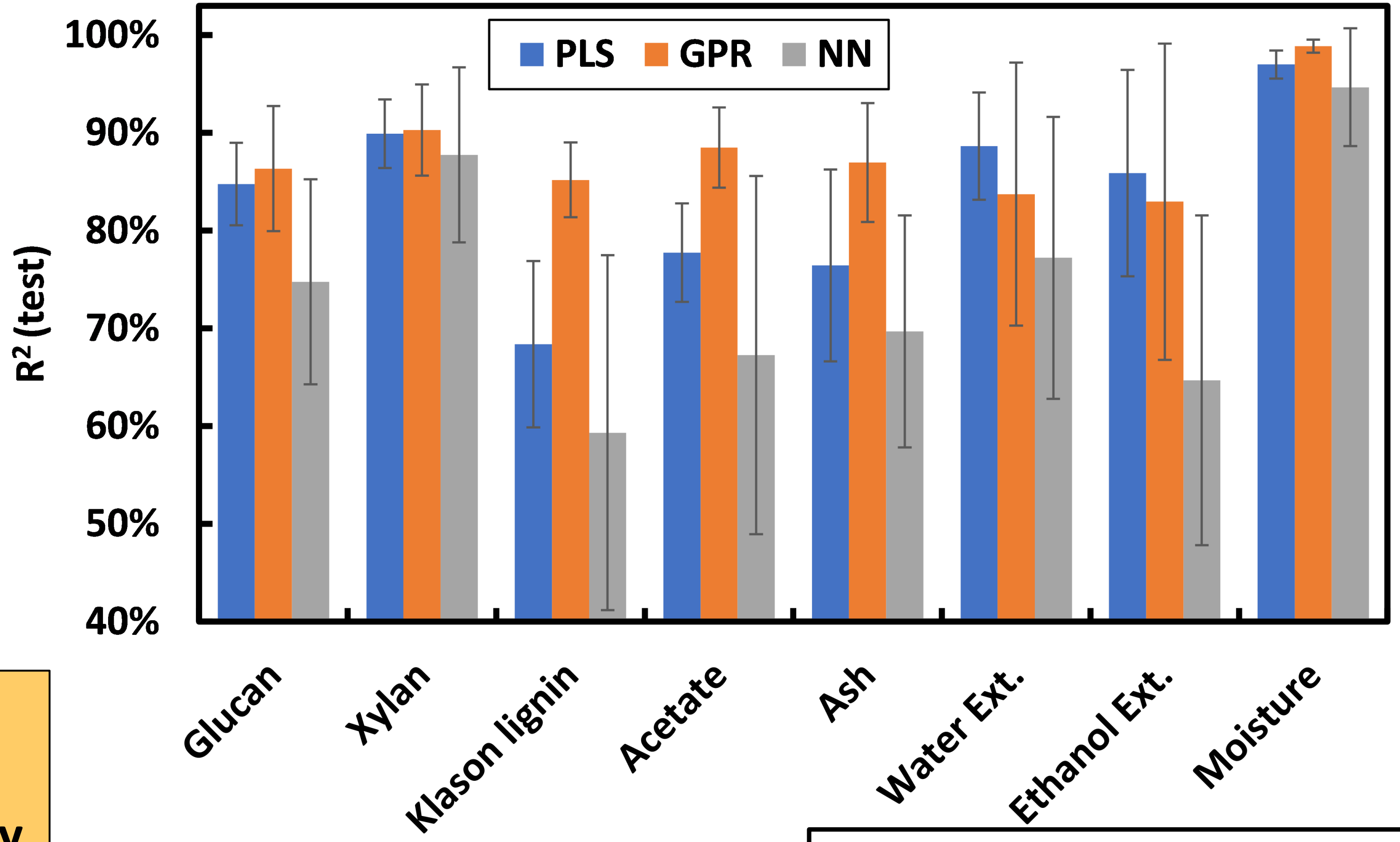

Models for predicting <u>composition</u> from truncated NIR spectra developed

- **100 models developed for each category**
- **R^2 from cross-validation of greater than 80% using project samples**

Cousins et al. 2022. *Front Energ Res* 10, 836690

PLS: Partial least squares
GPR: Gaussian process regression
NN: Neural networks

Task 3: Development and Assessment of Biomass Characterization Protocols

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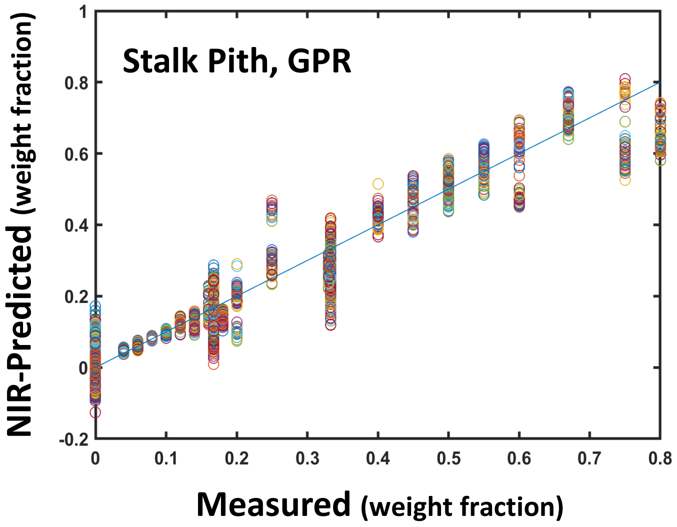
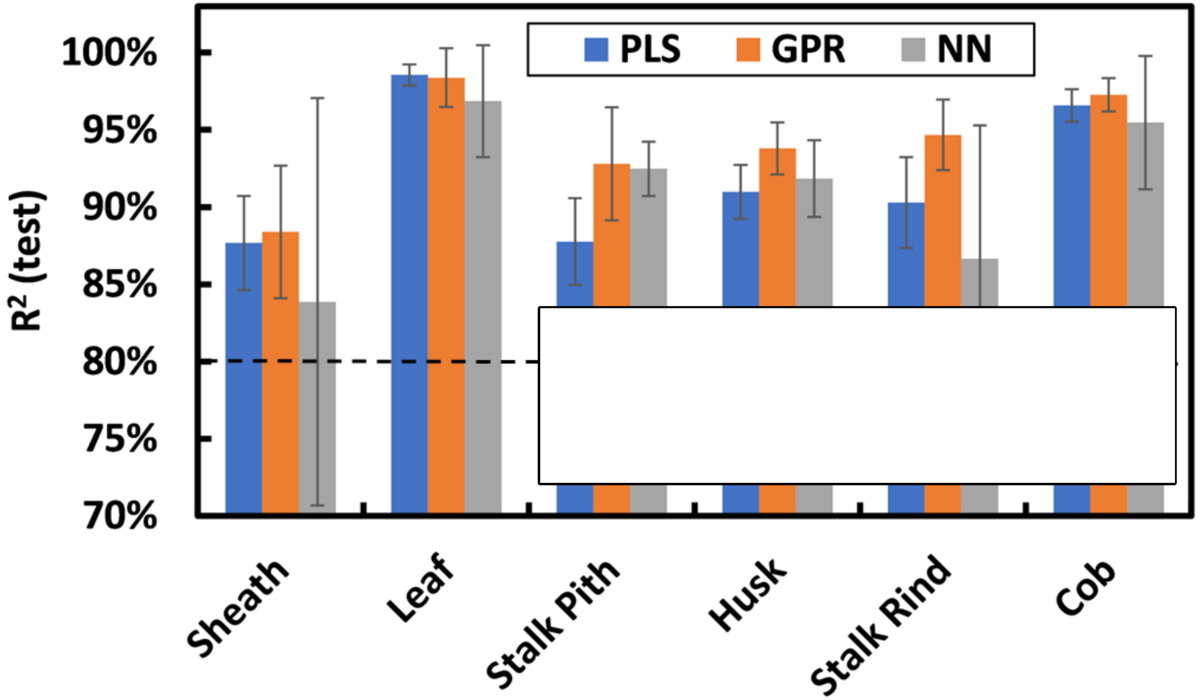
Completed

Models for predicting <u>anatomical fraction relative abundance</u> from truncated NIR spectra developed

- **100 models developed for each category**
- **R^2 from cross-validation of greater than 80% using project samples**
- **First demonstration of utilizing NIR for plant tissue prediction!**





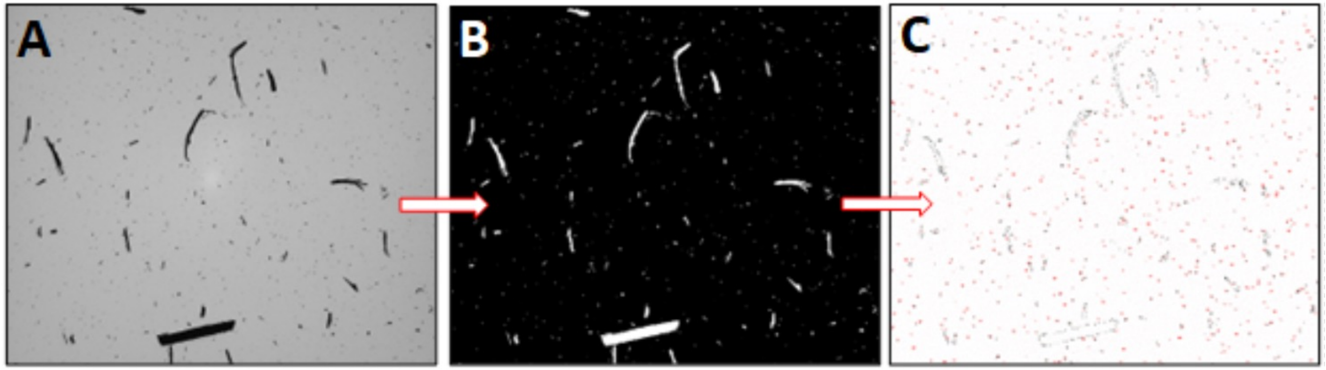
Significance:

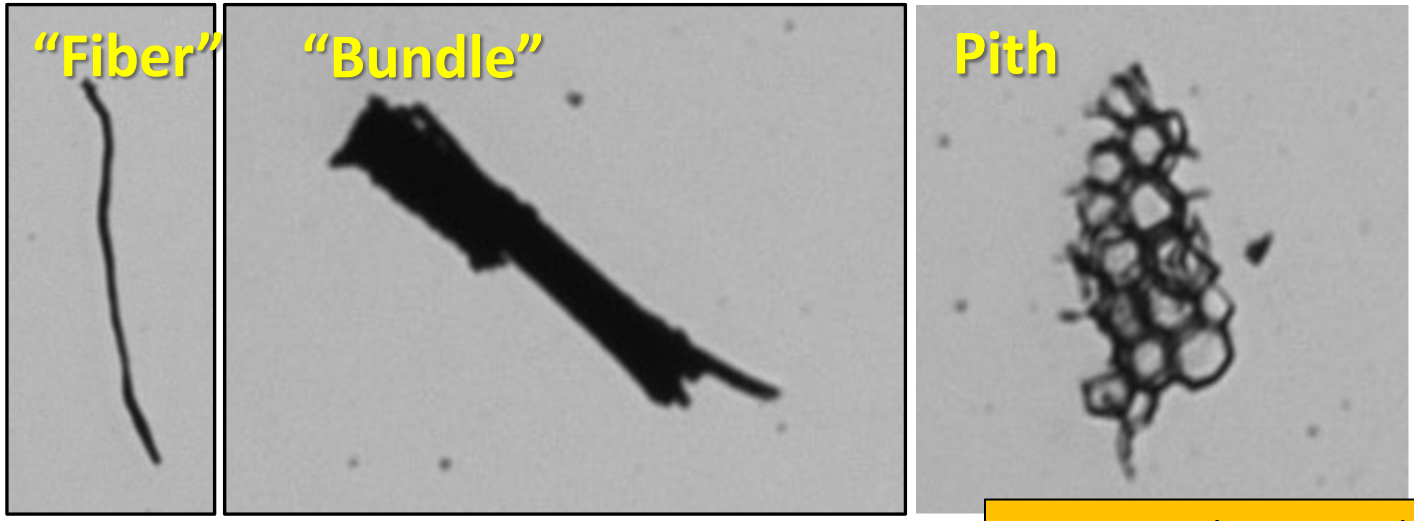
- Anatomical composition → mechanical conveyance (improve quality for handling), inform downstream unit operations for air classification
- Improved throughput analytical tool for assessing fractionation performance
- **Potential to replace wet chemistry (composition) and/or manual particle classification/assessment**

Task 3: Development and Assessment of Biomass Characterization Protocols

Subtask 3.2: Development of tools for assessing and classifying particle morphology

M3.2.1 – Determine feasibility of adapting automated dynamic image analysis for quantification of pith abundance and classification by particle morphology in corn stover





Cousins et al. 2023. *Ind Crop Prod*. 193, 116153

Goals:

- Automated image analysis coupled with machine learning to classify corn stover particles
  - Assign 18 different features for particles
  - Characterize relative abundance of pith

Particle Type	Bundle	Parenchyma	Fiber
Area	0.33	-0.075	-0.25
Perimeter	0.27	-0.18	-0.094
Width	0.31	-0.18	-0.12
Height	0.23	-0.25	0.028
Major	0.38	-0.31	-0.069
Minor	0.34	0.31	-0.66
Circularity	0.38	0.31	-0.70
Feret	0.29	-0.32	0.029
Minimum Feret	0.29	0.22	-0.51
Aspect Ratio	-0.18	-0.51	0.69
Roundness	-0.033	0.71	-0.67
Solidity	0.43	0.25	-0.68
%Area	0.33	-0.65	0.32

Task 3: Development and Assessment of Biomass Characterization Protocols

Subtask 3.2: Development of tools for assessing and classifying particle morphology

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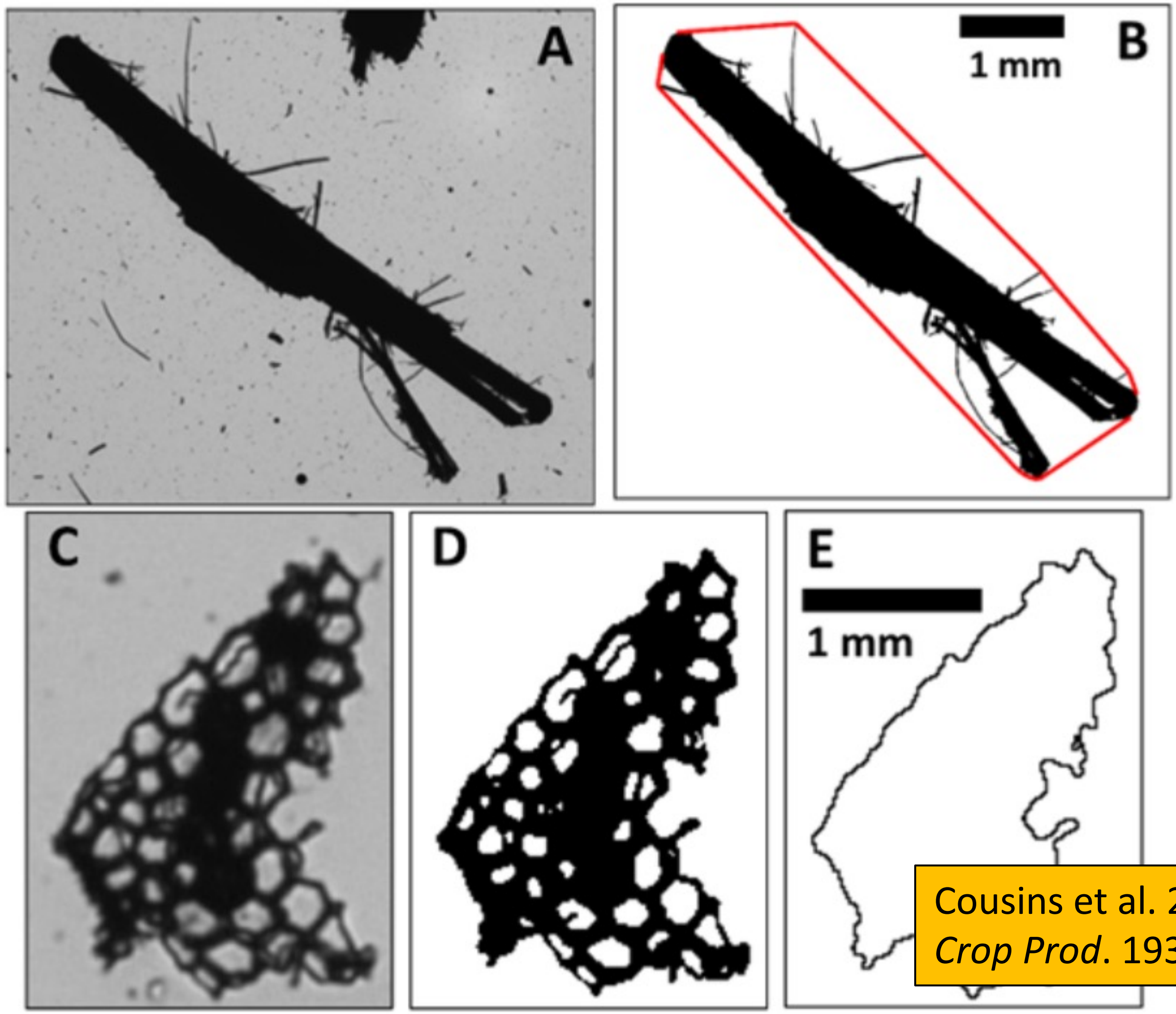

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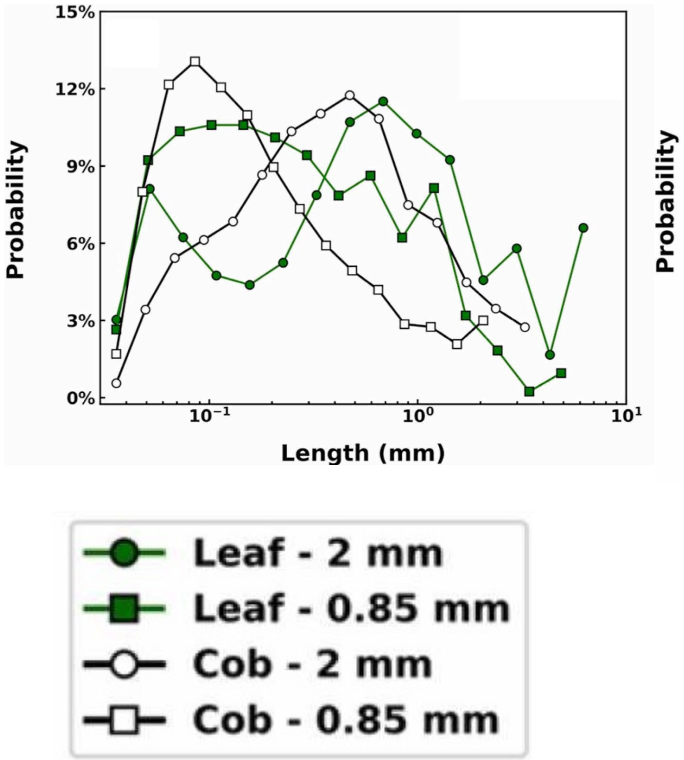
Task 3: Development and Assessment of Biomass Characterization Protocols

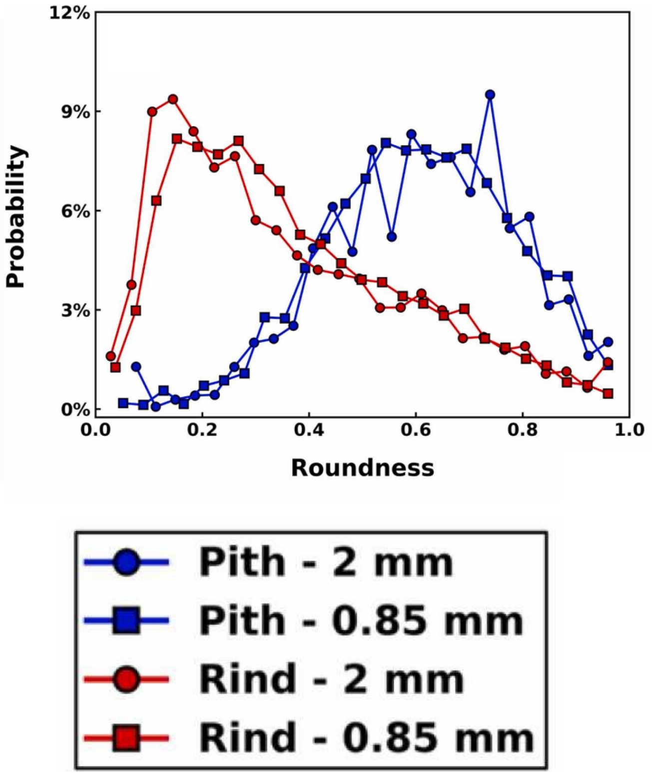
Subtask 3.2: Development of tools for assessing and classifying particle morphology

M3.2.1 – Determine feasibility of adapting automated dynamic image analysis for quantification of pith abundance and classification by particle morphology in corn stover

Completed

Cousins et al. 2023. *Ind Crop Prod*. 193, 116153





- Image-identified particle features can be used to distinguish anatomical fraction abundance

Developed combined imaging, analysis, and modeling methodology:

- **Application of quadratic support vector machine (Q-SVM) to correctly classify (99.4% accuracy) pith vs. "fiber"**
- **First demonstration of automated image analysis for classification of non-wood fibers!**

Significance:

- Knowledge of particle morphology can inform mechanical process operation (i.e. conveying, pumping) and chemical processing (i.e. reaction/diffusion limitations during hydrolysis)
- Application of this tool for assessing air classification performance

Task 3: Development and Assessment of Biomass Characterization Protocols

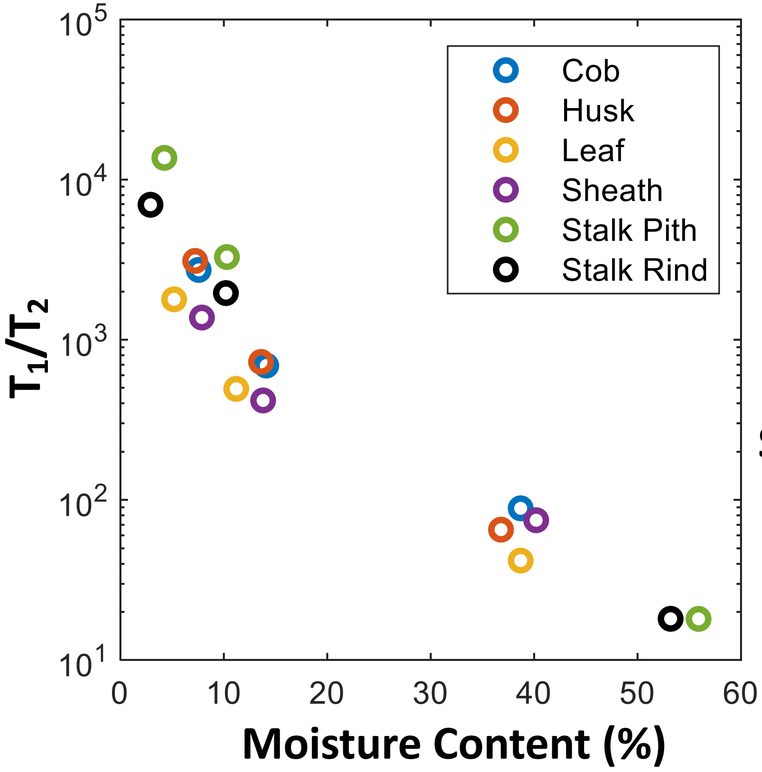
Subtask 3.2: Sorbed Water Properties

M3.3.1 – Generate baseline high-field NMR relaxometry data set for select well-defined corn stover anatomical fractions

Completed

Goals:

- Correlate molecular dynamics (which indicate water distribution in solvation, adsorbed and pore scale populations) with glucose yields to predict hydrolysis outcomes



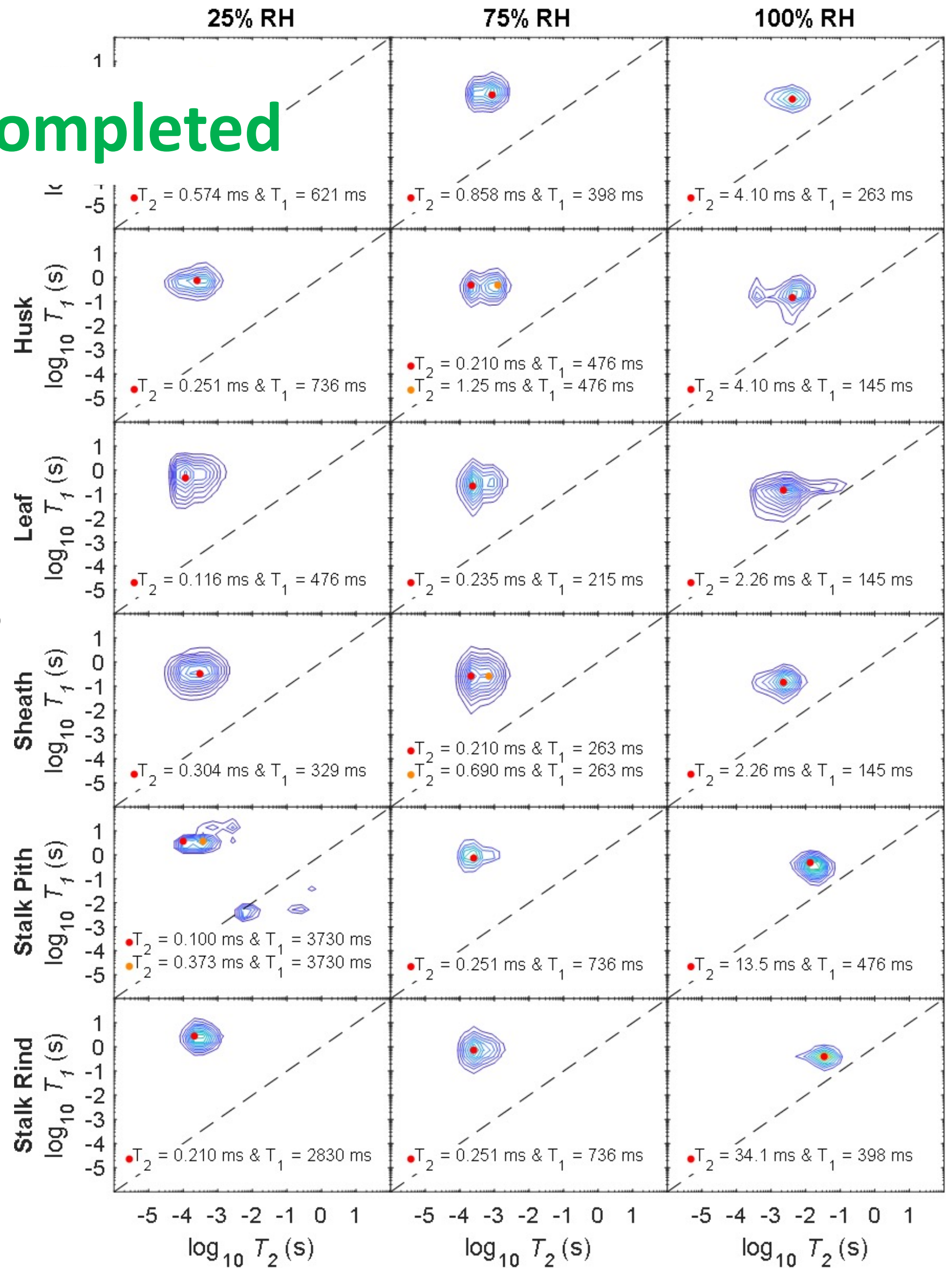
T_1/T_2 ratio:

- Measure of the surface energy related to the water adsorption strength and surface diffusive mobility
- Differs by anatomy
- Correlated to equilibrium moisture content

Significance – can be used to:

- Estimate water sorption equilibrium of different anatomies
- Estimate energy requirements for drying

Young et al. (submitted). *Cellulose*.



Task 3: Development and Assessment of Biomass Characterization Protocols

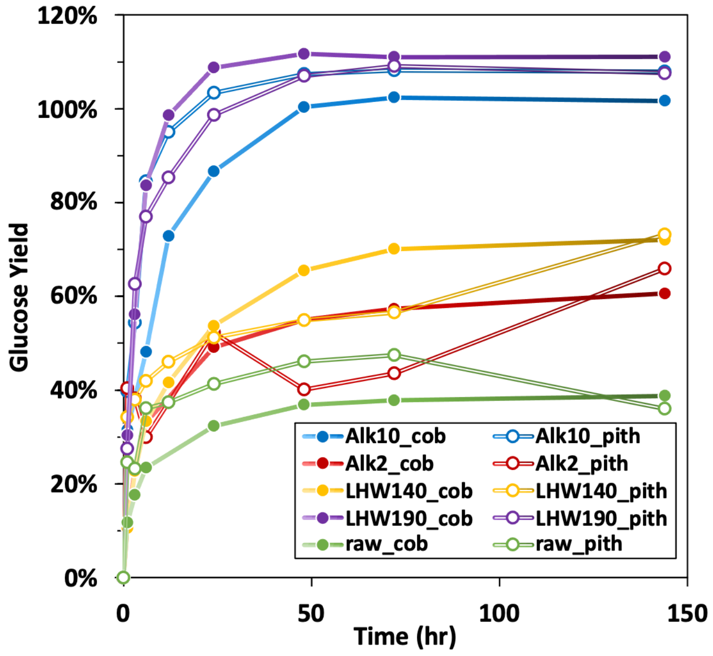
Subtask 3.2: Sorbed Water Properties

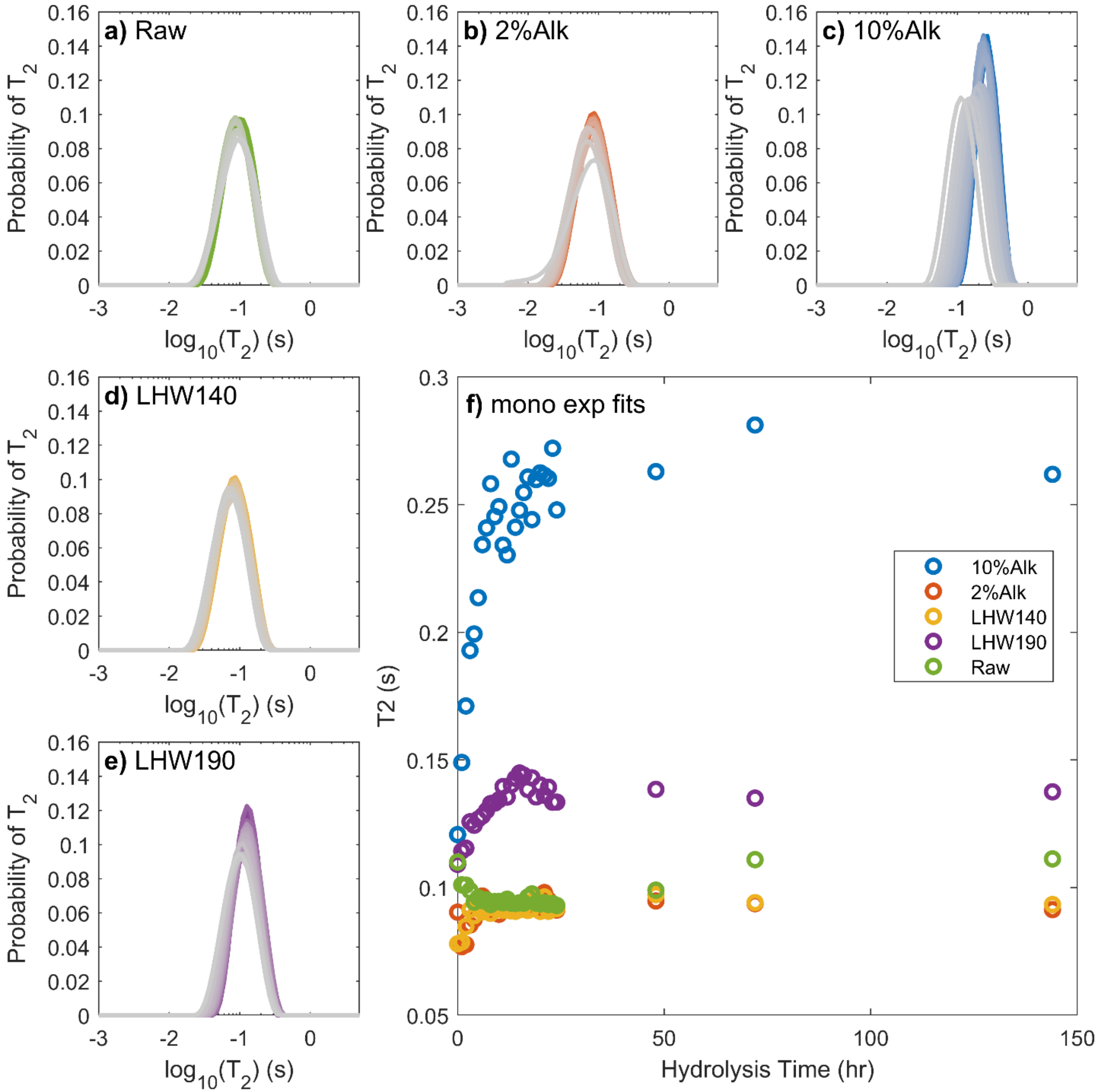
M3.3.2 – Determine feasibility of utilizing low-field NMR relaxometry as a quantitative characterization tool based on development of significant correlations between NMR-determined parameters and other quantified properties including pith content and response to enzymatic hydrolysis

Completed

Significance

- For select pretreatments, low-field NMR can track enzymatic hydrolysis, liquefaction of biomass





Task 5: Characterization of Physically Fractionated Corn Stover

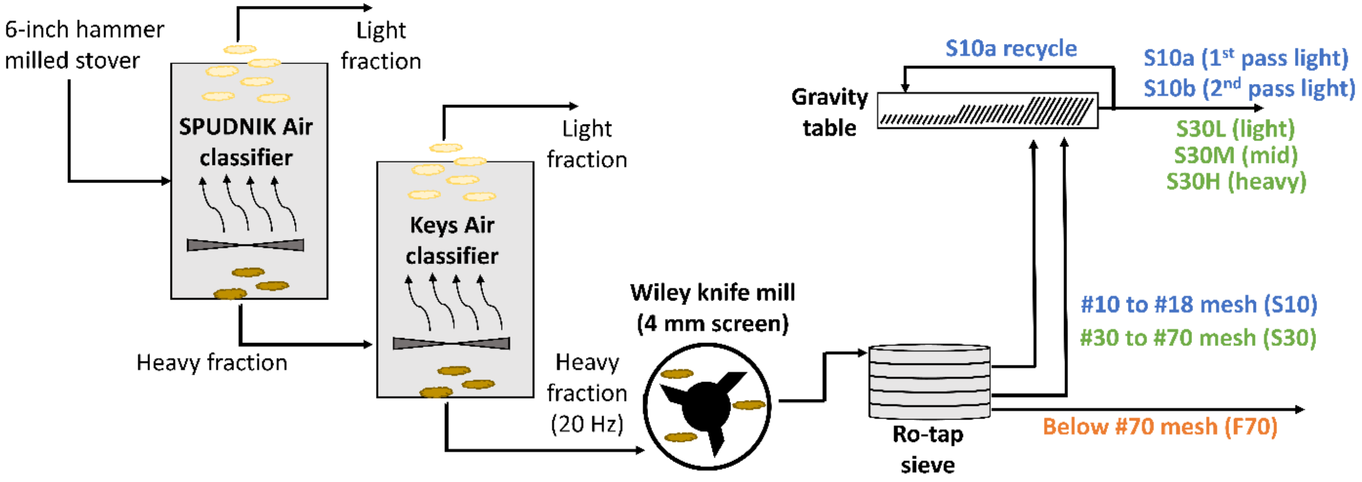
Subtask 5.1: Physical and Chemical Property Characterization

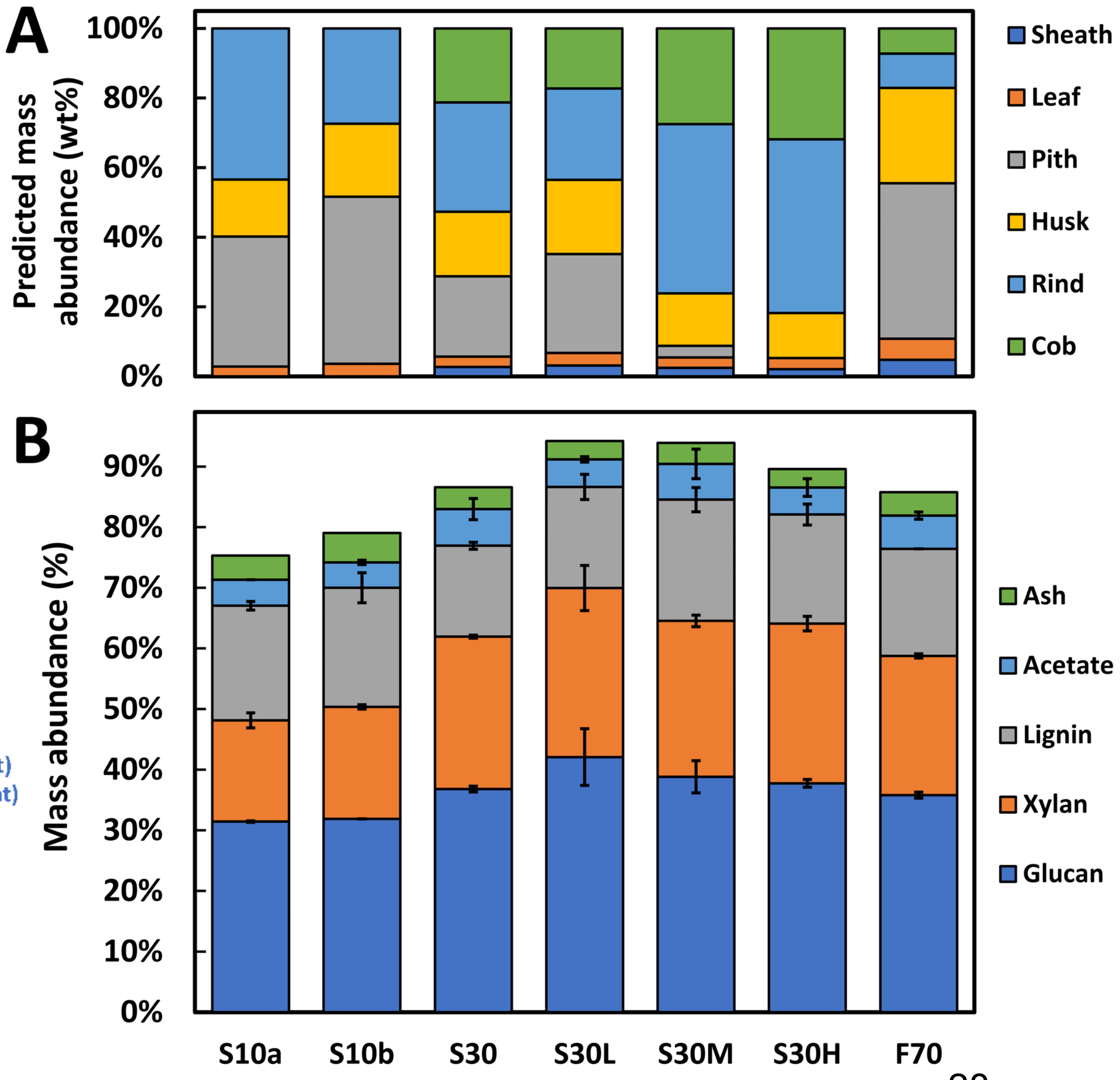
Goal: Generate comprehensive data sets on property characterization and response to processing for fractionated corn stover samples

M5.1.1 – Determine complete chemical composition characterization profiles of fractionated corn stover. **M5.1.2** – Apply image analysis to determine size and shape characterization profiles for fractionated corn stover sample sets

Example fractionation:
Integrated air classification, sieving, gravity table

Completed





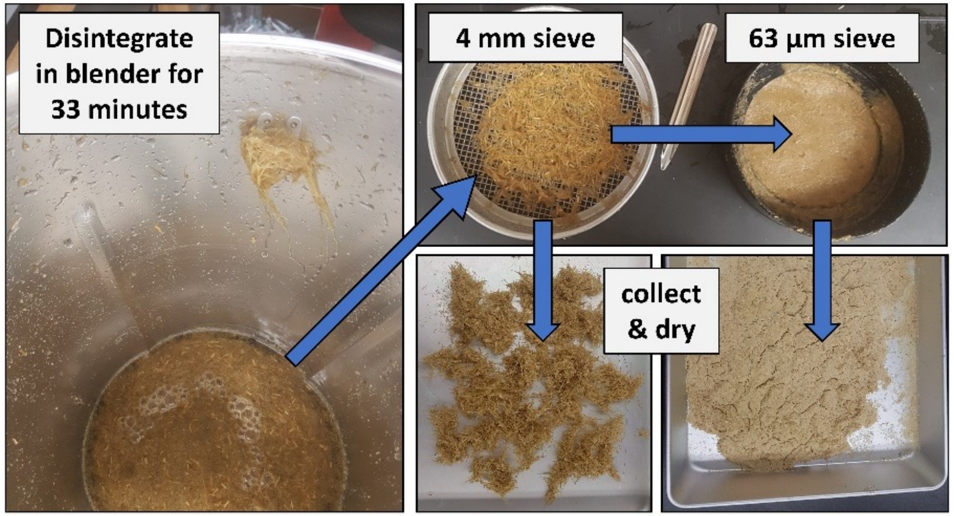
Task 5: Characterization of Physically Fractionated Corn Stover

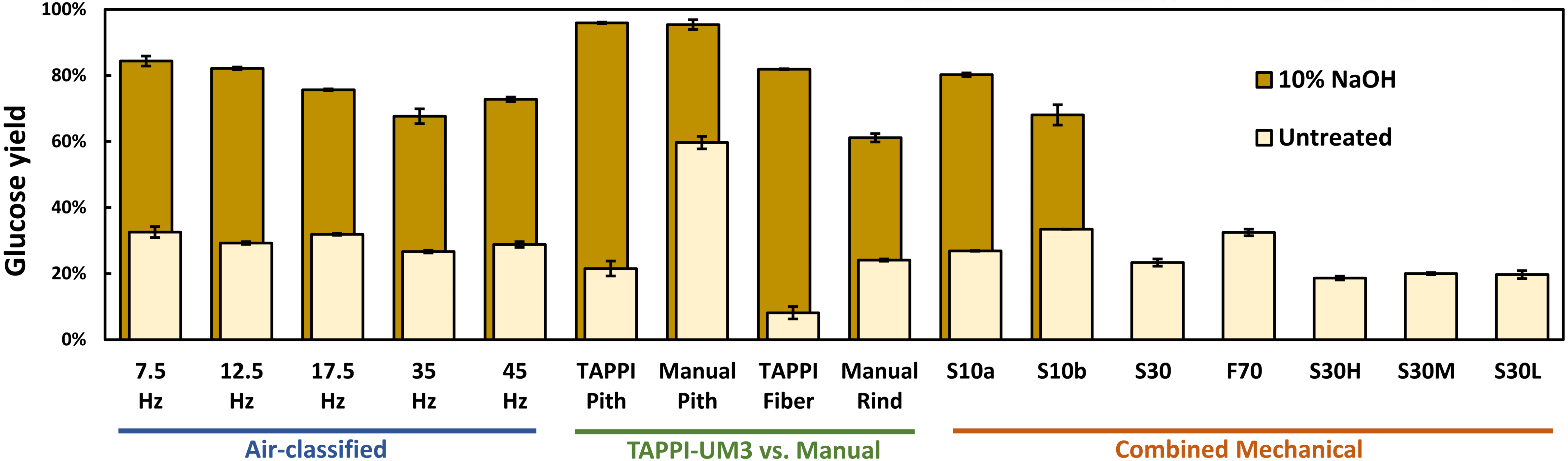
Subtask 5.2: Assessing Response of Fractionated Corn Stover to Processing

M5.1.1 – Screen biomass fractions for response to pretreatment and enzymatic hydrolysis

Completed

Example fractionation: "Wet depithing"





3 – Impact

- **Fundamental knowledge**
  - Developed comprehensive understanding of how corn stover properties impact preprocessing and conversion processes in a biorefinery, and inversely, how fractionation processing conditions impact the resulting biomass properties
- **Industry impacts**
  - Developed new analytical tools to better assess and characterize the heterogeneity within corn stover
  - Applied these analytical tools in conjunction with empirical models to assess preprocessing performance and predict corn stover fraction responses to biorefining
- **Dissemination of project results**
  - Peer-reviewed publications: 3 published, 1 in review, 2 in preparation
  - 10 presentations at national conferences

Summary

- Demonstrated how physical fractionation of corn stover can yield key processing outcomes:
  - Decreased heterogeneity in chemical and physical properties
  - Ash removal
  - Streamlined processing

- Identified physical fractionation approaches to achieve key processing targets

- Developed and adapted characterization tools and models for assessing physical fractionation performance

Quad Chart Overview

Timeline

- Project State Date: 10 January, 2020
- Project End Date: 30 September, 2023

	FY22 Costed	Total Award
DOE Funding	$507,253	$1,300,000
Project Cost Share	$46,010	$325,000

TRL at Project Start: 2
TRL at Project End: 4

Project Goal

- Develop "tunable" physical fractionation technologies for corn stover
- Develop new characterization tools for assessing feedstock performance in preprocessing and conversion operations

End of Project Milestone

Achieve target separations, develop and validate robust characterization tools that either singly or in combination can serve as proxy measurements for enzymatic hydrolysis yields following pretreatment. Provide final report to DOE that includes a summary of the key findings and recommendations from the project.

Funding Mechanism

DE-EE0008907 FY19 BETO MultiTopic FOA

Project Partners

- Idaho National Laboratory

Additional Slides

Responses for Previous Peer Reviewers' Comments

"Authors are asked to rank potential for newly defined cleaning/characterization technology to be scaled up for commercial use"

- **Response**: As the project progresses, we will commit to comparing and ranking potential new technologies relative to the benchmark case. While this has not been performed by the project team yet, this will be performed prior to project completion.

"Authors asked to rank the cost of the various commercial options ... This level of cost analysis may be beyond the scope of work for this study. However, it is valuable result for biorefinery design." and *"Emphasis on valorizing these fractions would help to understand how much could practically be spent on pre-processing. For example, if the value justifies the extra-cost of pre-processing, or optimization of the down-stream process (can this be quantified?)."*

- **Response**: Although technoeconomic analysis (TEA) was not originally proposed for the project, we will commit to performing TEA characterization of select biomass characterization and fractionation scenarios as one output of this project. While this has not been performed by the project team yet, this will be performed prior to project completion.

"It is not clear how the dataset for training or regression and the dataset for testing or validation will set up, such as the data set size, major data variables, modeling evaluation criteria. R^2>0.8 is required for predictive models. Why 0.8 is a threshold? R2 is not the only criterion to judge the robustness and sufficiency of a model … a paired test could provide the p-values of correlation significance of these paired variables"

- **Response**: We are developing other models and means of relating characterization results to processing outcomes. We presented results with respect to the R-squared threshold of 0.8 because this was a target metric defined in the original FOA that we are addressing.

Publications, Patents, Presentations, Awards, and Commercialization

Peer-Reviewed Publications

1. Young MC , Nelson ML, Cousins DS, Hodge DB, Seymour JD (submitted). NMR relaxometry characterization of water adsorption in corn stover anatomical fractions. *Cellulose*.

2. Cousins DS, Otto WG, Pedersen KP, Rony AH, Lacey JA, Aston JE, Hodge DB (2023). Particle classification by image analysis improves understanding of corn stover morphological evolution during deconstruction. *Ind Crop Prod.* 193, 116153. DOI: 10.1016/j.indcrop.2022.116153

3. Cousins DS, Rony AH, Otto WG, Pedersen KP, Hernandez S, Lacey JA, Aston JE, Hodge DB (2022). Predictive models enhance feedstock quality of corn stover via air classification. *Biomass Conv Bioref.* DOI: 10.1007/s13399-022-03307-1

4. Cousins DS, Otto WG, Rony AH, Pedersen K, Aston JE, Hodge DB (2022). Near-infrared spectroscopy can predict anatomical abundance in corn stover. *Front Energ Res.* 10, 836690. DOI: 10.3389/fenrg.2022.836690

Publications, Patents, Presentations, Awards, and Commercialization

Presentations

1. Cousins DS, Otto W, Rony AH, Aston J, Hodge DB, Enhanced Feedstock Characterization and Modeling to Facilitate Optimal Preprocessing and Deconstruction of Corn Stover. (Oral) AIChE Fall Meeting, November 13, Phoenix, AZ.
2. Cousins DS, Hodge DB (2022). Particle image analysis informs biomass feedstock quality and processing parameters. (Poster) Symposium on Biotechnology for Fuels and Chemicals, May 1, New Orleans, LA.
3. Otto W, Hodge DB (2022). Coupling Characterization Techniques for Analysis of Corn Stover Deconstruction Across Anatomical Components. (Poster) Symposium on Biotechnology for Fuels and Chemicals, May 1, New Orleans, LA.
4. Cousins DS, Otto W, Rony AH, Lacey J, Aston J, Hodge DB. (2022). Enhanced Feedstock Characterization and Modeling to Facilitate Optimal Preprocessing and Deconstruction of Corn Stover. (Oral) Symposium on Biotechnology for Fuels and Chemicals, May 1, New Orleans, LA.
5. Rony AH, Cousins D, Hodge DB, Lacy J, Aston J. Corn Bale Deconstruction Using Physical Techniques for Improved Anatomical Separation. AIChE Spring Meeting. (Oral). 12 April 2022. San Antonio, TX
6. Otto W, Cousins D, Hodge DB (2021). Water Retention Value as a Characterization Approach for Predictive Modeling of Corn Stover Deconstruction. (Poster) AIChE Fall Meeting, November 8, Boston, MA.
7. Cousins D, Aston JE, Lacey JA, Hodge DB (2021). Coupled Near Infrared Spectroscopy and Air Classification of Corn Stover for Improved Feedstock Quality. (Oral) AIChE Fall Meeting, November 8, Boston, MA.
8. Otto W, Cousins D, Hodge DB (2021). Water Retention Value as a Characterization Approach for Predictive Modeling of Corn Stover Deconstruction. (Poster) SIM SBFC Meeting, April 26, virtual.
9. Cousins D, Otto W, Hernandez S, Lacey JA, Aston JE, Hodge DB (2021). Modeling the air classification of corn stover anatomical fractions for enhanced feedstock quality. (Poster) SIM SBFC Meeting, April 26, virtual.
10. Hodge DB (2021). Enhanced Feedstock Characterization and Modeling to Facilitate Optimal Preprocessing and Deconstruction of Corn Stover. DOE BETO 2021 Project Peer Review, March 10, virtual.