

Feedstock-Conversion Interface Consortium:

Unveiling Signatures of Feedstock Variability

April 28, 2021



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1-slide guide to the FCIC

The Feedstock-Conversion Interface Consortium is led by DOE as a collaborative effort among researchers from 9 National Labs

Key Ideas

- Biomass feedstock properties are **variable** and **different** from other commodities
- **Empirical** approaches to address these issues have been **unsuccessful**

We are developing **first-principles** based knowledge and tools to **understand** and **mitigate** the effects of biomass feedstock and process **variability** on biorefineries



<https://energy.gov/fcic>



Dr. Allison Ray, Idaho National Laboratory

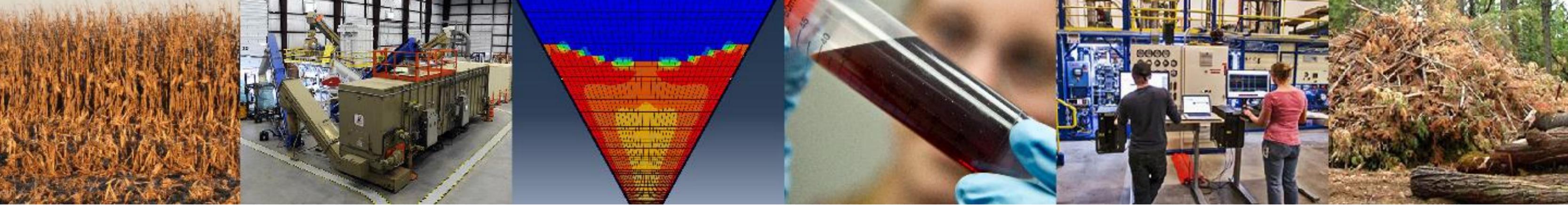
Dr. Allison Ray is a Senior Scientist and the Research Excellence Lead for Science & Technology at Idaho National Laboratory. Dr. Ray is the task lead for FCIC's Feedstock Variability task. She has a broad range of expertise in biomass and biofuels R&D spanning feedstock supply, logistics, feedstock quality improvement, preprocessing, and conversion. She received her Ph.D. in Environmental Microbiology from Idaho State University.



Dr. Bryon Donohoe, National Renewable Energy Laboratory

Dr. Bryon Donohoe is a Senior Scientist at the National Renewable Energy Laboratory. Dr. Donohoe leads research projects using NREL's Biomass Surface Characterization Laboratory to understand structural changes during biomass conversion and using electron tomography to study the complex 3-D architecture of the plant cell walls at the macromolecular scale. He received his PhD in Molecular and Cellular Biology from the University of Colorado.





Unveiling signatures of feedstock variability ***Part 1 – Sources of variability and impacts on quality***

Allison E. Ray, Ph.D. (INL)

Bryon S. Donohoe, Ph.D. (NREL)

Feedstock-Conversion Interface Consortium (FCIC) Webinar Series

April 29, 2021



Feedstock Variability Team: Collaborating Across Six Labs



Chenlin Li, Ph.D.



Kuan-Ting Lin, Ph.D.



Ling Ding, Ph.D.



Allison Ray, Ph.D.



Bryon Donohoe, Ph.D.



Josie Gruber



Renee Happs, Ph.D.



Gary Groenewold, Ph.D.



Brittany Hodges Ph.D.



Amber Hoover



Kenneth Sale, Ph.D.



Yooli Light, Ph.D.



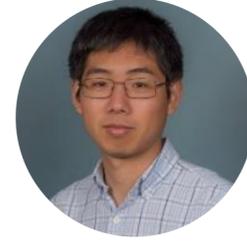
Yining Zeng, Ph.D.



Elizabeth Bose



Deepti Tanjore, Ph.D.



Jipeng Yan, Ph.D.



Troy Semelsberger, Ph.D.



Juan Leal, Ph.D.



Erin Webb, Ph.D.



Femi Oyedeji, Ph.D.



Ning Sun, Ph.D.



Oslo Jacobson

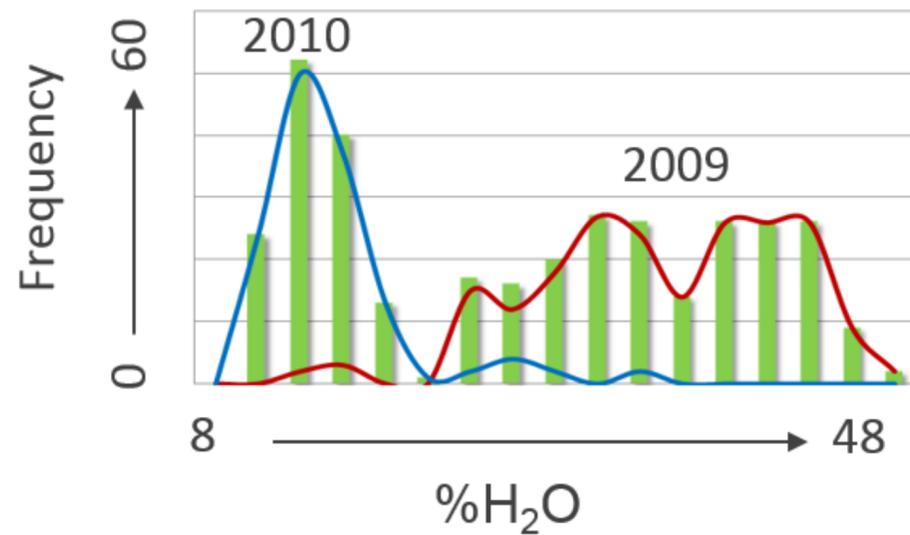


Ethan Oksen

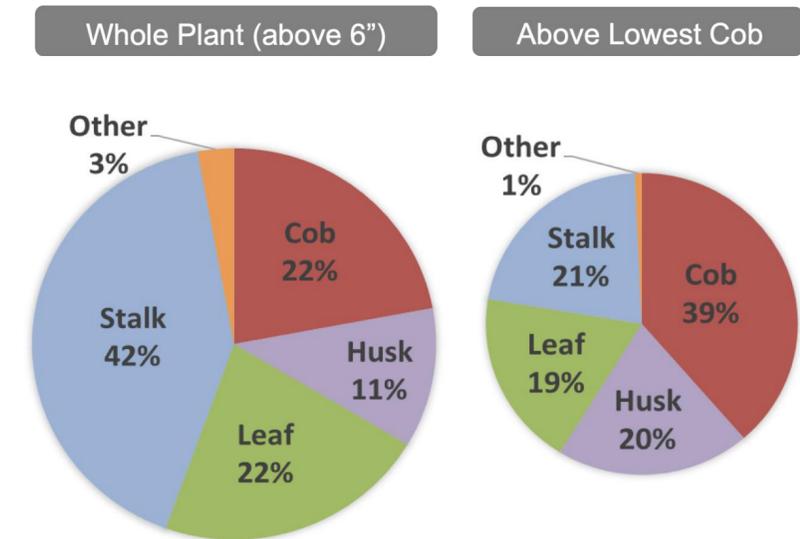
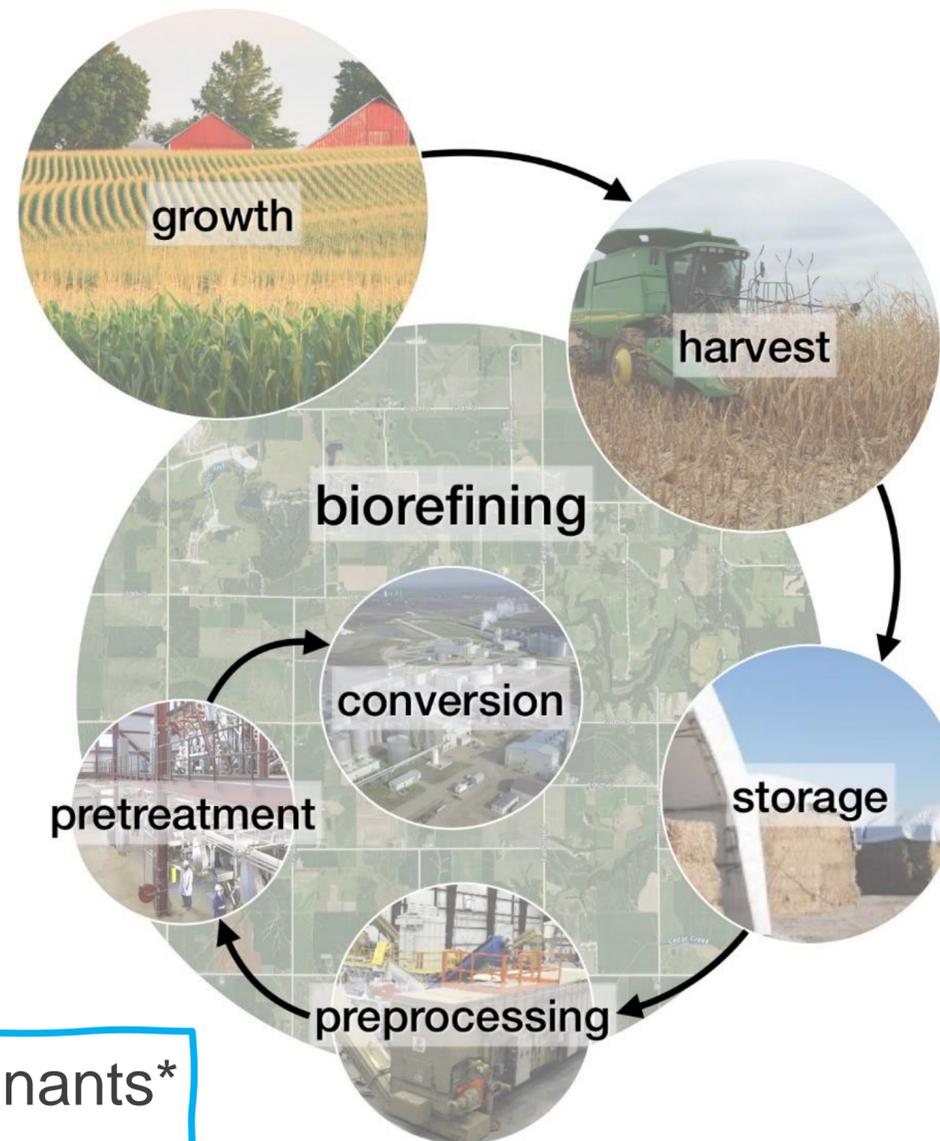


Feedstock variability cited as a major operational challenge*

feedstock variability



moisture content variability in corn stover



anatomical fractions represent a source of **inherent variability**



field-side storage of corn stover represents a source of **introduced variability** due to biological degradation that alters bulk composition and structural integrity

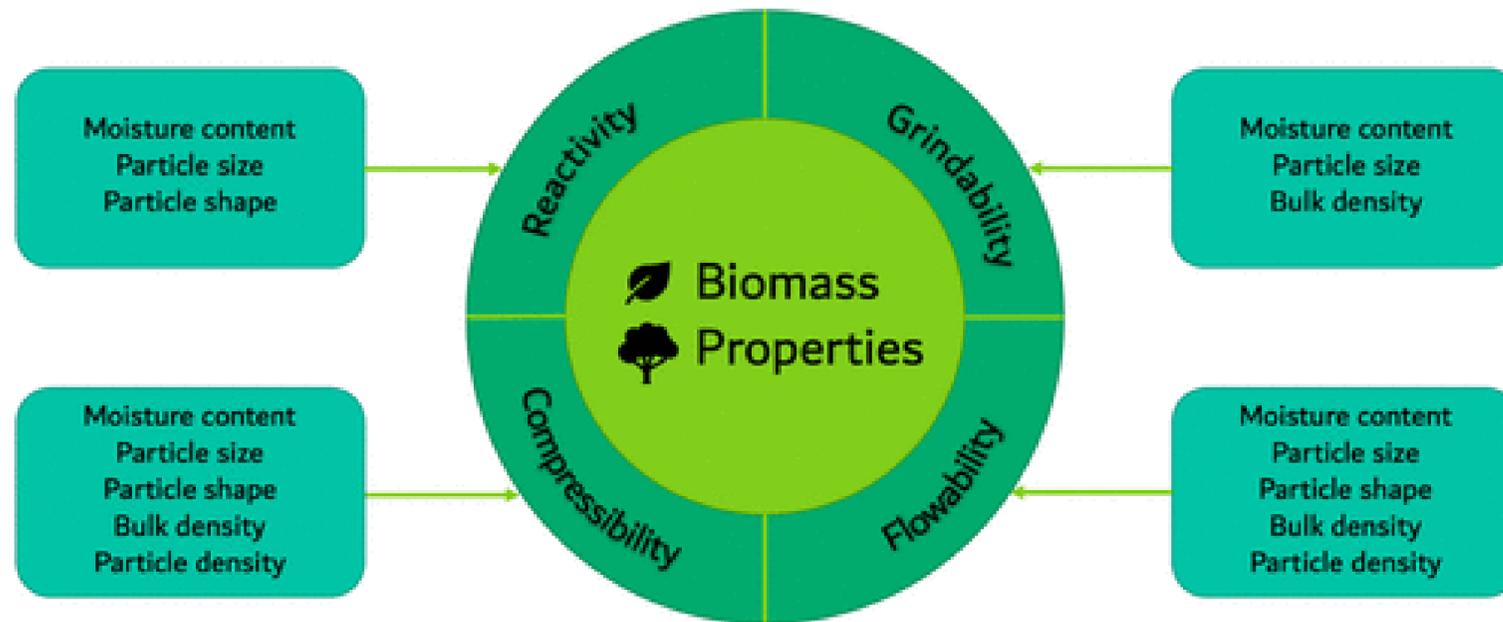
Intrinsic ash, moisture, and soil contaminants* noted as critical factors that impact biomass quality, process uptime, and throughput



Characterizing variability in lignocellulosic biomass



Advanced characterization is required for understanding feedstock variability and material attributes that impact quality.

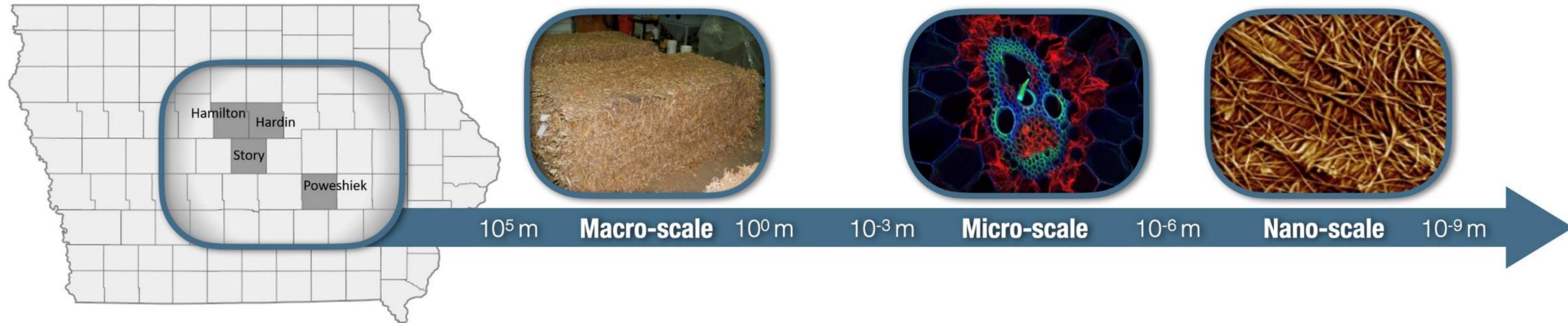


- Feedstock variability is understood largely based on chemical composition
 - Physical and mechanical properties impact behavior in various unit operations
 - Varies with biomass type and processing method
 - Requires advanced analytical methods
- Knowledge of complex property interactions is essential for understanding implications on downstream preprocessing and conversion
- Develop tools that quantify & understand the sources of biomass resource and feedstock variability

J. Yan, O. Oyediji, J.H. Leal, B.S. Donohoe, T.A. Semelsberger, C. Li, A.N. Hoover, E. Webb, E.A. Bose, Y. Zeng, C.L. Williams, K.D. Schaller, N. Sun, A.E. Ray, & D. Tanjore, Characterizing Variability in Lignocellulosic Biomass: A Review, *ACS Sus Chem Eng*, 2020 8 (22), 8059-8085, DOI: [10.1021/acssuschemeng.9b06263](https://doi.org/10.1021/acssuschemeng.9b06263).



Biomass attributes vary at all spatial scales



Motivation

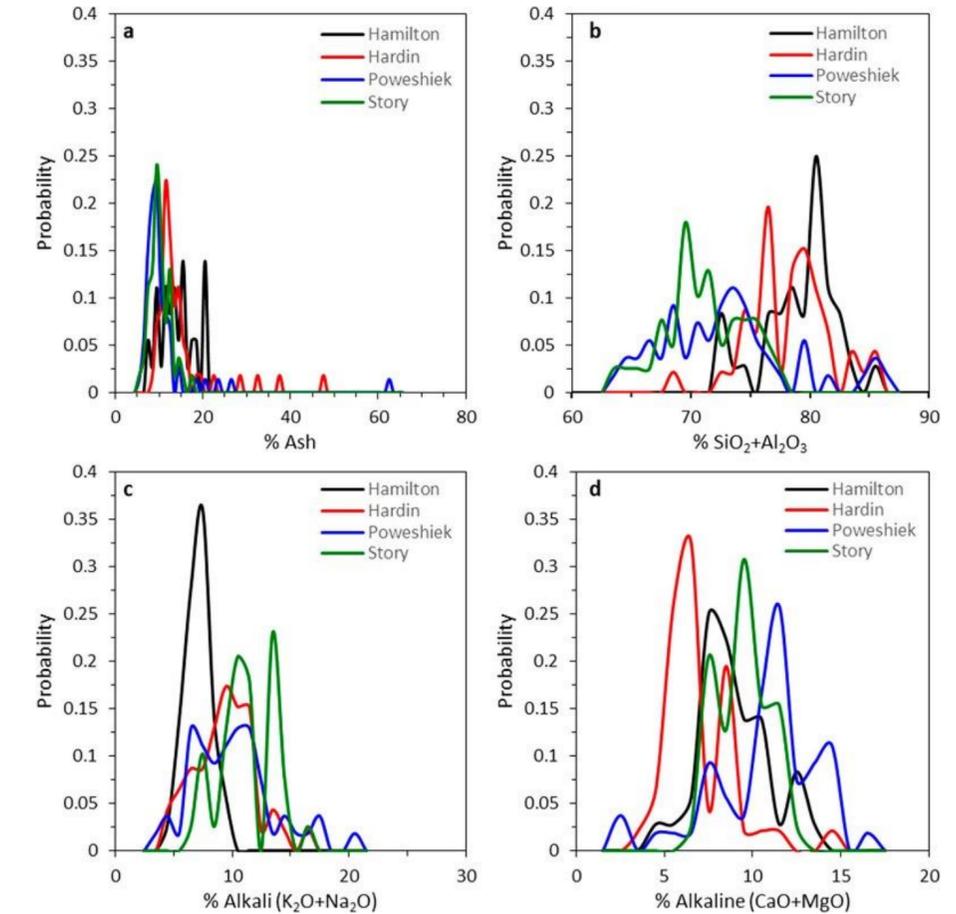
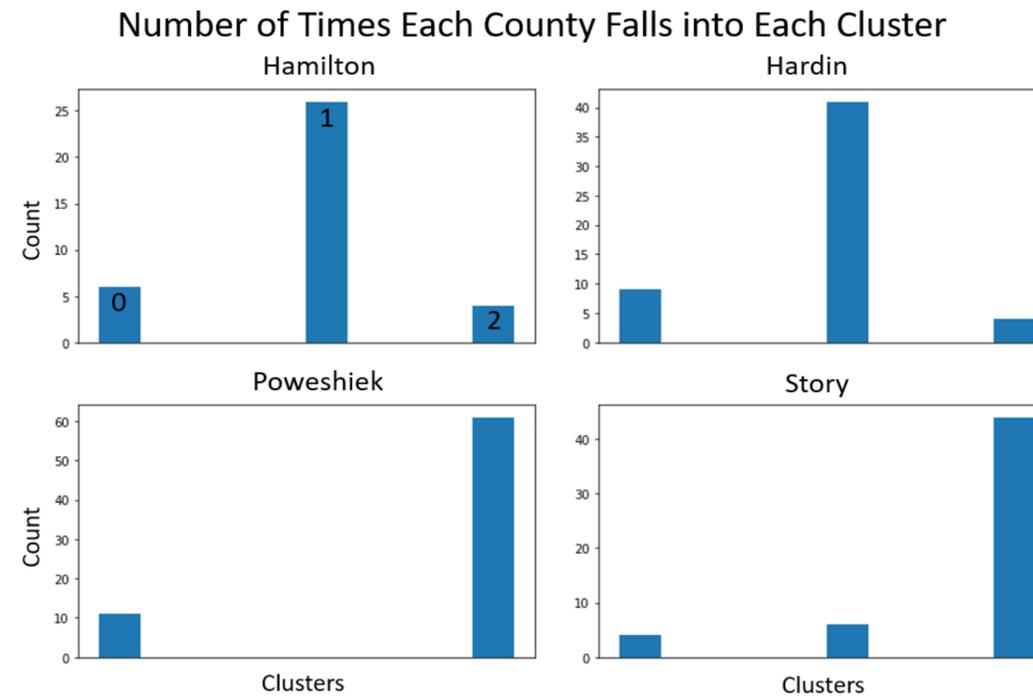
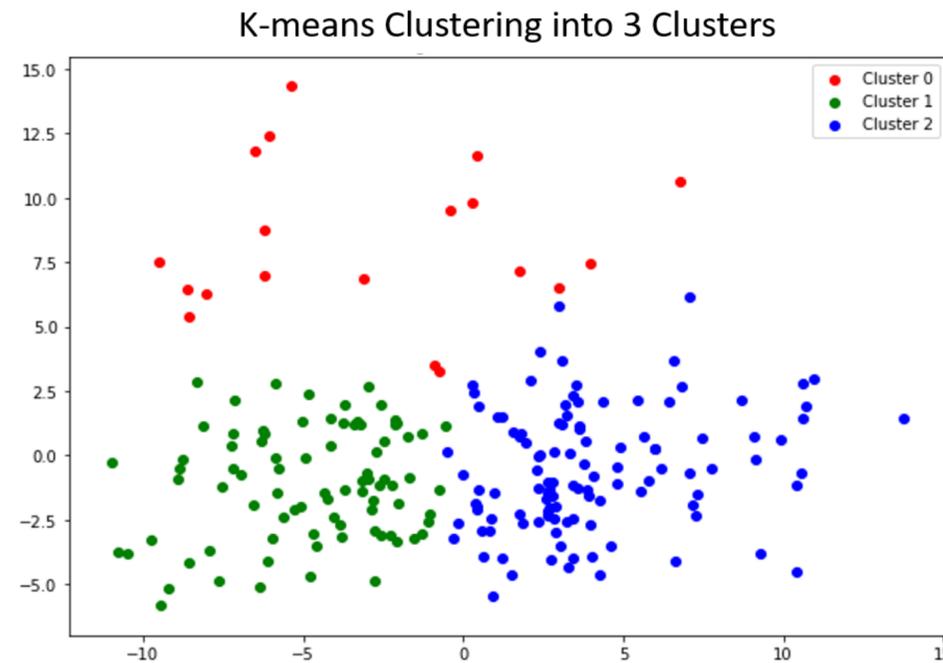
Physicochemical and structural variability exist at multiple scales; each scale offers unique insights to the sources of variability and material attributes that impact the biomass value chain.

Emergent properties increase the complexity and cost of biorefinery operations.

A.E. Ray, C.L. Williams, A.N. Hoover, C. Li, K.L. Sale, R.M. Emerson, J. Klinger, E. Oksen, A. Narani, J. Yan, C.M. Beavers, D. Tanjore, M. Yunes, E. Bose, J.H. Leal, J.L. Bowen, E.J. Wolfrum, M.G. Resch, T.A. Semelsberger, & B.S. Donohoe, Multiscale Characterization of Lignocellulosic Biomass Variability and Its Implications to Preprocessing and Conversion: a Case Study for Corn Stover, *ACS Sustainable Chemistry & Engineering* 2020 8 (8), 3218-3230, DOI: [10.1021/acssuschemeng.9b06763](https://doi.org/10.1021/acssuschemeng.9b06763).



Data analytics for understanding county-level, regional variability



Clustering results of 16 combined organic and inorganic features reveal connection back to county

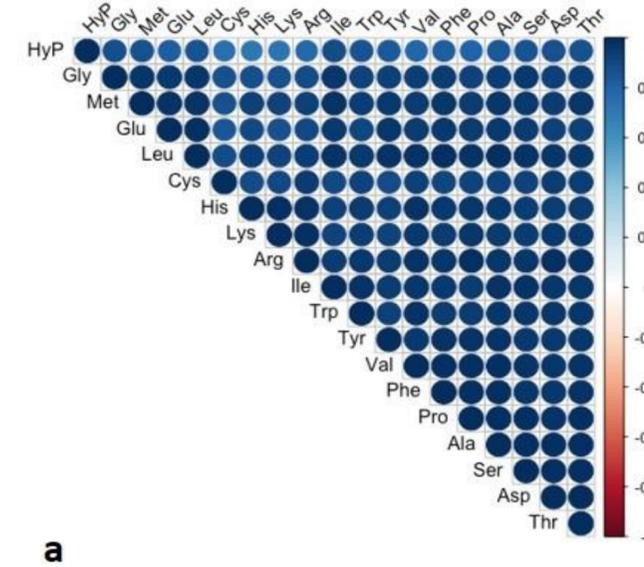
- Variability exists among counties within a realistic, biorefinery supply shed
- Bulk measures of moisture and ash are not sufficient for understanding biomass variability

Data analytics reveal insights to origins of variability

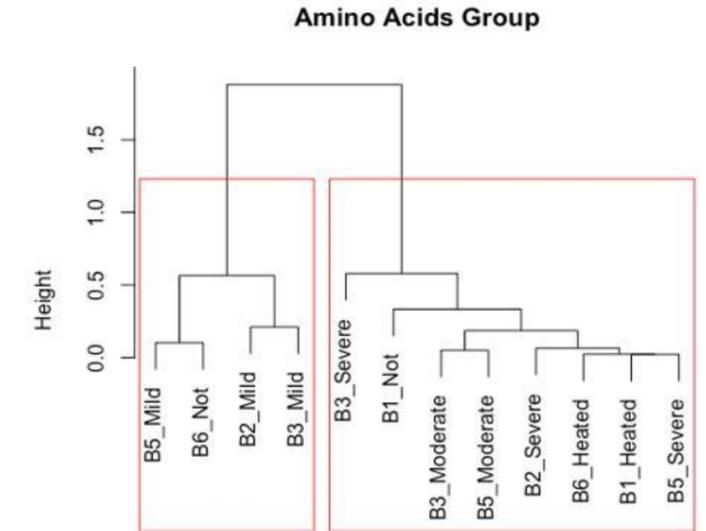


Data analytics can be used to glean key insights about sources of variability that affect biomass quality

- Correlation matrices and hierarchical clustering of compositional components
- Insights to key sources of variability
- Amino acid profiles in degraded stover (a) revealed that samples clustered as a function of extent of biological degradation (exception, bale 1 (b))
- Inorganic species in degraded samples (c) revealed a connection to harvest, mapping back to the original bales (exception, bale 3 (d))

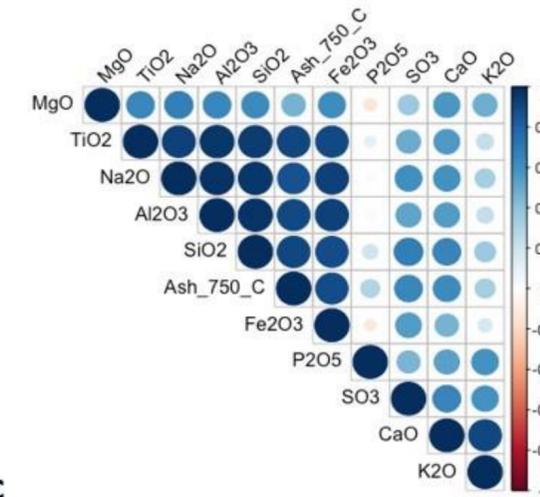


a

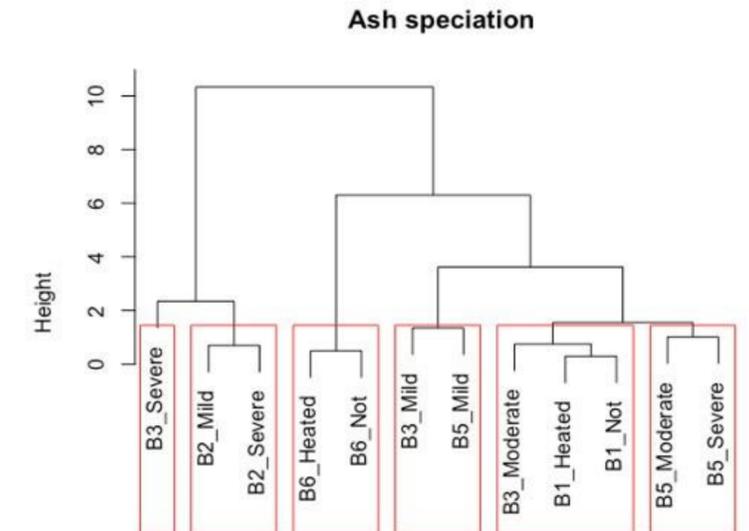


b

d
hclust (*, "ward.D2")



c



d

d
hclust (*, "ward.D2")

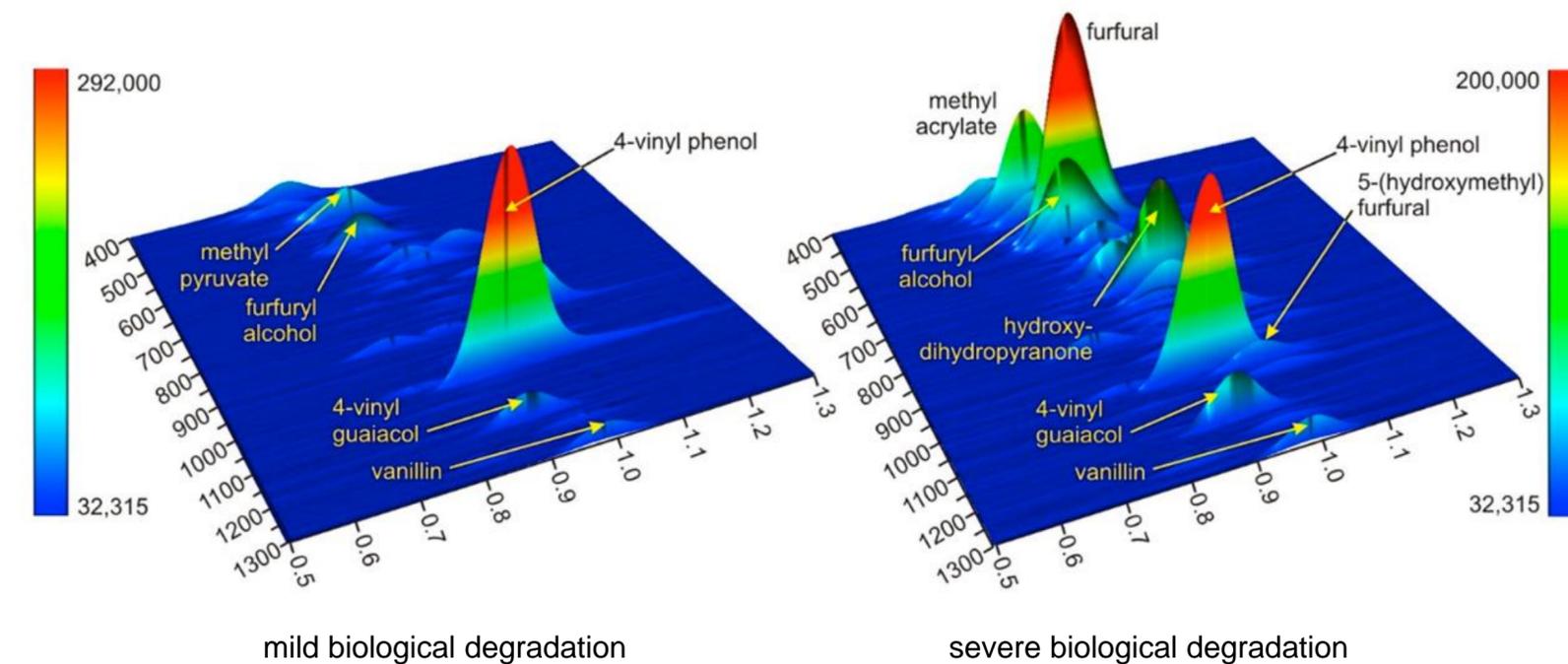


Unveiling signatures of biologically driven biopolymer modification - hemicellulose



Findings suggest that biological heating disrupts cell wall structure, fragmenting the hemicellulose or cellulose chains

- Molecular characterization approach to elucidate cell wall modification in biologically degraded corn stover.
- Low-temperature (400°C), analytical pyrolysis may offer improved characterization for identification of cell wall structural changes.
- Enhances understanding and management of variability to inform harvest and storage practices to enable the biomass value chain.



Unveiling signatures of biologically driven biopolymer modification – hemicellulose & lignin



Structural properties of chemical components, hemicellulose and lignin, were modified during biological degradation affecting enzyme hydrolysis.

Current Knowledge Gap

- Biological degradation during storage was observed by appearance and color change. There is lack of fundamental characterization of structural modifications in response to biological degradation.

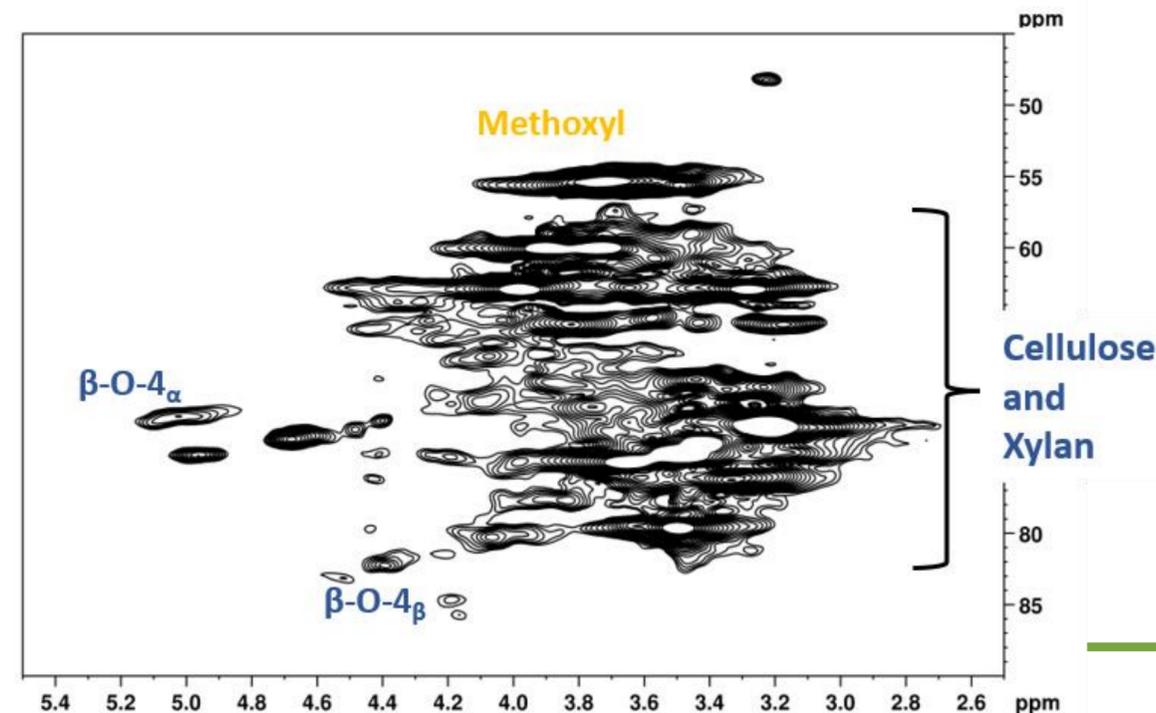
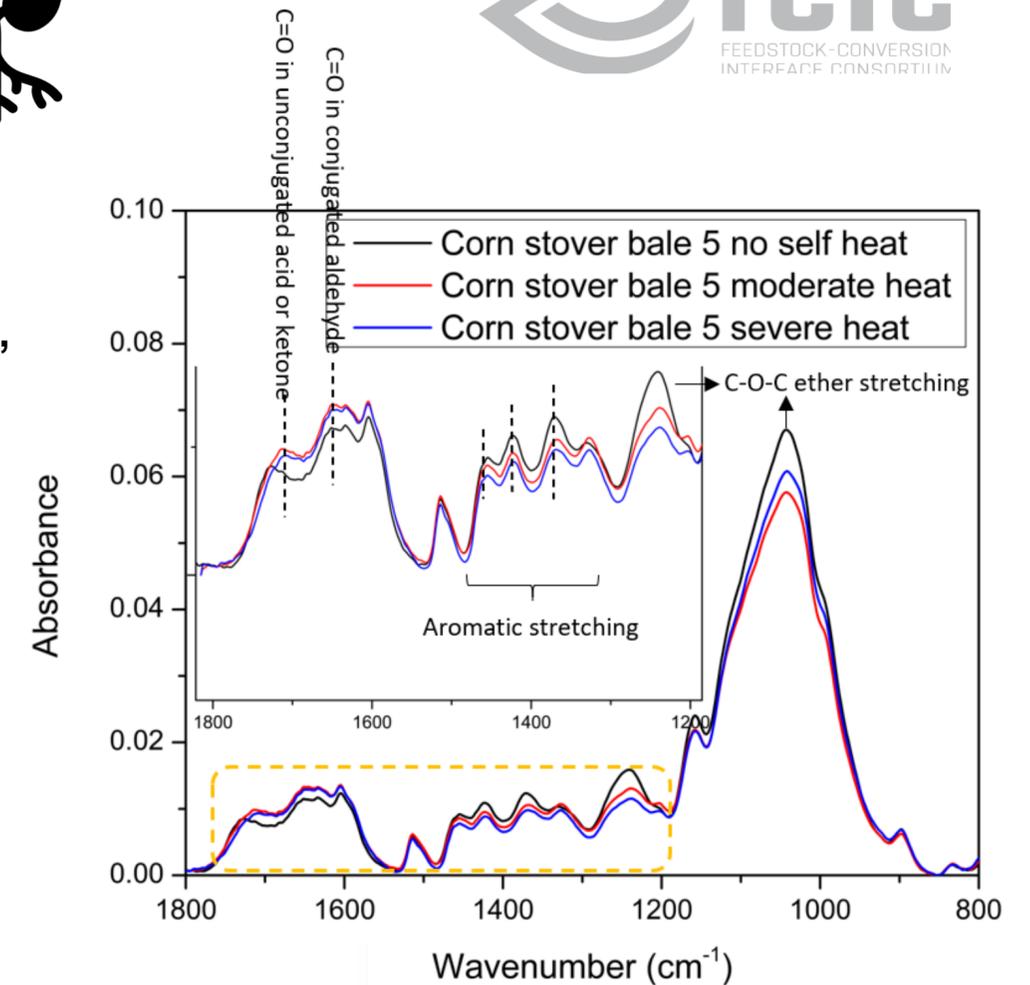
Achievement

- FT-IR and HSQC NMR spectroscopy were applied to understand the structural properties of lignin and hemicellulose.
- Results suggest oxidation of lignin, ether cleavage of lignin, and hydrolysis of hemicellulose occurred in degraded corn stover, consistent with py-GCMS.

Relevance

- Provides insights to understand the mechanism of biological self-heating process.
- Informs critical structural changes to pretreatment and conversion process.

FT-IR:
Kuan-Ting Lin,
Ph.D. (INL)



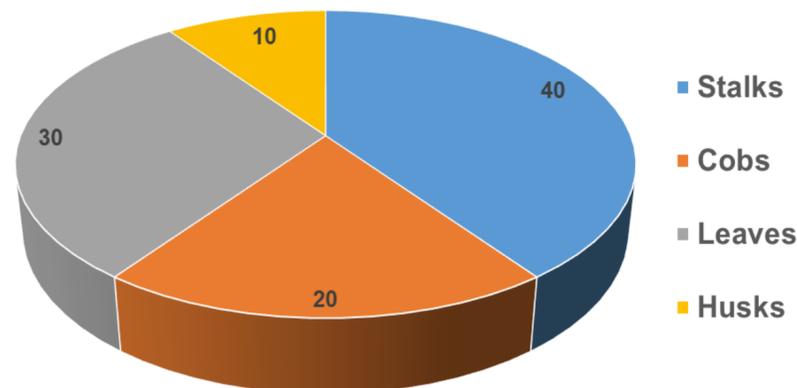
HSQC NMR:
Renee Happs,
Ph.D. (NREL)



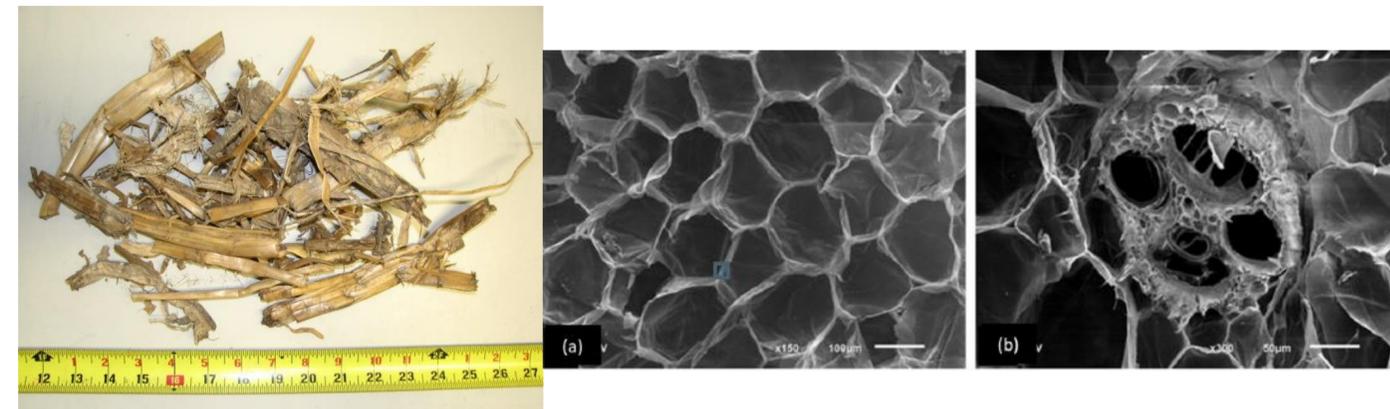
Variability is inherent to biomass

- Traditional approaches of whole plant utilization ignore the *inherent variability* at the *anatomical and tissue scale*
- Anatomical fractions have *variable responses* to mechanical and chemical processing

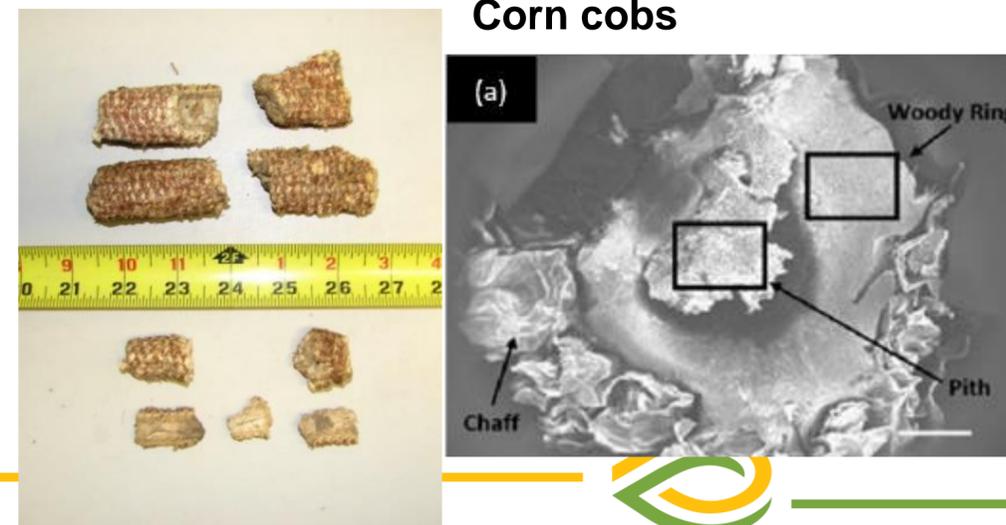
% of Fractions in Corn Stover Bale



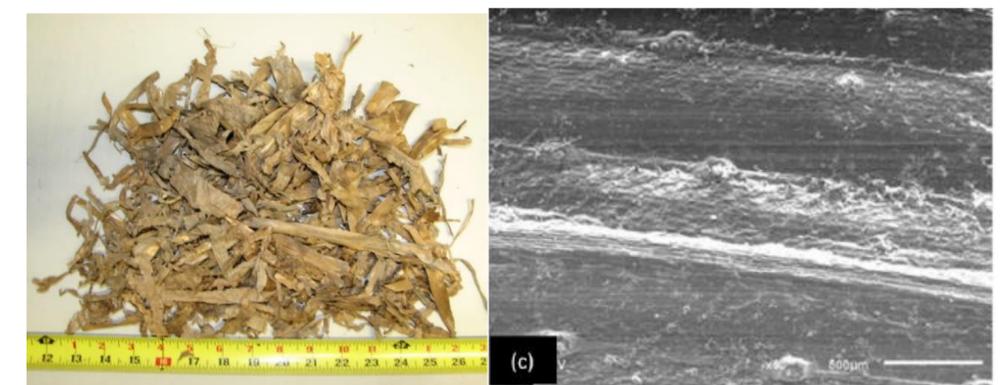
Corn stover stalks



Corn cobs



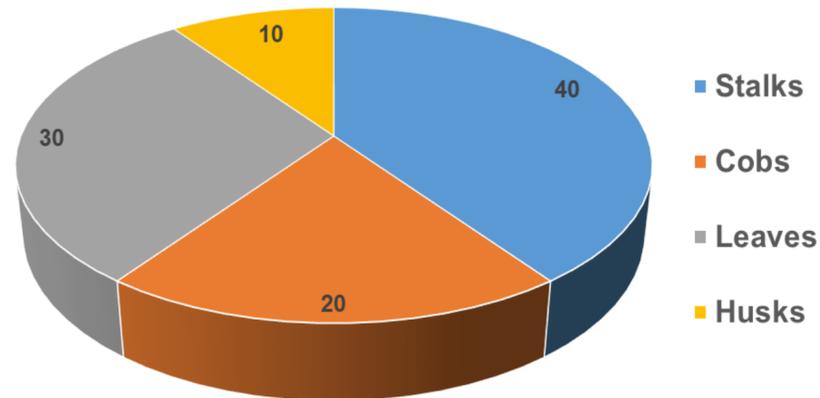
Corn stover leaves



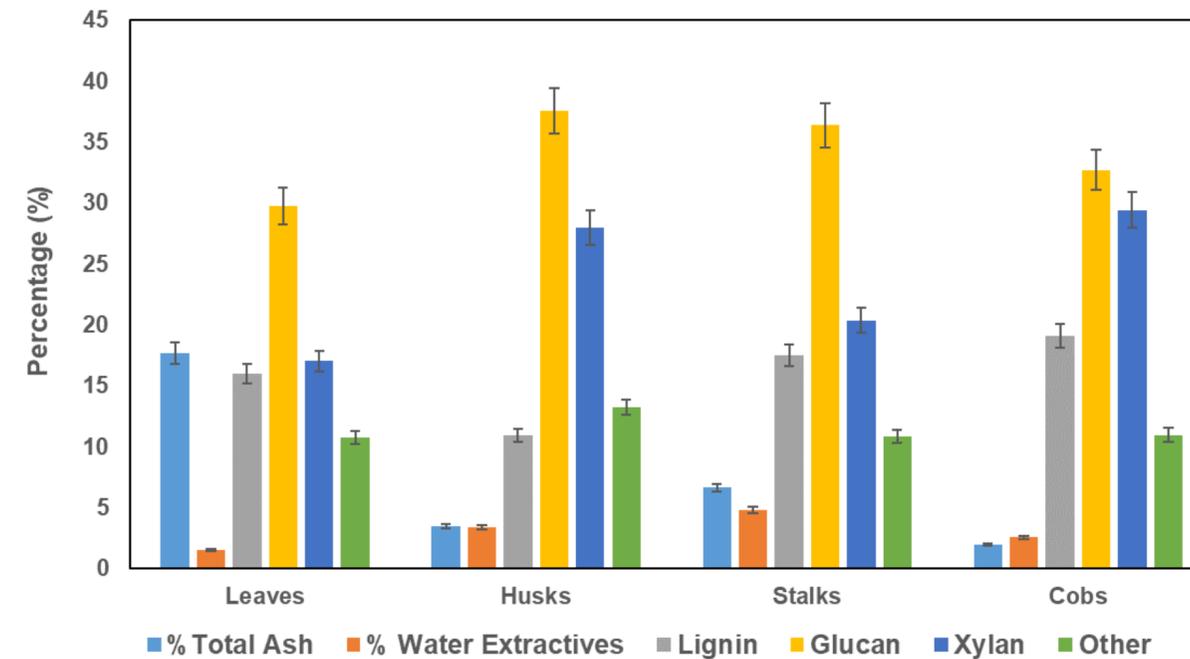
Inherent and Introduced Compositional Variability in Anatomical Fractions



% of Fractions in Corn Stover Bale

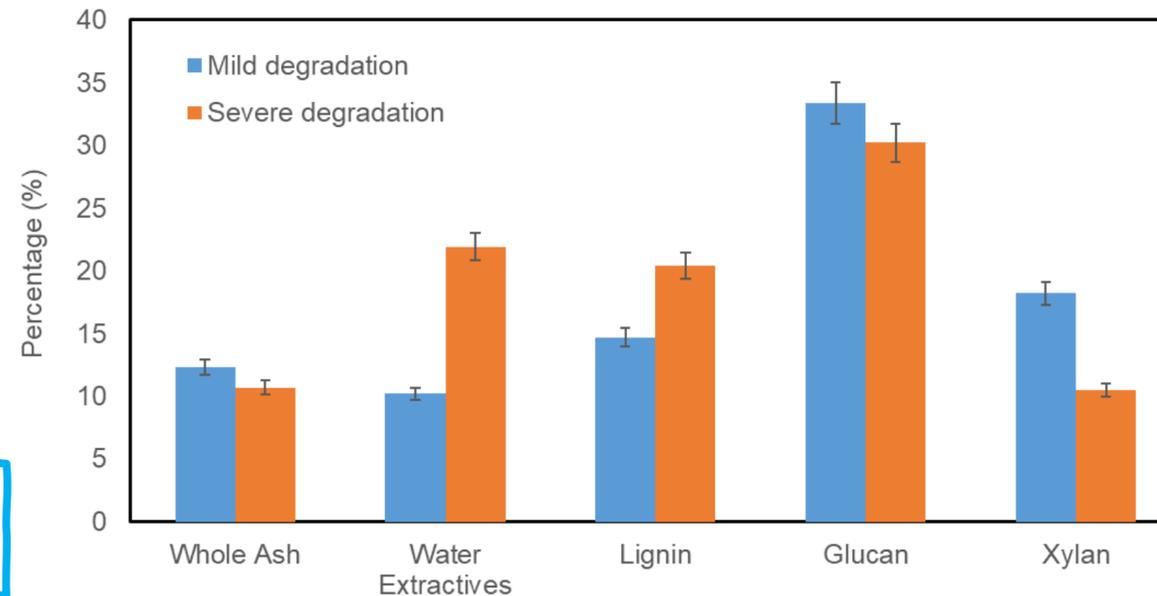


Chemical Compositions of Corn Stover Fractions from Hand Harvest



Dissection of biologically-degraded corn stover bales collected from field-side storage

Changes of Bulk Corn Stover Composition under Biological Degradation



Variations of Inorganic Species in Fractions under Mild and Severe Degradation

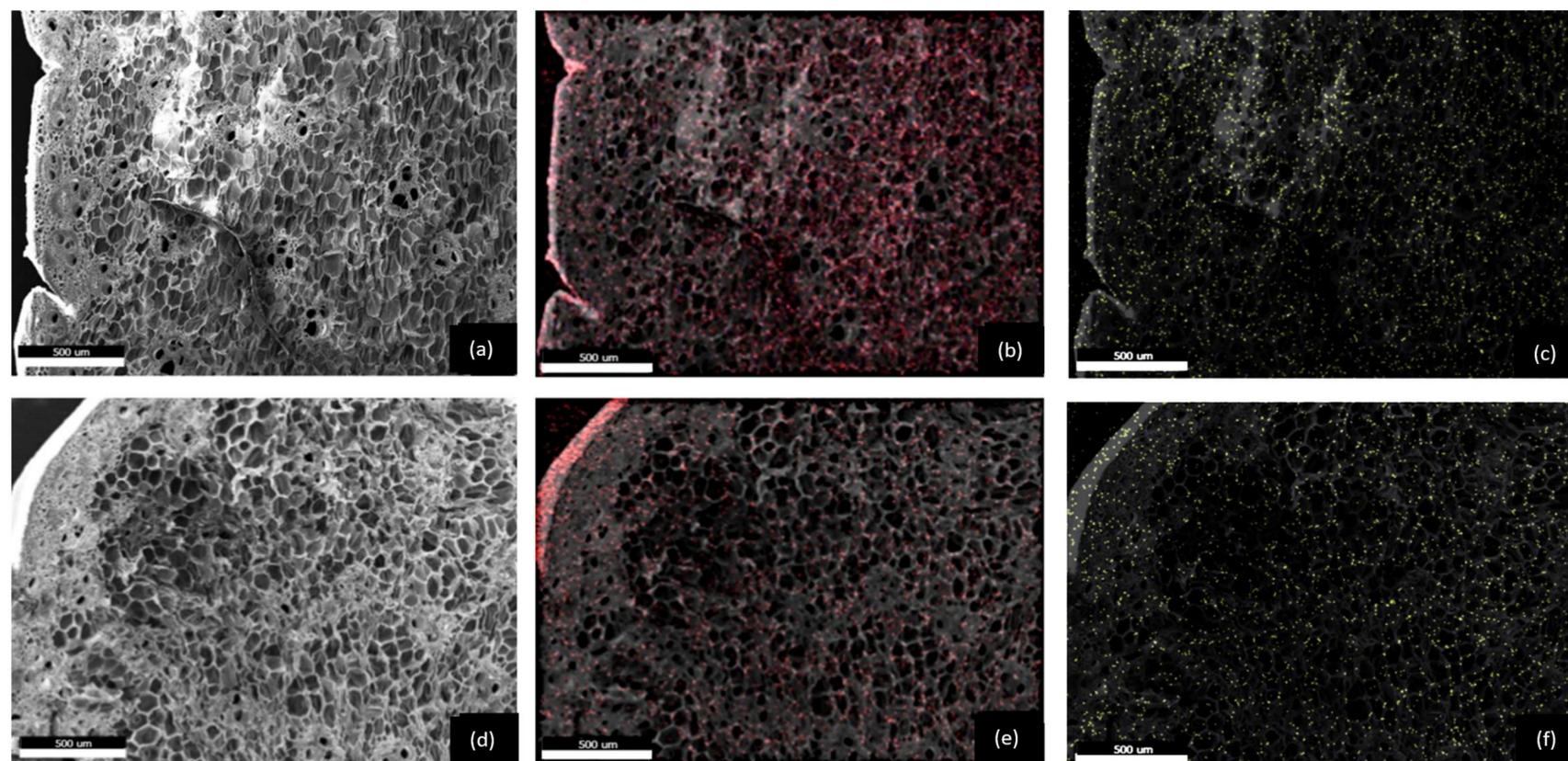
Stover Fraction and Degradation Level	Total Ash % w/w	Al as	Ca as	K as	Si as
		Al ₂ O ₃ % w/w	CaO % w/w	K ₂ O % w/w	SiO ₂ % w/w
Cob – Mild	4.91	0.26	0.10	1.00	2.67
Cob – Severe	3.92	0.19	0.15	1.07	1.83
Stem – Mild	7.28	0.40	0.34	1.69	3.60
Stem – Severe	5.98	0.26	0.34	1.41	2.89
Leaf – Mild	14.21	0.61	0.67	1.36	9.72
Leaf – Severe	13.07	0.57	0.69	1.40	8.48



Elemental Distribution and Compositional Variability Introduced by Storage Degradation

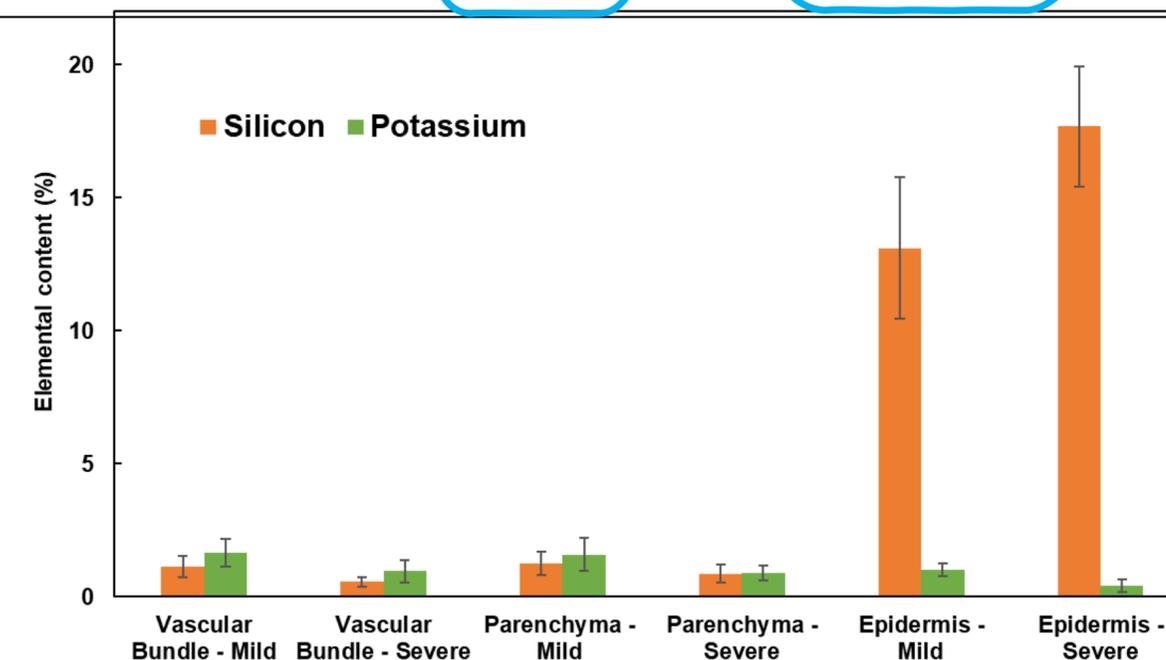


SEM-EDS mapping of inorganic elemental distribution and variability in the stem cross-section (top: mild degradation; bottom: severe degradation)



Compositional Analysis of Corn Stover Stems under Mild and Severe Biological Degradation

Degradation	Whole Ash	Whole Protein	Water Extractives	Lignin	Glucan	Xylan	Other	Mass Balance
Mild	7.89 (0.11)	2.83 (0.18)	9.61 (0.02)	18.33 (0.47)	37.16 (0.50)	18.11 (0.35)	5.45 (0.47)	97.86 (2.29)
Severe	6.39 (0.02)	2.93 (0.11)	20.67 (0.14)	22.16 (1.18)	32.90 (1.39)	11.94 (0.10)	1.68 (0.02)	98.96 (0.10)

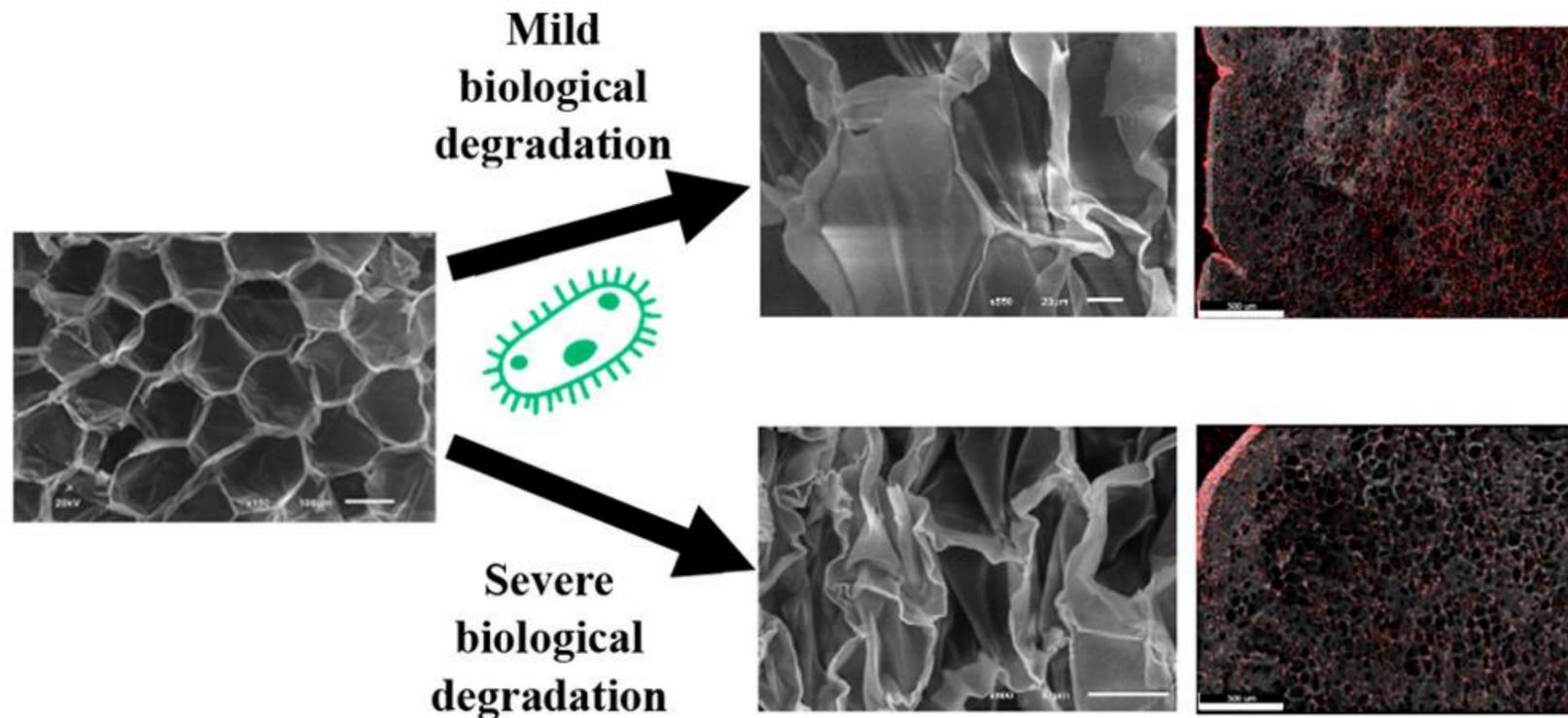


- Severe biological degradation caused the translocation of silicon (red) from pith to outer epidermal tissues, as well as a reduction of potassium (green) in the corn stover stalk
- Increase of degradation severity also led to dramatic decrease of glucan and xylan contents and increase of extractives.

Characterizing inorganic species variability in corn stover fractions



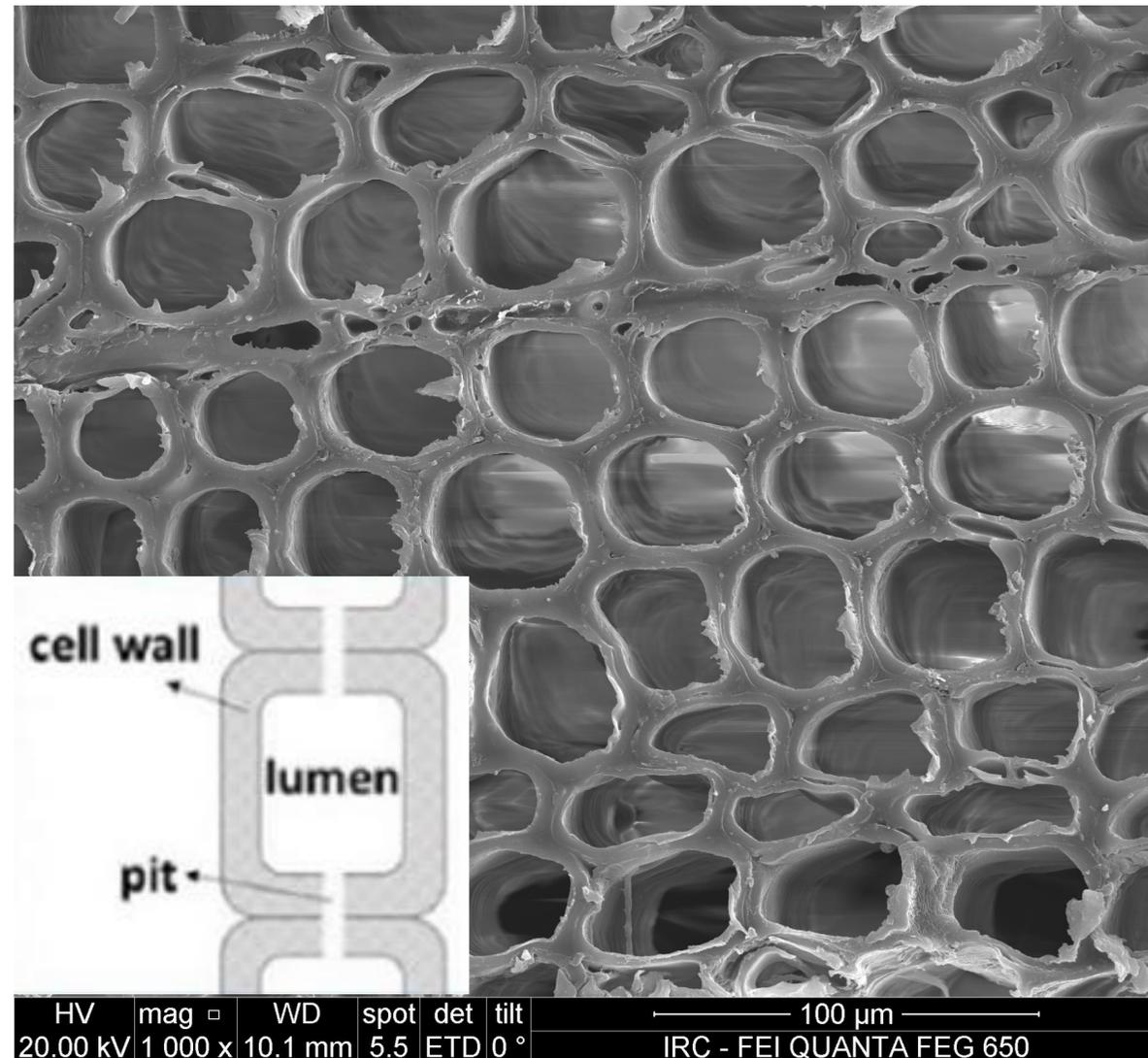
Inorganic species mapping revealed that biological heating and degradation resulted in translocation of silica from the pith to the outer epidermal tissues



- Conducted a first-of-a-kind study on the dynamic, elemental variability and distributions observed in corn stover fractions as functions of storage and biological heating.
- Provides fundamental understanding to inform strategies for harvest and collection, wear abrasion, selective biomass preprocessing technologies and equipment design toward enhanced valorization.

Water location and state

- The location and state of water varies due to the complex physical structure and chemical composition of lignocellulosic biomass



SEM image of loblolly pine chip (Photo credit: Dr. Ling Ding).

- “Water pools” in lignocellulosic biomass include bound and free water
- Free water in cell lumen and bound water interacting closely with cell wall polymers
- Water status, distribution, and interactions with microstructure influence physical and chemical changes during storage and preprocessing.

Dr. Ling Ding, Time Domain-NMR for resolution of bound and free water in anatomical fractions of pine residues and corn stover as functions of biological degradation, *Symposium for Biomaterials, Fuels, and Chemicals*, Virtual Conference, April 28, 2021.

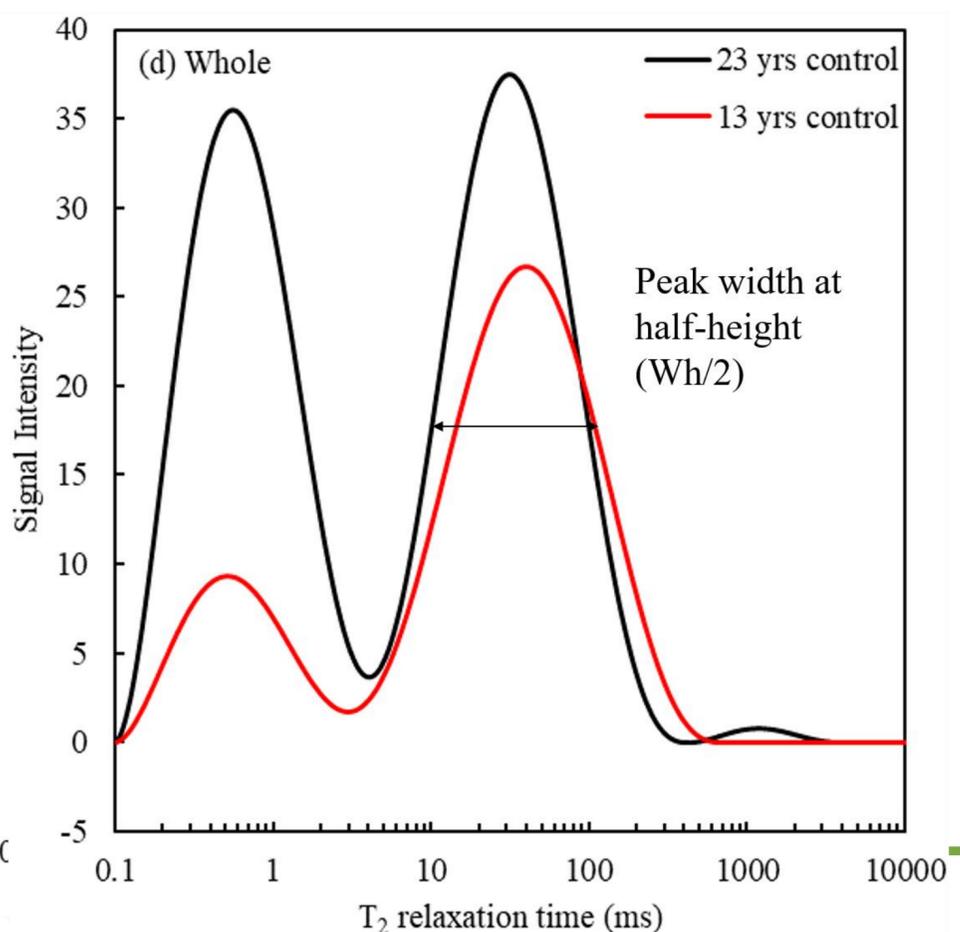
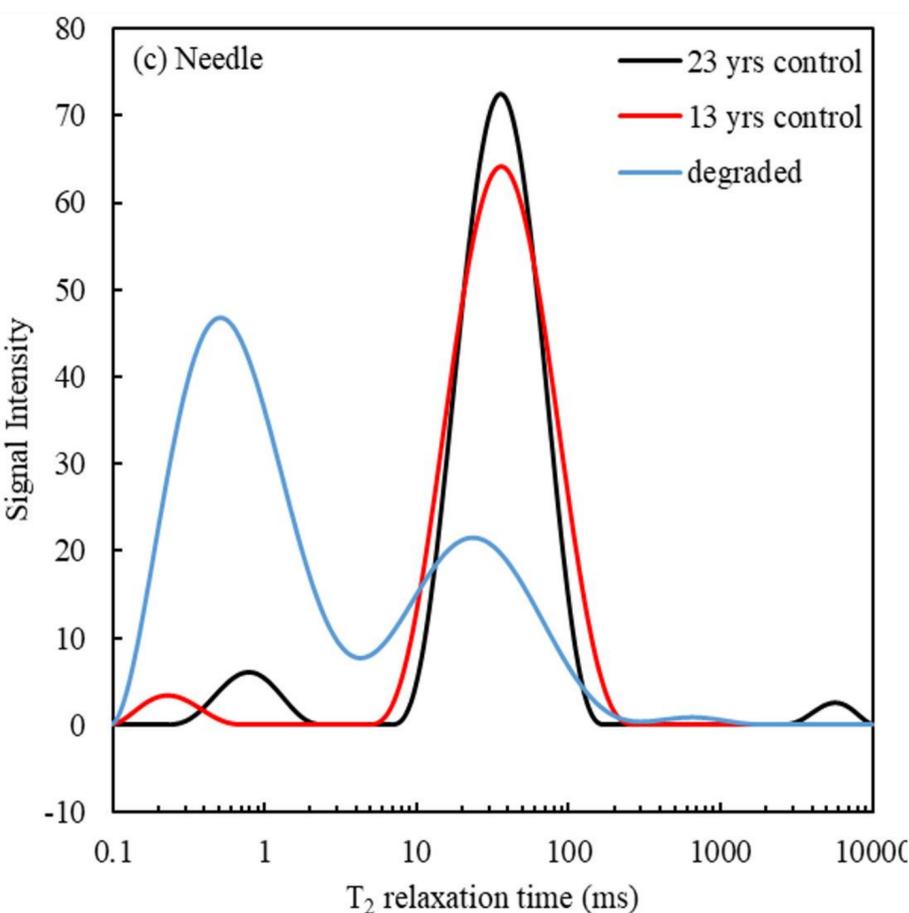
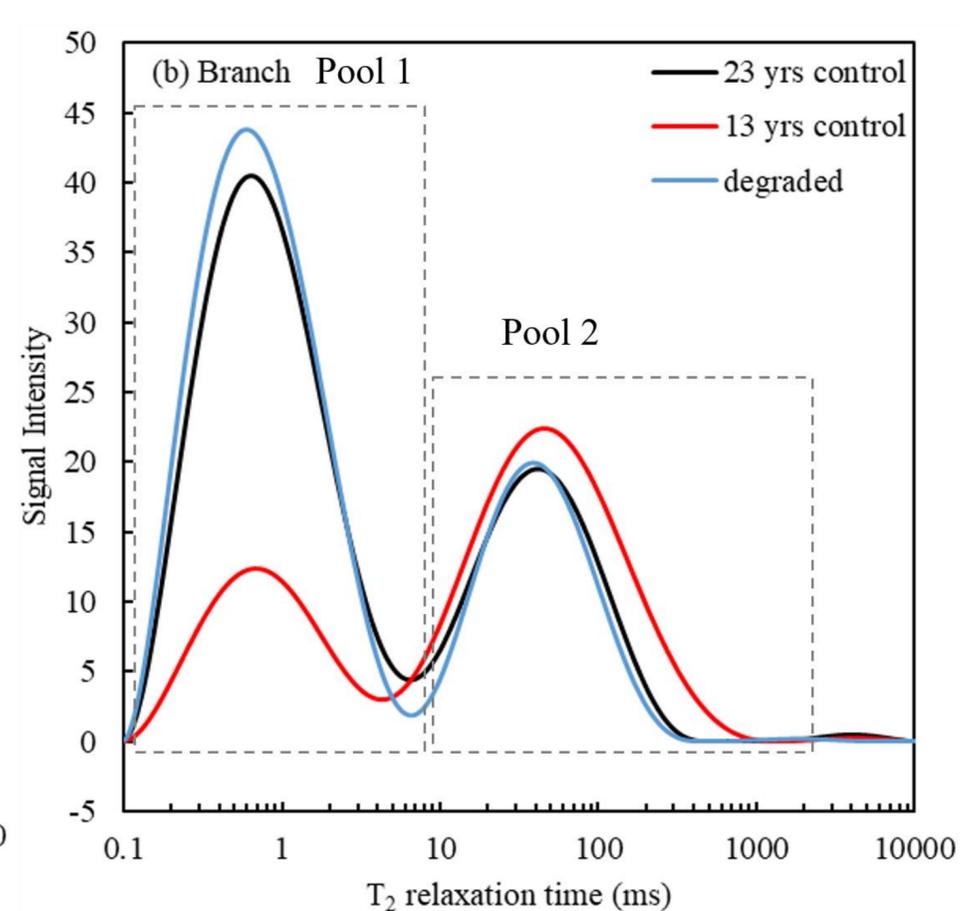
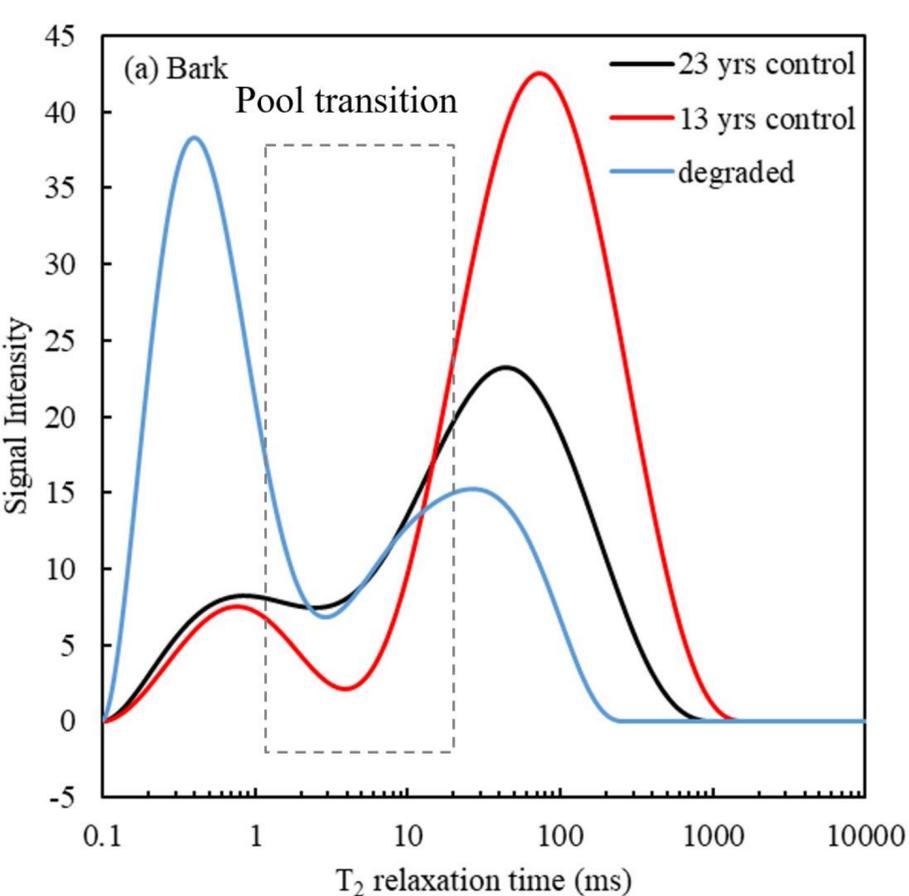


TD-NMR for resolution of water distribution in pine anatomical fractions

- Variability in relaxation times across anatomical fractions and as a function of degradation
- Peaks in the T_2 distribution denoted as pool 1 and 2 corresponding to the bound and free water associated with the cell-wall and to water in the lumen.

Dr. Ling Ding, Time Domain-NMR for resolution of bound and free water in anatomical fractions of pine residues and corn stover as functions of biological degradation, *Symposium for Biomaterials, Fuels, and Chemicals*, Virtual Conference, April 28, 2021.

Ding et al., Manuscript in preparation.

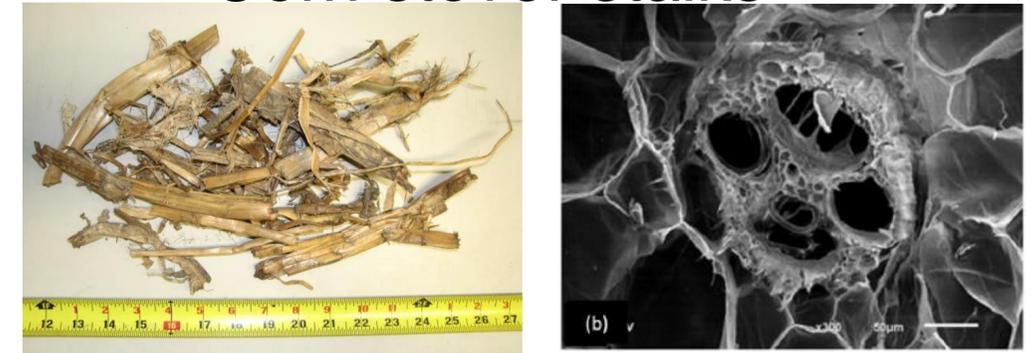


Characterization of inherent and introduced variability informs advanced fractionation and valorization

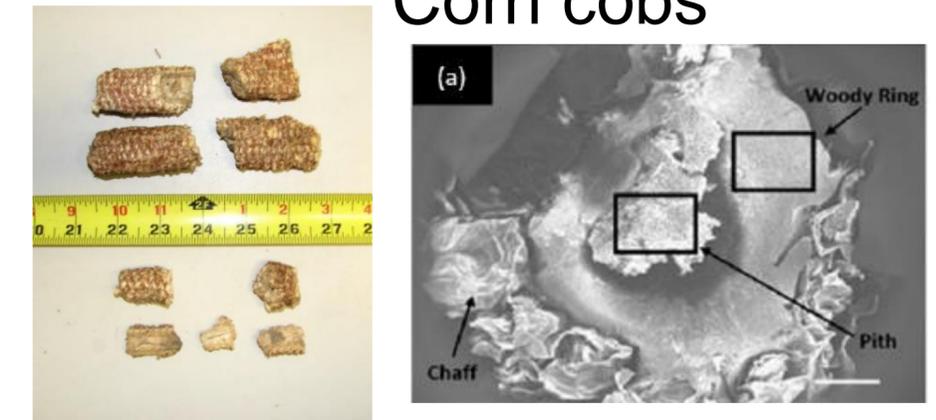


- Biological degradation and anatomical fractions are key sources of variability that confound standard approaches to bioprocessing.
 - Lignin modification measured as a function of degradation may affect potential for lignin utilization.
 - Anatomical fractions have variable responses in mechanical and chemical processing.
- Feedstock variability can be exploited to derive value from 'overlooked' fractions of biomass.
- Fundamental understanding of material attributes guides selection of process configurations and thermochemical or biological tools that counter variability for enhanced utilization and valorization.

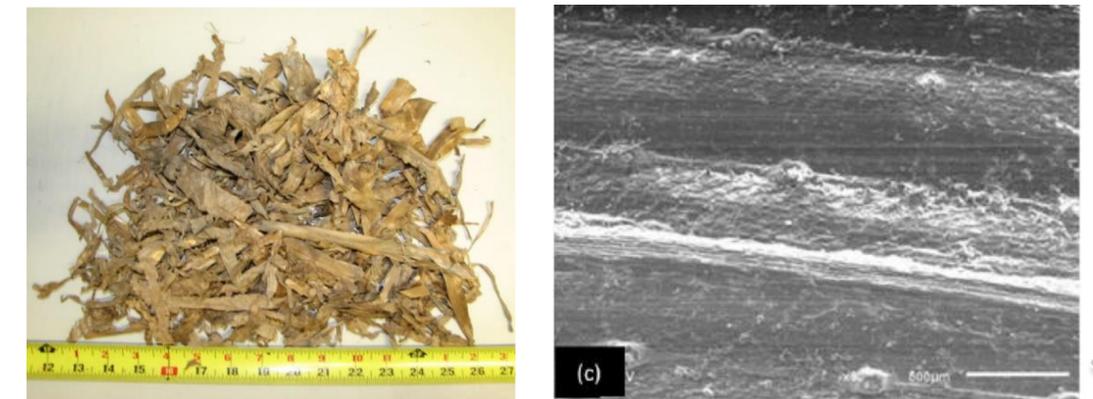
Corn stover stalks



Corn cobs



Corn stover leaves



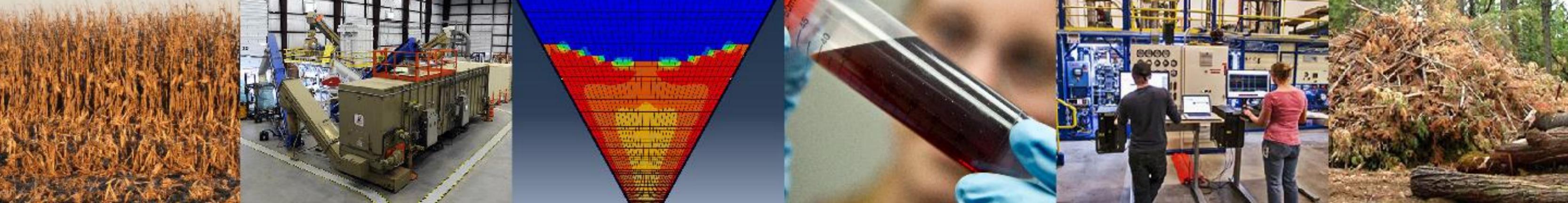
Speaker Contact Information

Allison Ray, Ph.D.

Research Excellence Lead, Science & Technology
Senior Research Scientist
Idaho National Laboratory

allison.ray@inl.gov



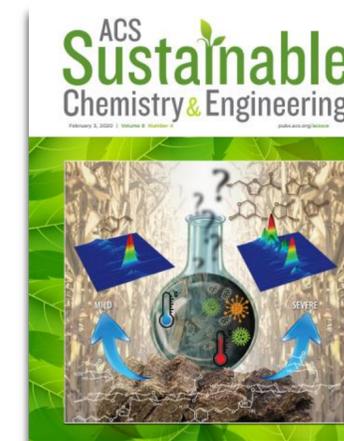
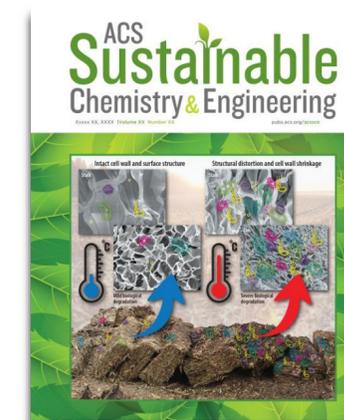


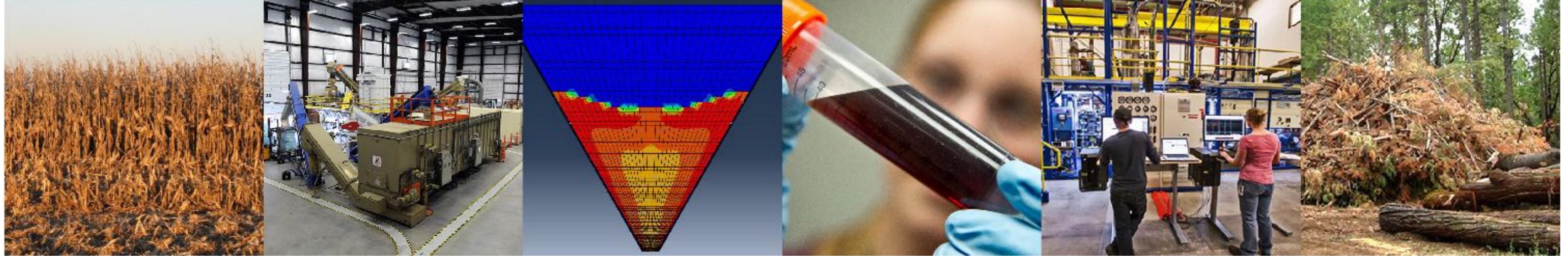
Thank you
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Unveiling signatures of feedstock variability

- E. Bose, J.H. Leal, A.N. Hoover, Y. Zeng, C. Li, A.E. Ray, T.A. Semelsberger*, and B.S. Donohoe*, Impacts of biological heating and degradation during bale storage on the surface properties of corn stover. *ACS Sus Chem Eng* 2020, 8 (37), 13973-13983. DOI: 10.1021/acssuschemeng.0c03356
- Yan, J., Oyedeji, O., Leal, J., Donohoe, B., Semelsberger, T., Li, C., Hoover, A., Sun, N., Webb, E., Bose, E., Zeng, Y., Williams, C., Schaller, K., Ray, A.*, Tanjore, D*. Characterizing variability in lignocellulosic biomass - A review. *ACS Sus Chem Eng* 2020, 8 (22), 8059-8085. DOI: 10.1021/acssuschemeng.9b06263
- Li, C.*, Kerner, P., Williams, C.L., Hoover, A., Ray, A.E.*. Characterization and Localization of Dynamic Cell Wall Structure and Inorganic Species Variability in Harvested and Stored Corn Stover Fractions as Functions of Biological Degradation. *ACS Sus Chem Eng* 2020, 8 (18), 6924-6934. DOI: 10.1021/acssuschemeng.9b06977
- Ray, A.E.*, Williams, C., Hoover, A., Li, C., Sale, K., Emerson, R., Klinger, J., Oksen, E., Narani, A., Yan, J., Beavers, C., Tanjore, D., Yunes, M., Bose, E., Leal, J., Bowen, J., Wolfrum, E., Resch, M., Semelsberger, T., Donohoe, B*. Multi-scale characterization of lignocellulosic biomass variability and its implications to preprocessing and conversion —a case study for corn stover. *ACS Sus Chem Eng* 2020, 8 (8), 3218-3230. DOI: 10.1021/acssuschemeng.9b06763
- G. Groenewold*, B. Hodges, A. Hoover, C. Li, C. Zarzana, K. Rigg, A.E. Ray*, Signatures of Biologically Driven Hemicellulose Modification Quantified by Analytical Pyrolysis Coupled with Multidimensional Gas Chromatography Mass Spectrometry, *ACS Sus Chem Eng* 2020, 8 (4), 1989-1997. DOI: 10.1021/acssuschemeng.9b06524
- Leal, J., Torres, E., Rouse, W., Moore, C., Sutton, A., Hoover, A., Li, C., Resch, M., Donohoe, B., Ray, A., Semelsberger, T*. Impacts of inorganic material (total ash) on surface energy, wettability & cohesion of corn stover. *ACS Sus Chem Eng* 2020, 8 (4), 2061-2072. DOI: 10.1021/acssuschemeng.9b06759





Unveiling Signatures of Feedstock Variability: Impacts on Materials Handling and Flowability

April 29, 2021

Feedstock-Conversion Interface Consortium Webinar Series

Bryon Donohoe & Allison Ray

National Renewable Energy Laboratory &

Idaho National Laboratory

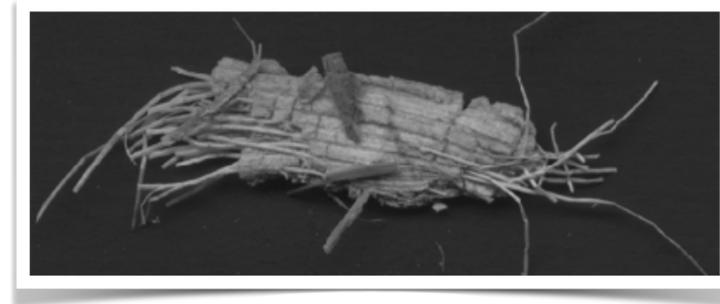


Outline

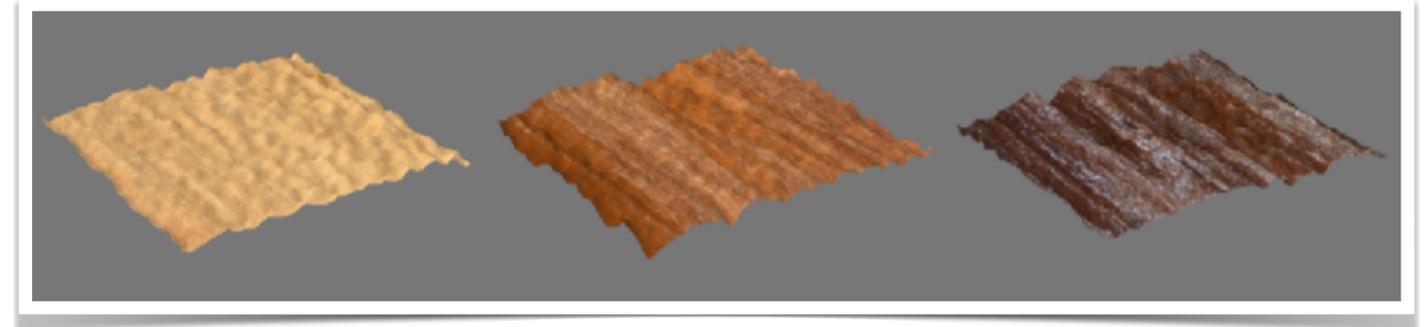
- problem/perspective



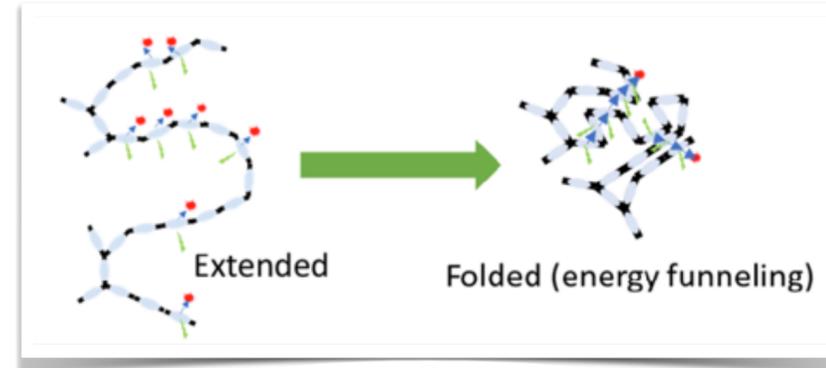
- particle morphology



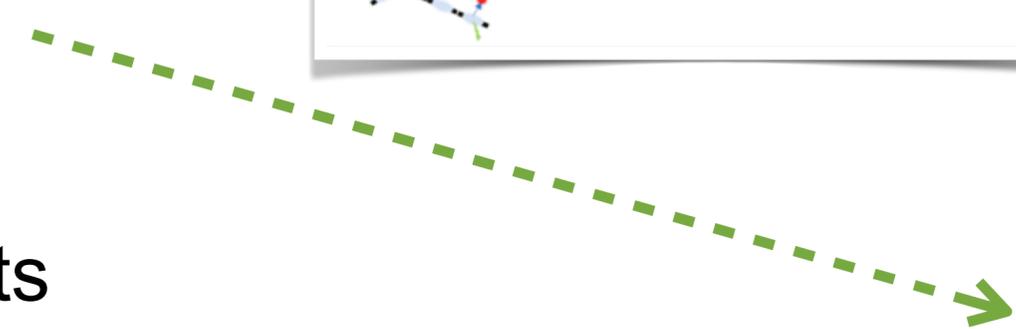
- surface properties



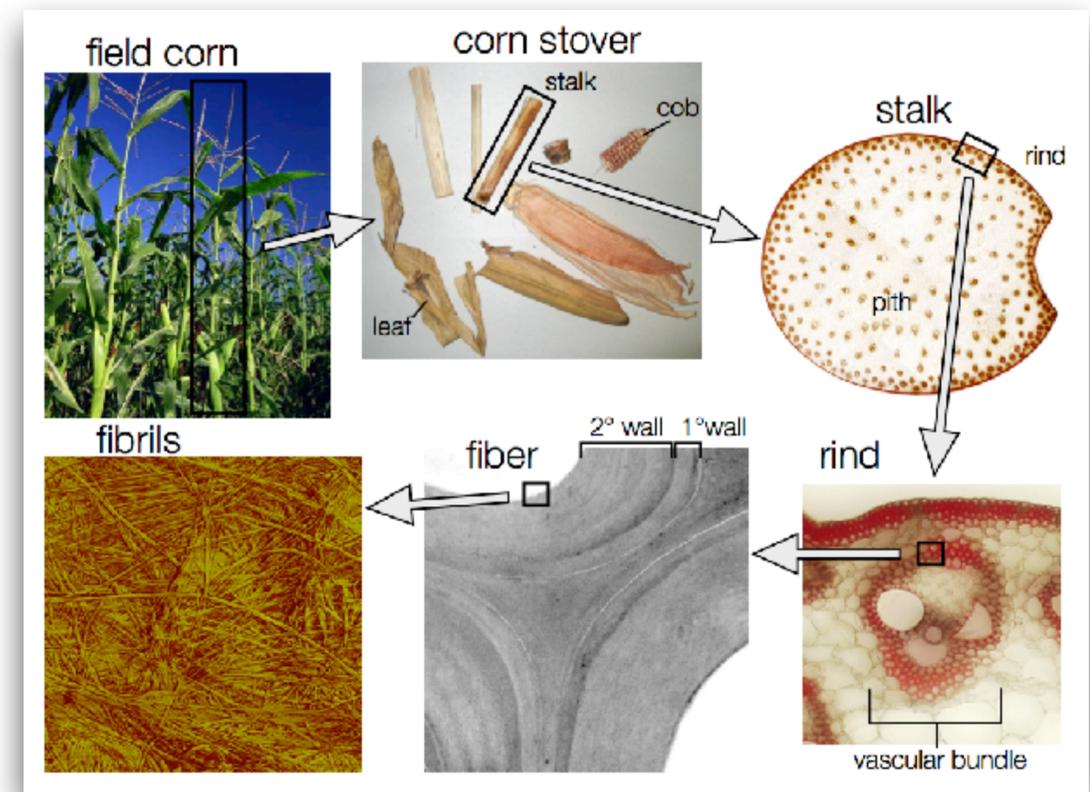
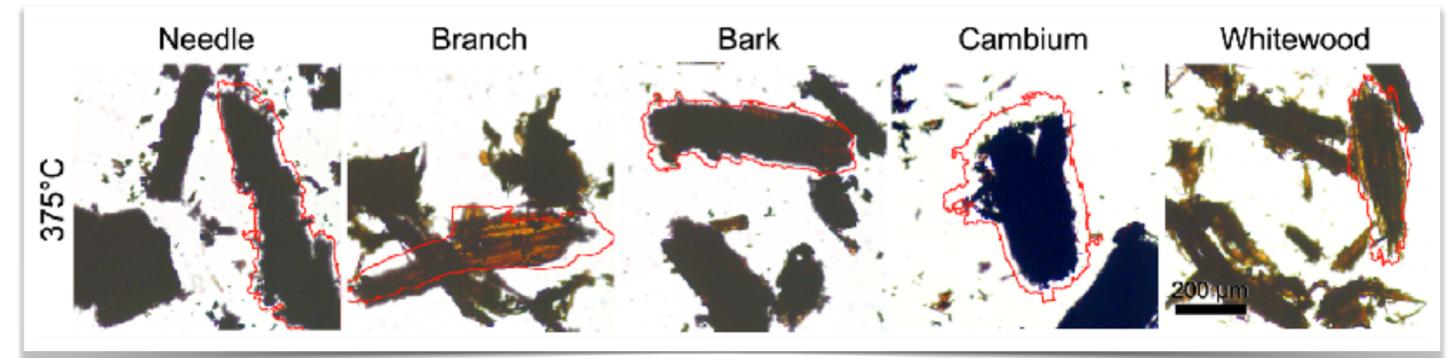
- lignin structure



- pine tissues



- final thoughts



Feedstock flowability, variability are major challenges

Challenges, recommendations, and lessons learned from over 100 participants (industry, NL, academic)

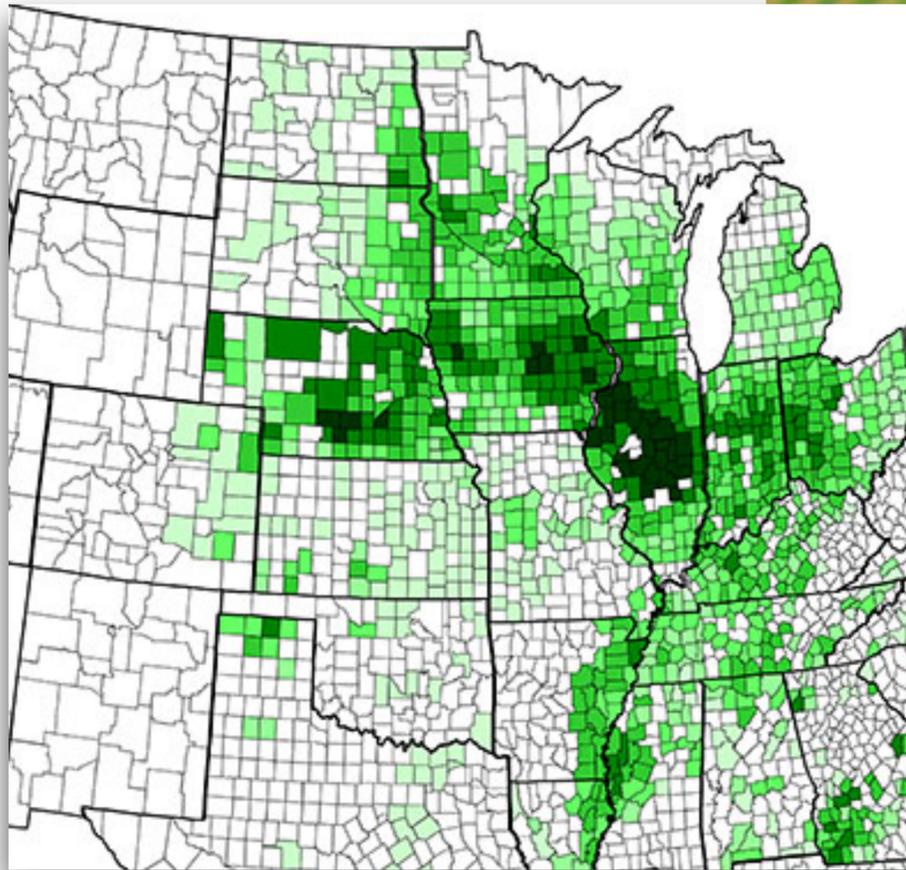
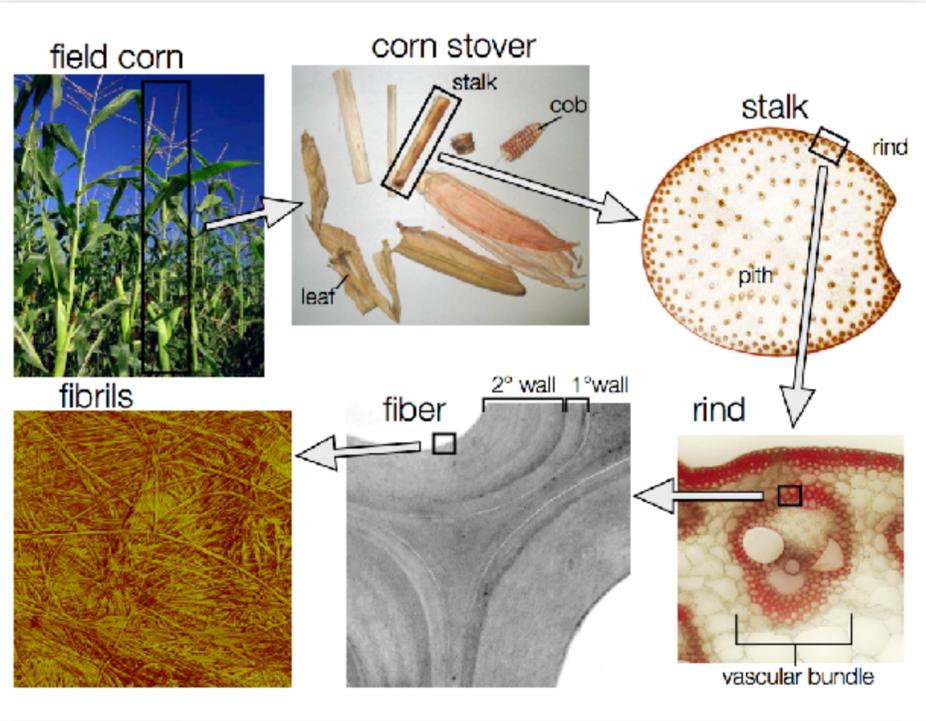
Top Concerns

- **Feedstock Flowability**
- **Feedstock Variability**
- Equipment Uptime
- Lack of Equipment Performance Data
- **Undefined Feedstock Specifications**



Variability exists at multiple scales

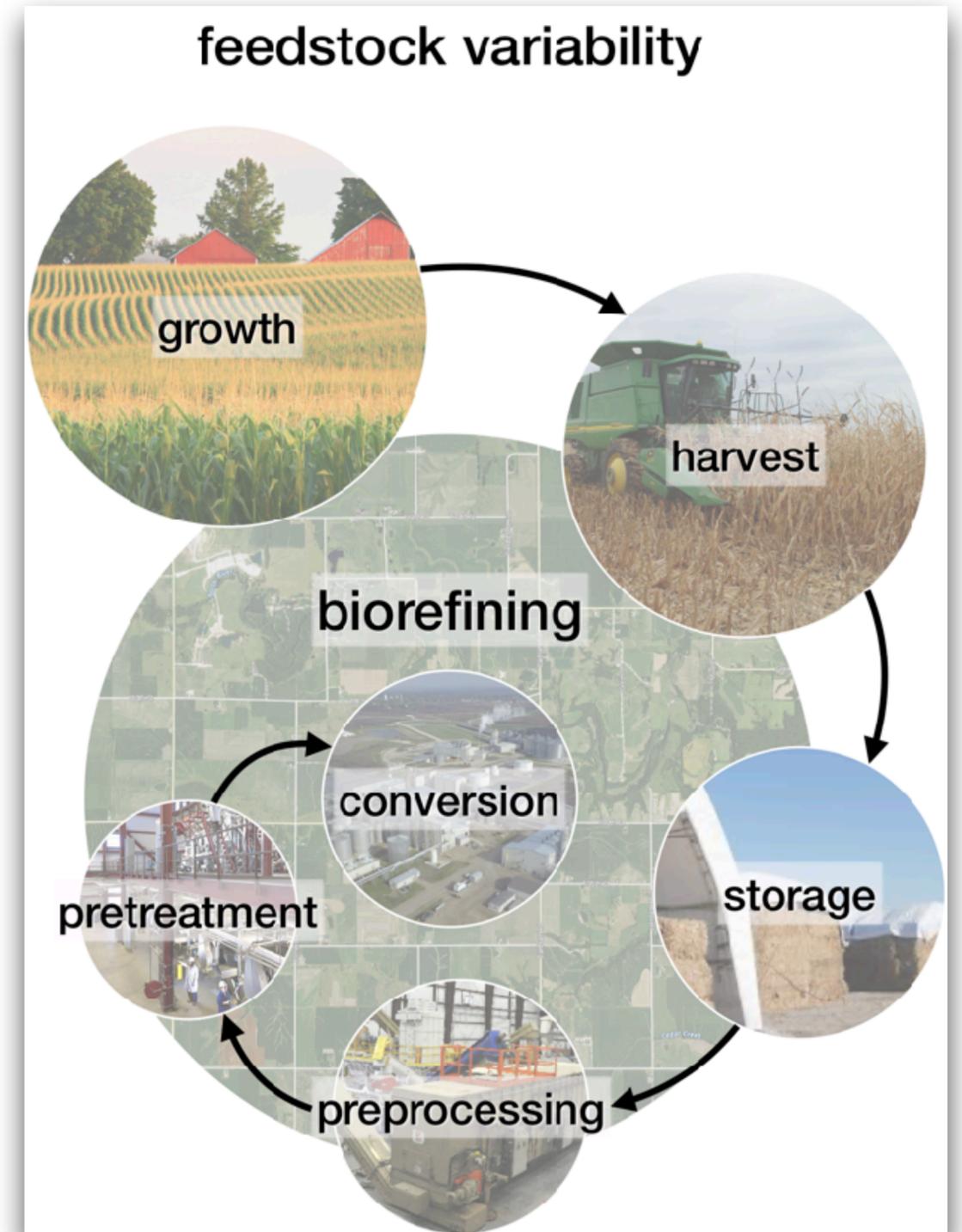
- How and at **what scale** should variability be measured?
- Focus on scales that we can affect for practical solutions
- Deep dive for fundamental understanding of variability



Variability originates from multiple sources

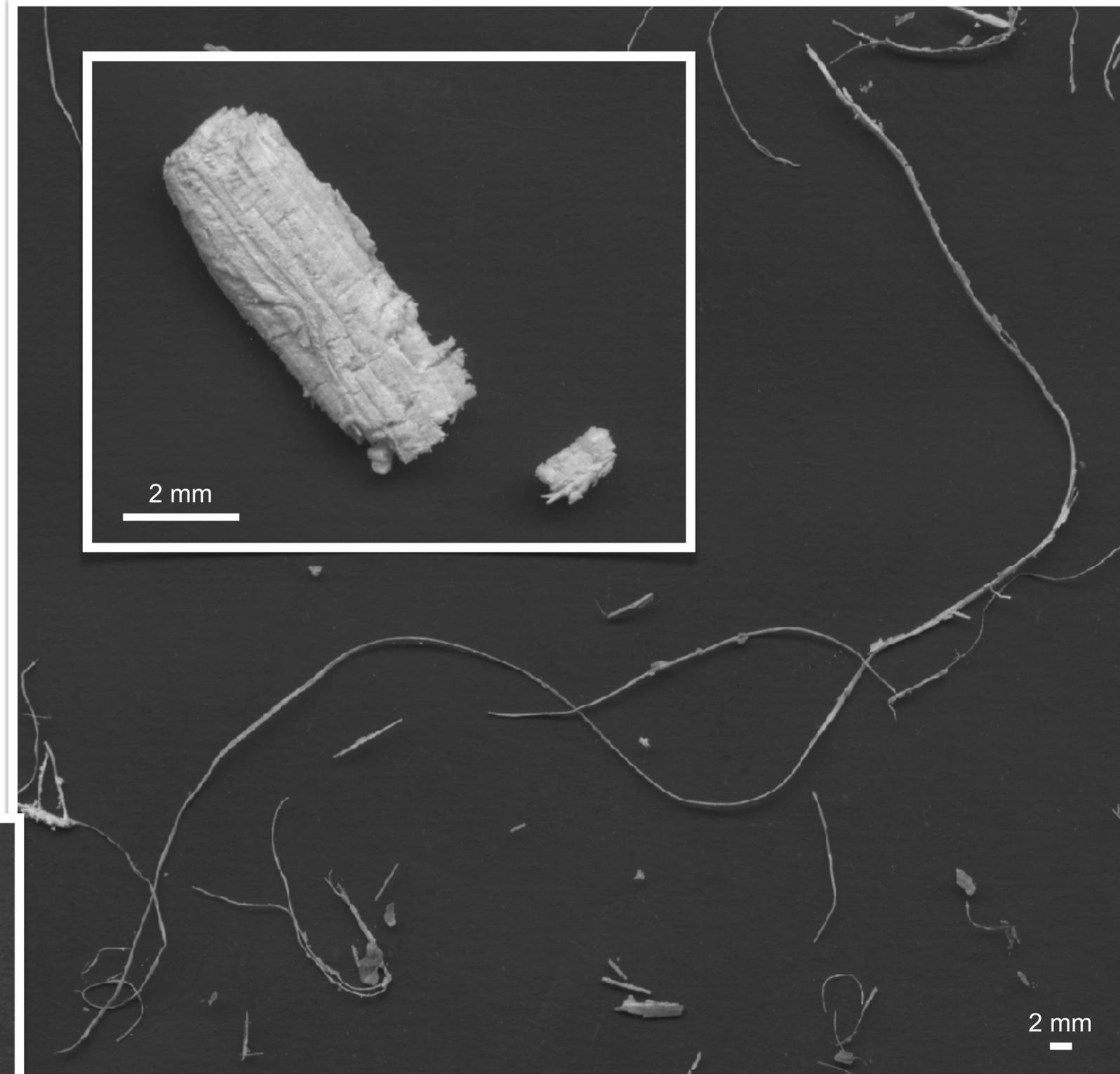
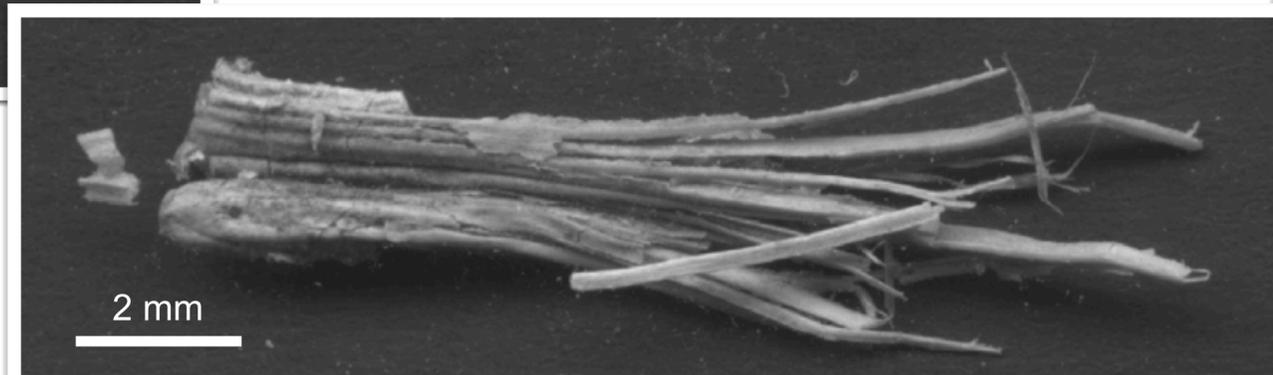
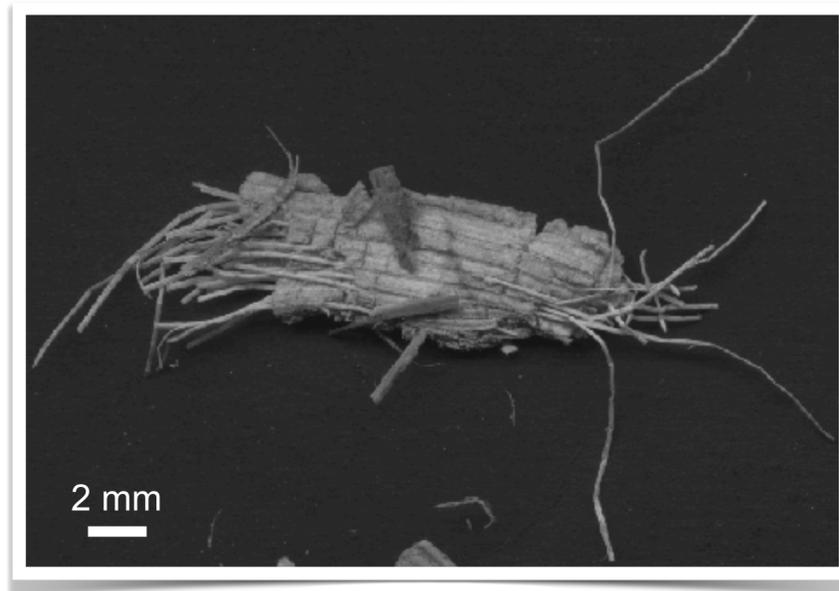
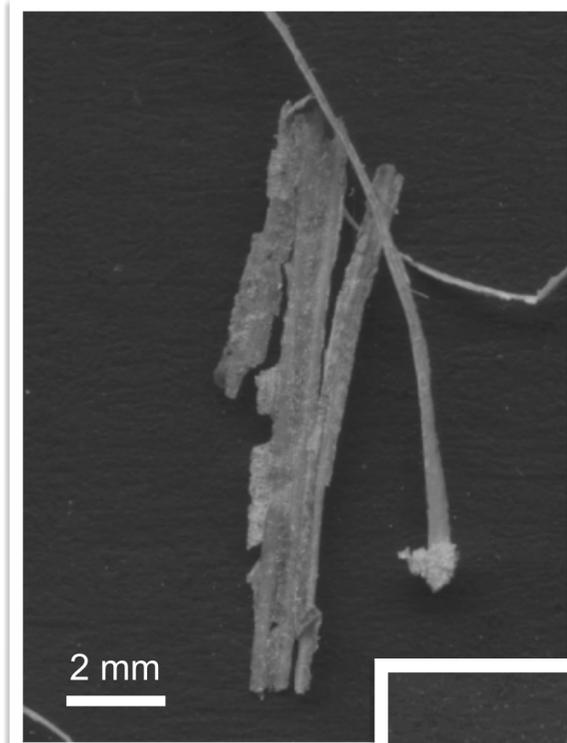
Hypothesis: better understanding, and management of variability will help biorefineries operate continuously and profitably.

- Where does **variability originate** and what impact does it have?
- What is the relative importance of **inherent vs. introduced** variability?
- What are the fundamental **material attributes** underlying variability?



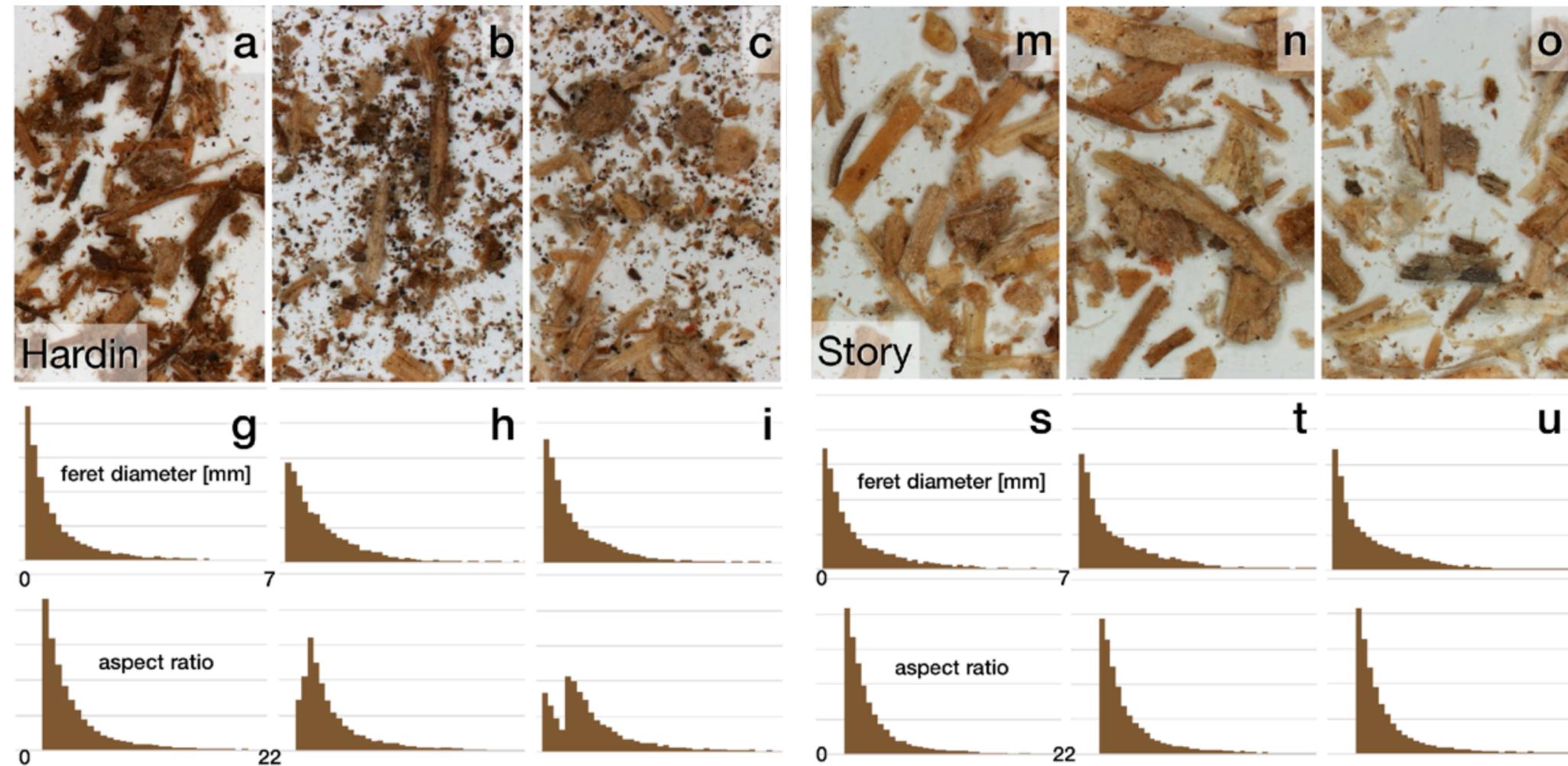
Variability in particle size and morphology

- particle size distribution is considered a critical attribute
- biomass particles are anisotropic
- biomass particles have highly irregular margins
- high aspect ratio particles originate from vascular tissues



High aspect ratio particles and fines lead to poor material flow

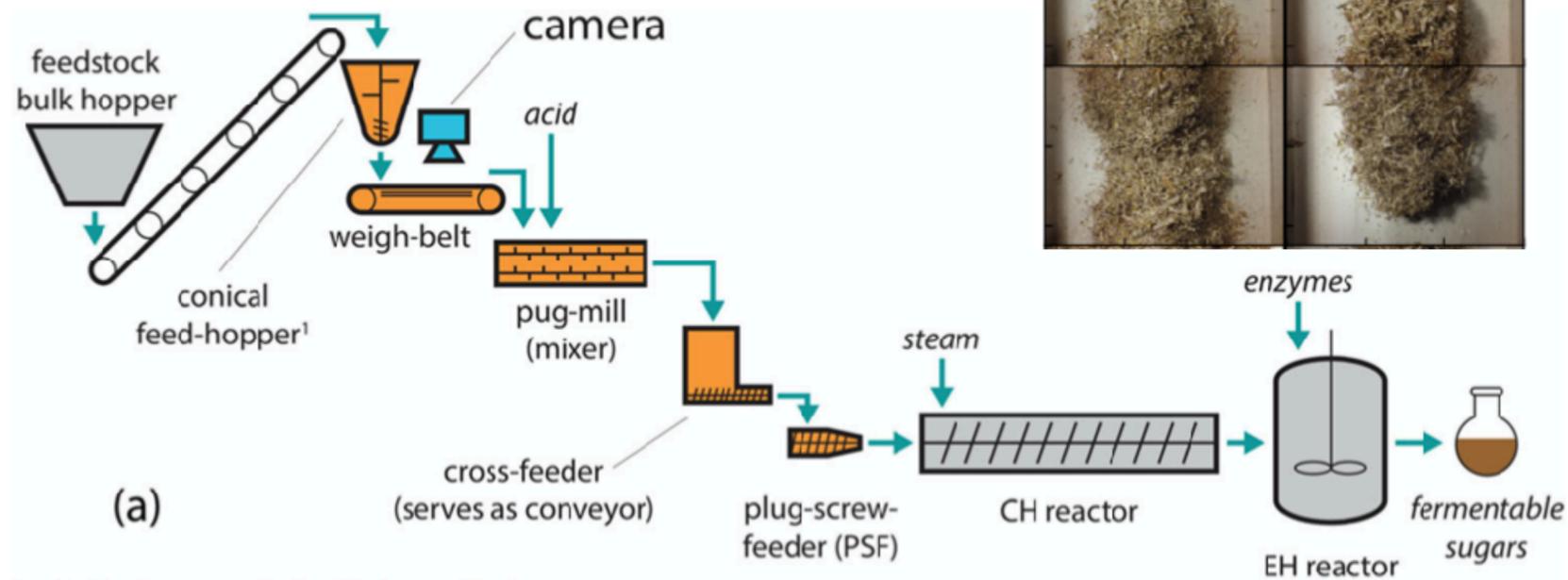
- stereoscope micrographs and particle morphology of milled, sieved corn stover particles sampled across multiple bales and bale sections
- samples collected in Hardin county displayed a greater distribution of variability in color, particle diameter, and aspect ratio among and within different bales
- samples collected from Story county displayed less variability
- extrinsic ash contributes to fines and can act as a grinding medium for particle size reduction



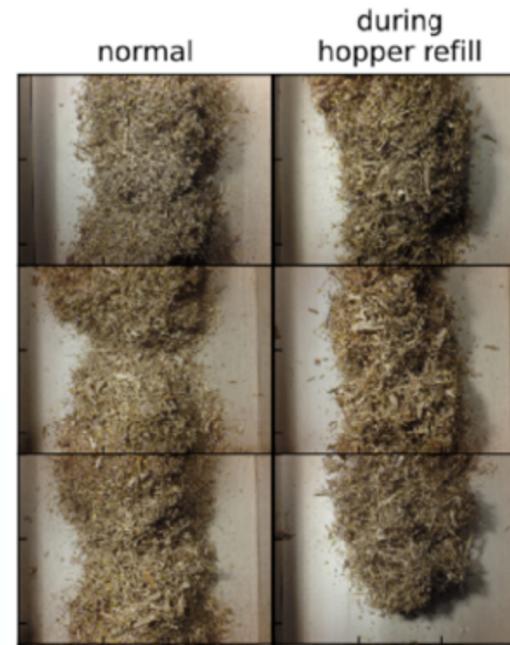
Ray, A. E. et al. (2020). *Multiscale Characterization of Lignocellulosic Biomass Variability and Its Implications to Preprocessing and Conversion: a Case Study for Corn Stover*. ACS Sustainable Chemistry & Engineering

Texture analysis as a proxy for individual particle analysis

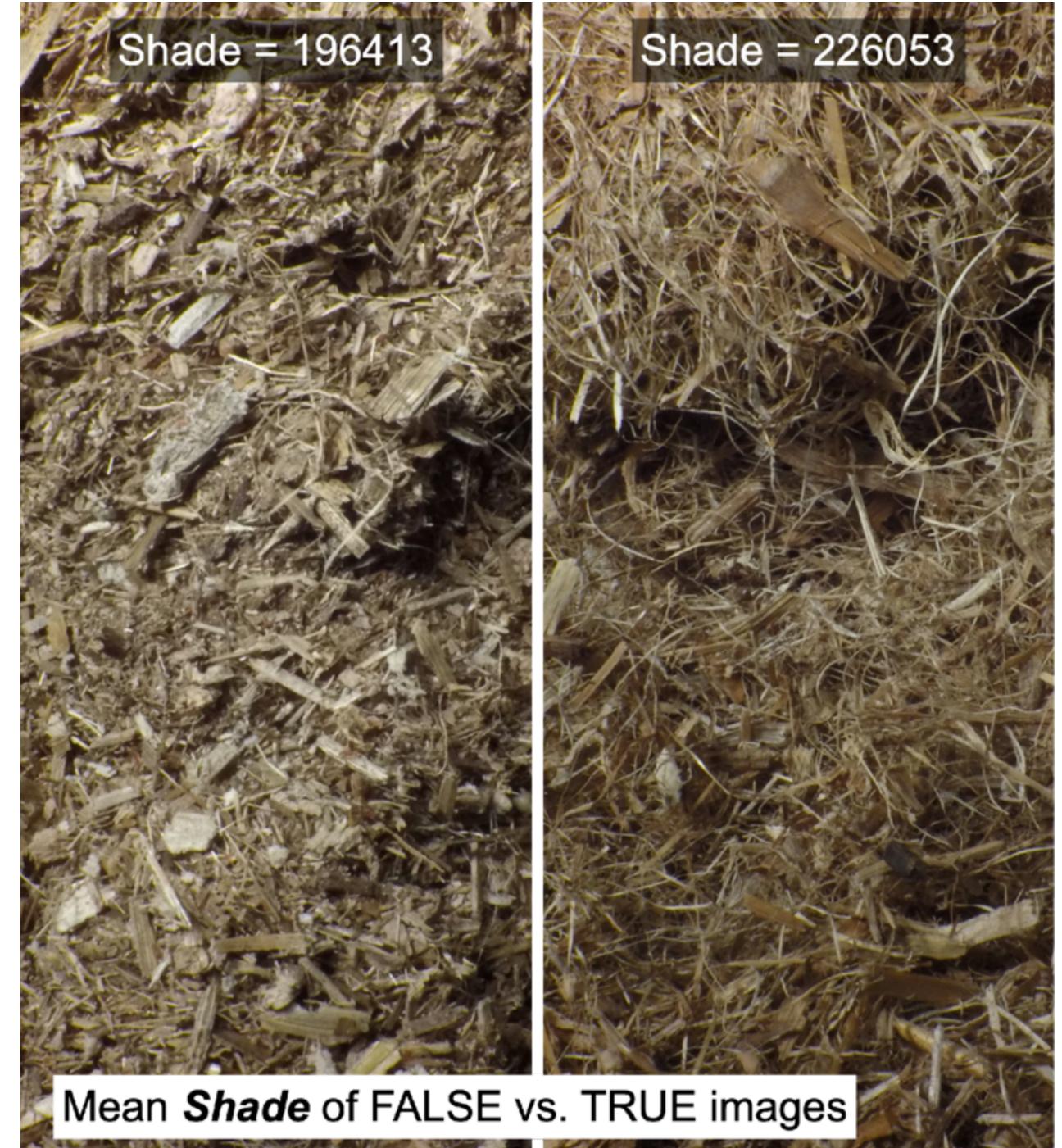
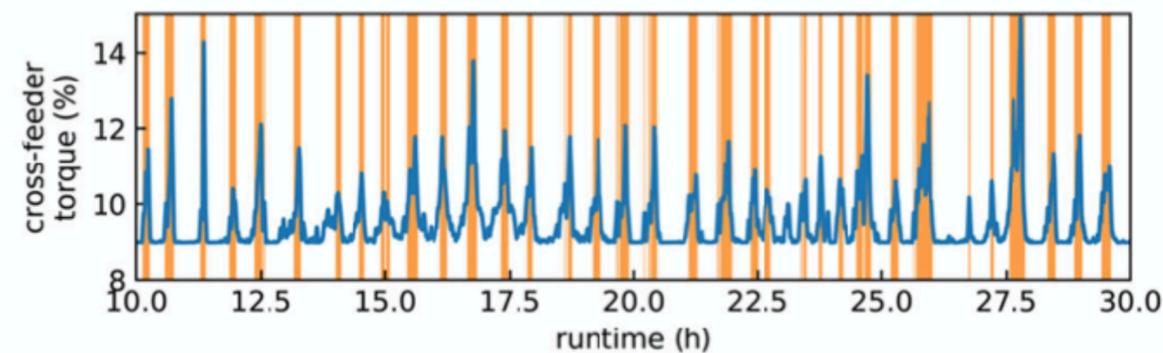
- particle information from piles and eventually from bales



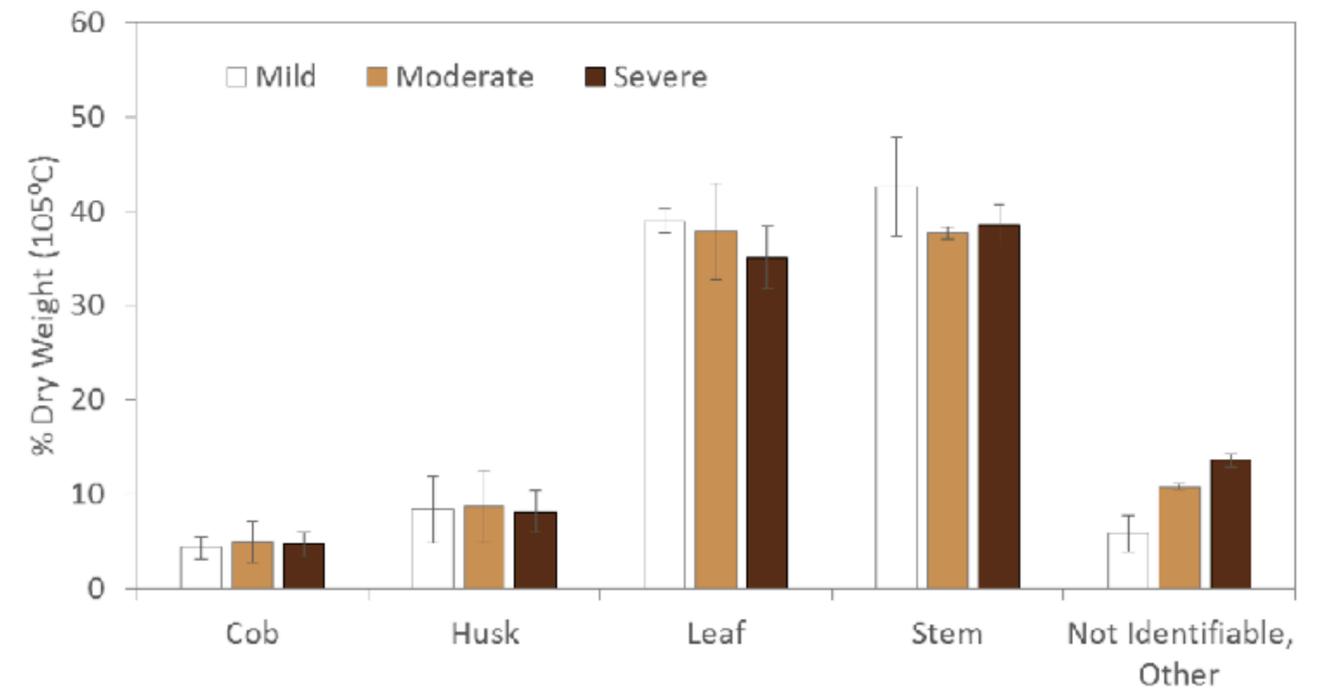
¹conical feed-hopper cyclically refilled every ~30 min



(a)



Biologically degraded bales



% dry weight of the primary anatomical fractions in the mild, moderate, and severely biologically degraded samples

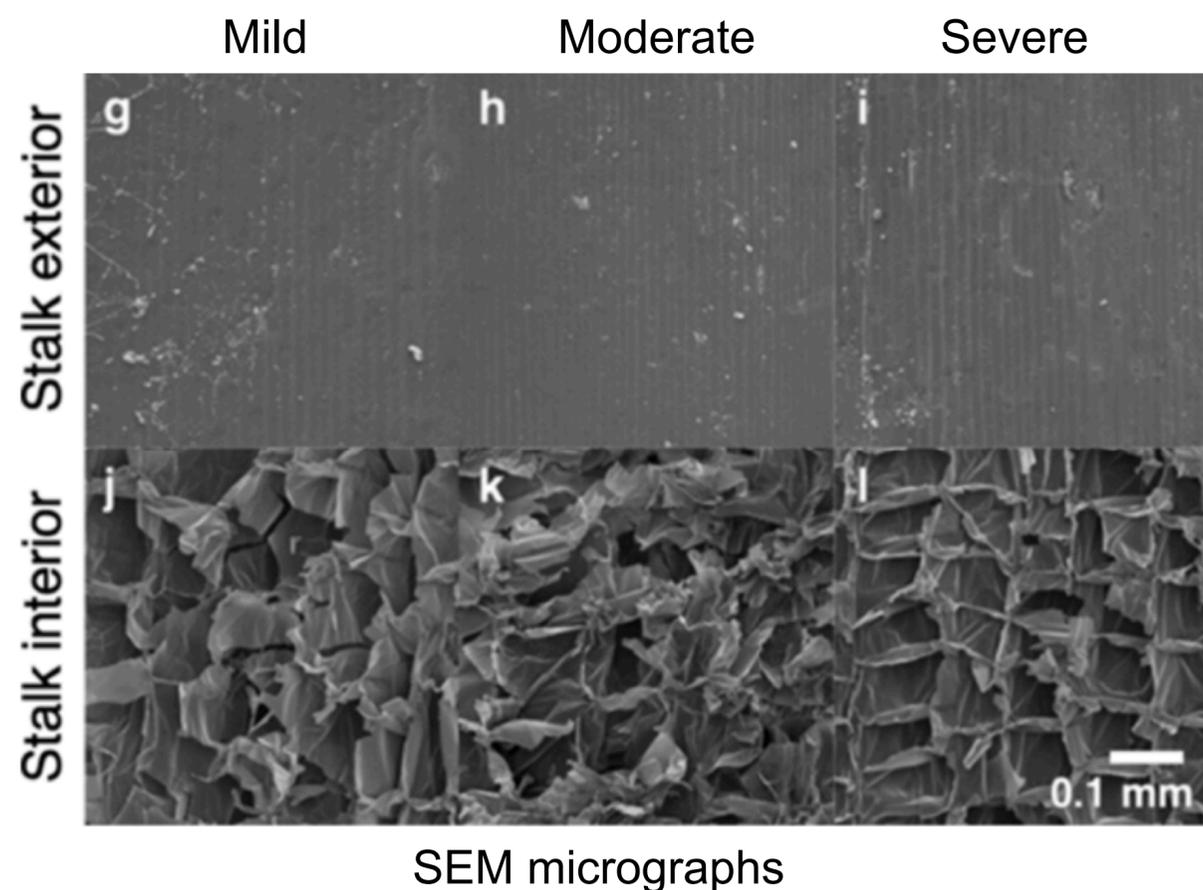
Photos from Idaho National Laboratory (INL)



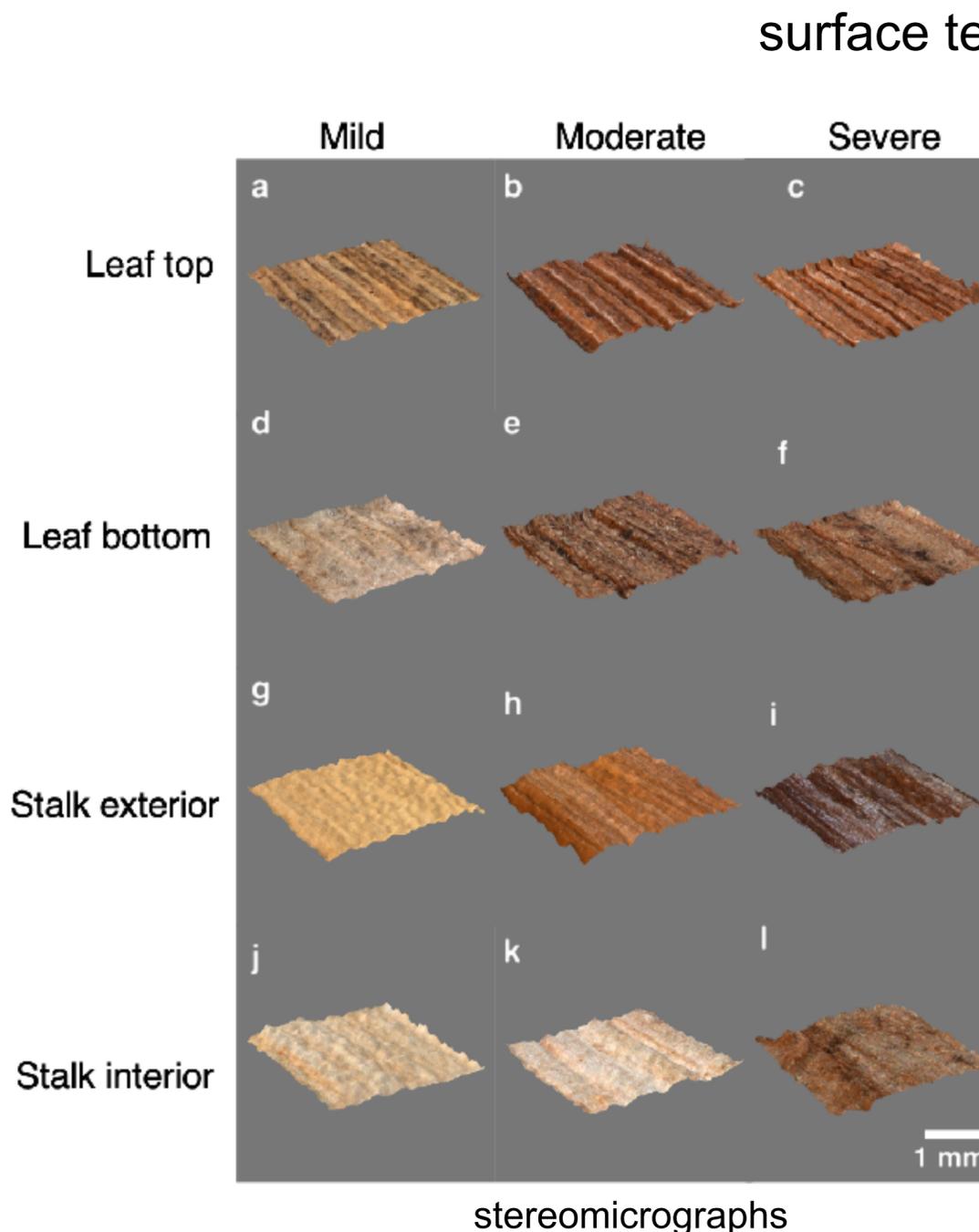
- Amber Hoover

Surface attributes at the mm and μm scale

- topographical surface texture was measured by image analysis to reveal trends with extent of biological degradation
- increased surface texture can cause higher inter-particle friction that results in poor feeding and flowability
- higher surface roughness is also correlated with hydrophobicity

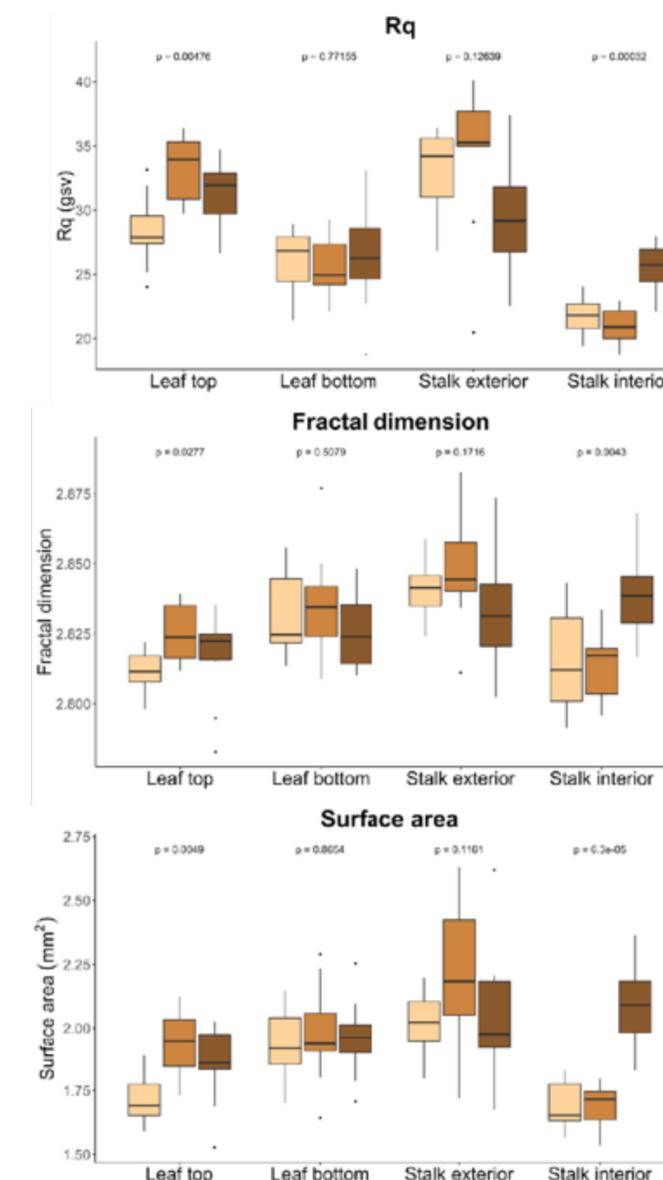


SEM micrographs



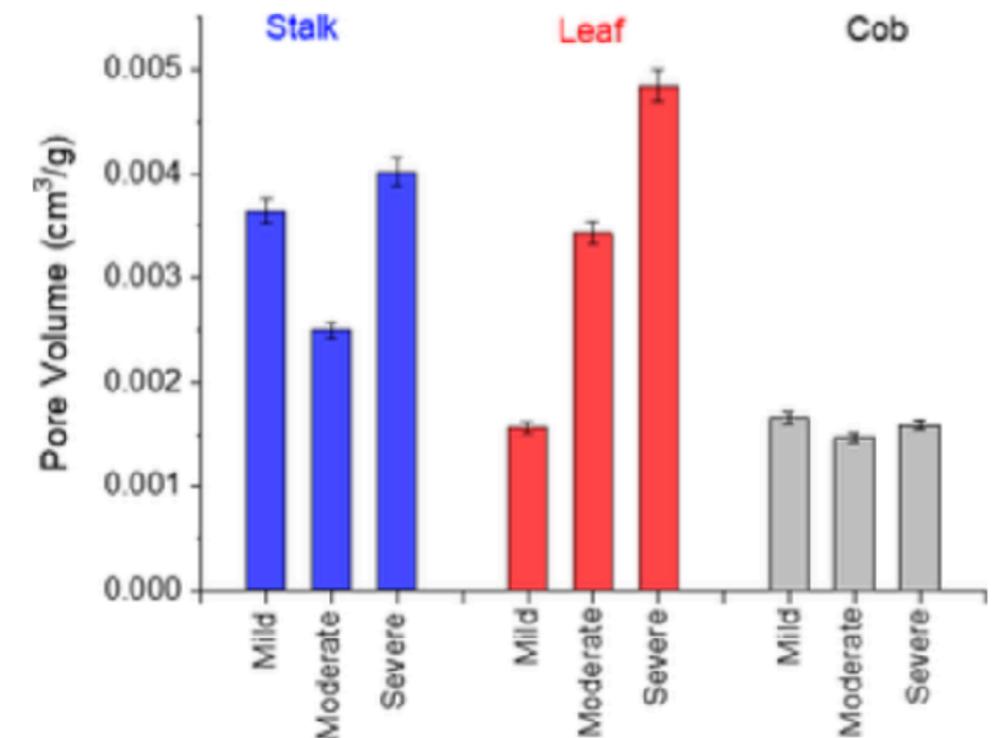
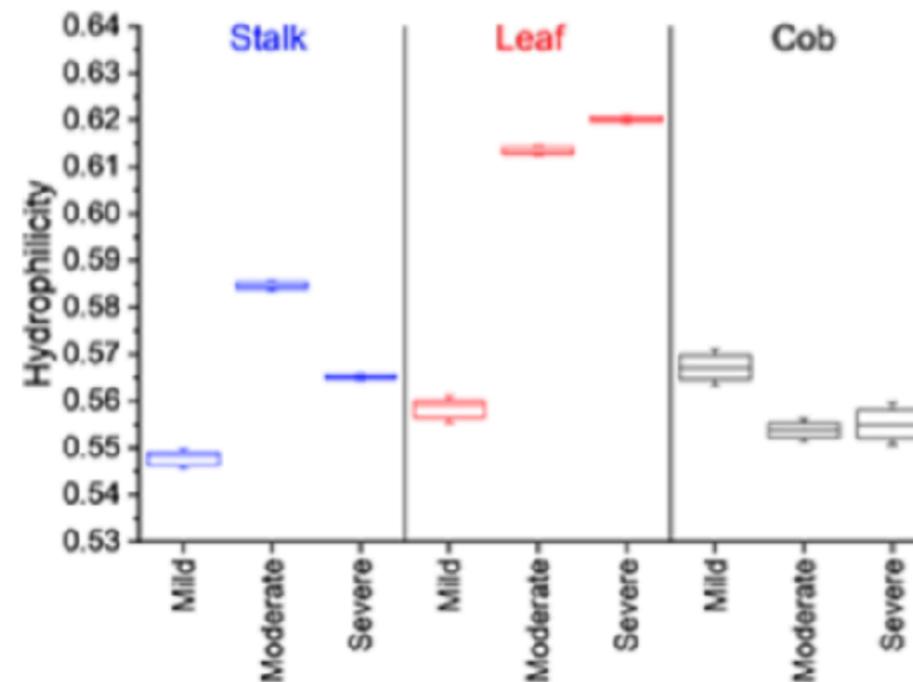
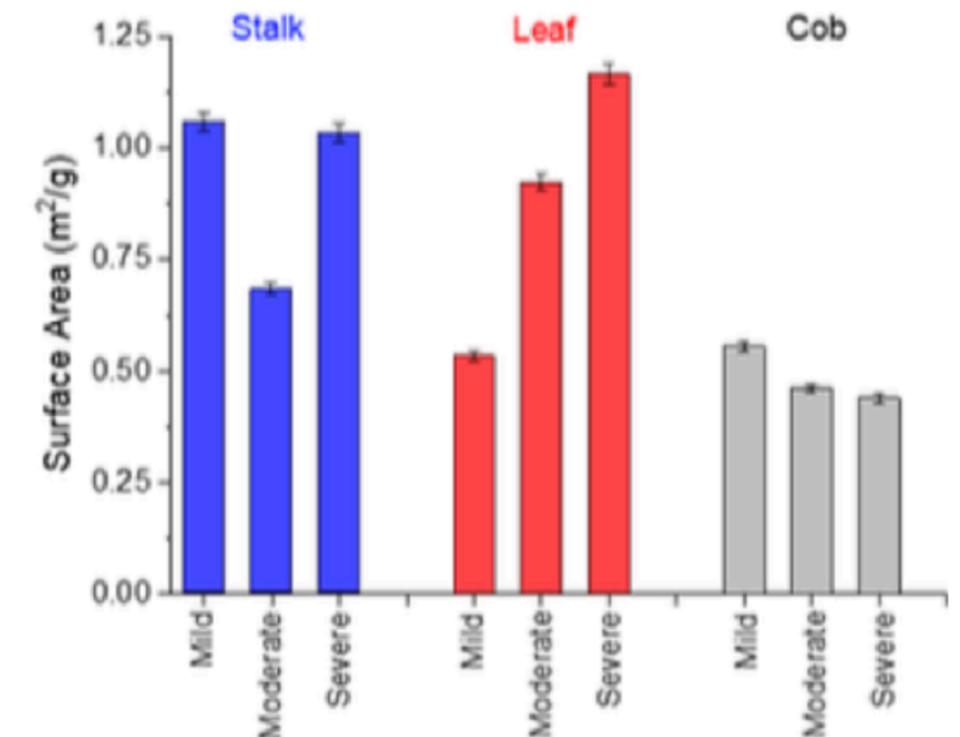
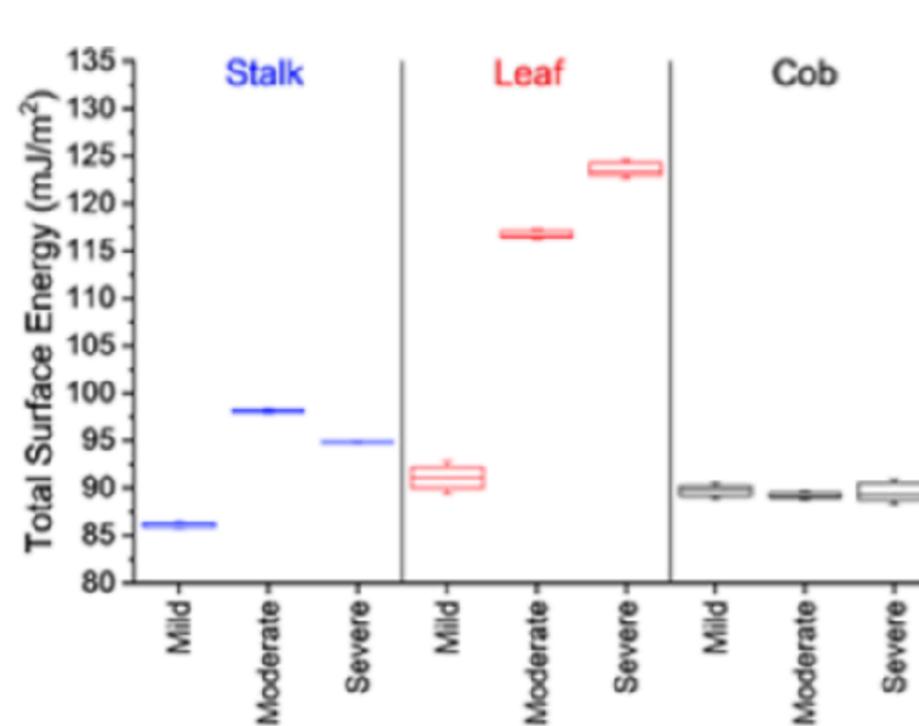
stereomicrographs

surface texture

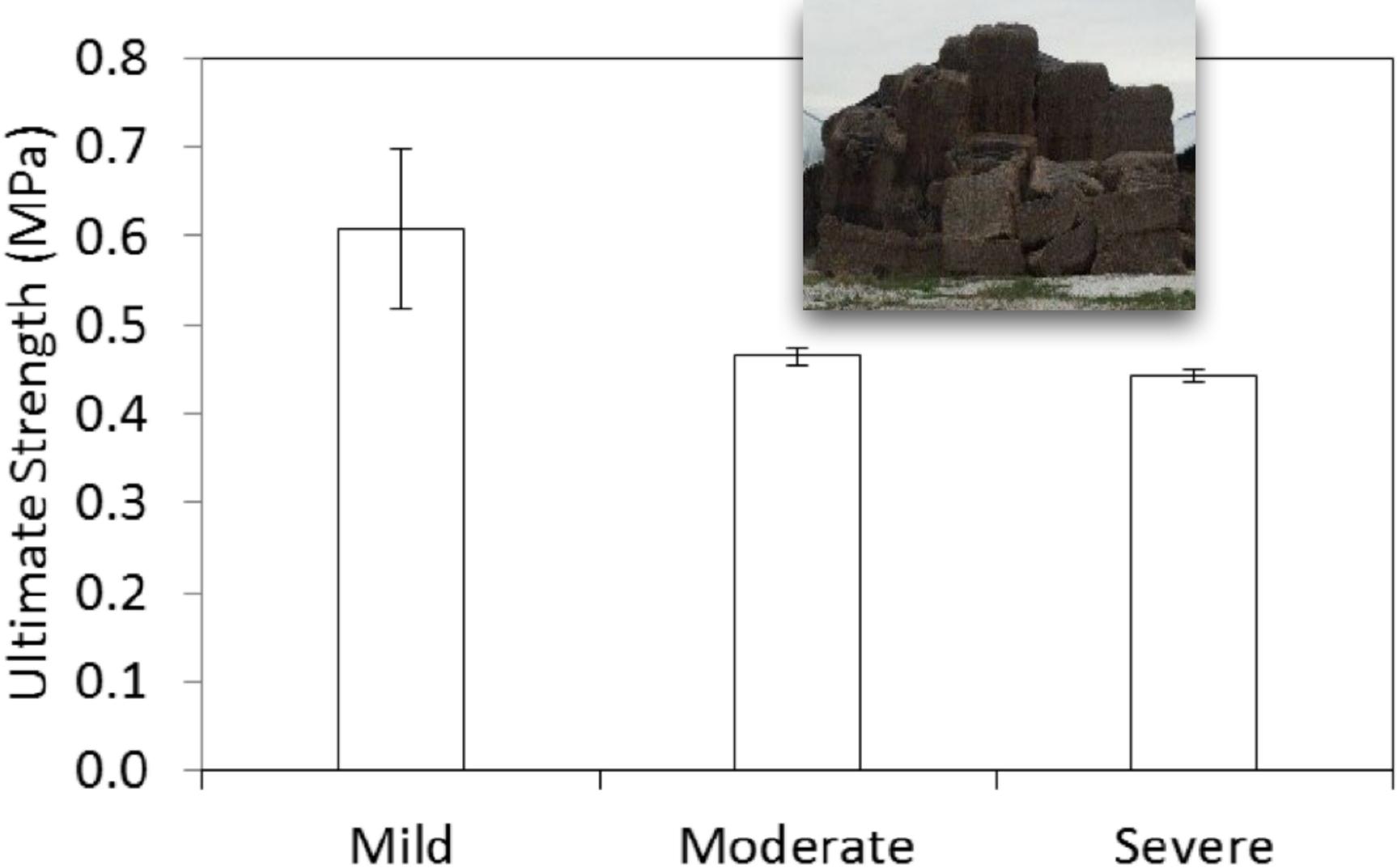


Impacts of biological self-heating on surface energy, surface area, hydrophilicity, and pore volume

- the **leaf fraction** was the **most sensitive** anatomical fraction to self-heating with large **increases** in surface energy (35%), surface area (118%), and pore volume (210%)
- the **cob fraction** was the **most resistant** to self-heating with very little changes in surface energy, pore volume and surface area
- changes in surface energy are directly related to the changes in the surface chemistry from biological self-heating



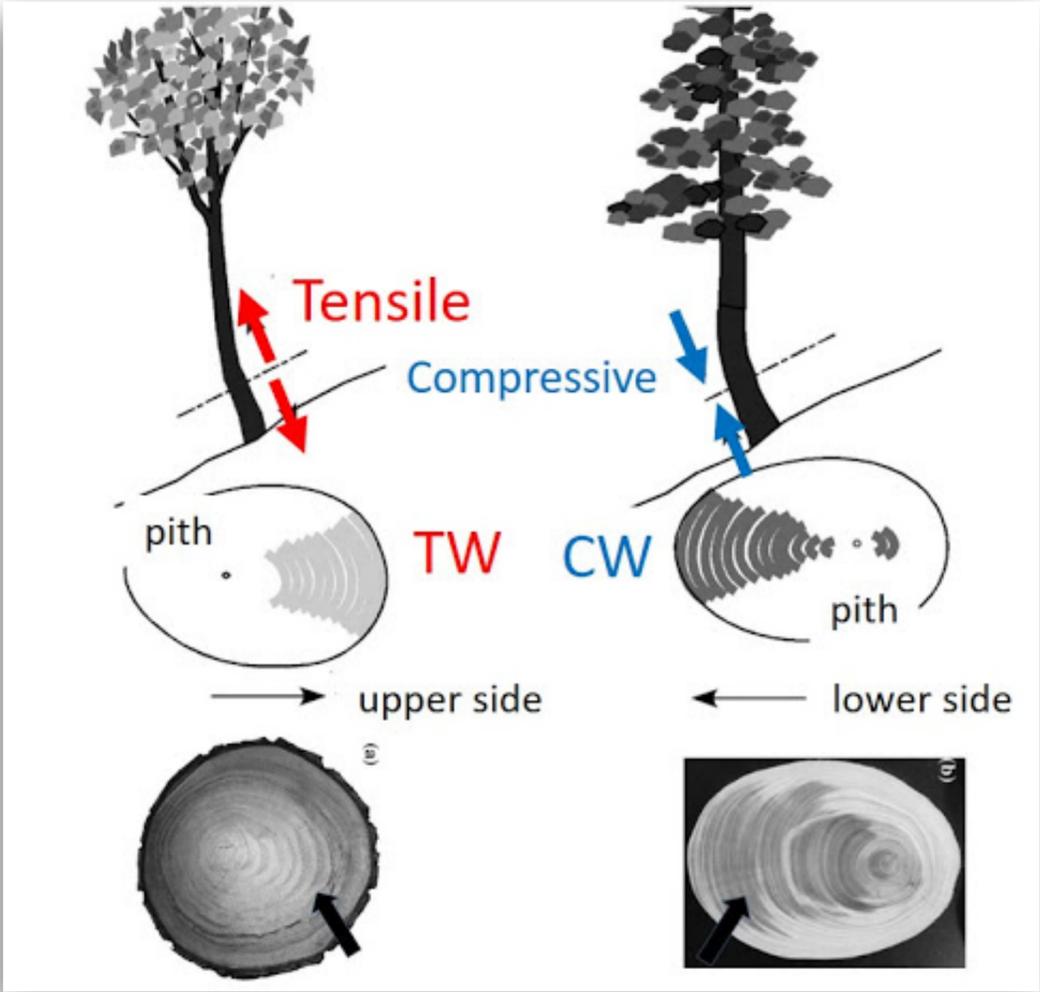
Highly degraded materials exhibited lower ultimate breaking strength



Ultimate breaking strength (MPa) of biologically degraded corn stover stalks

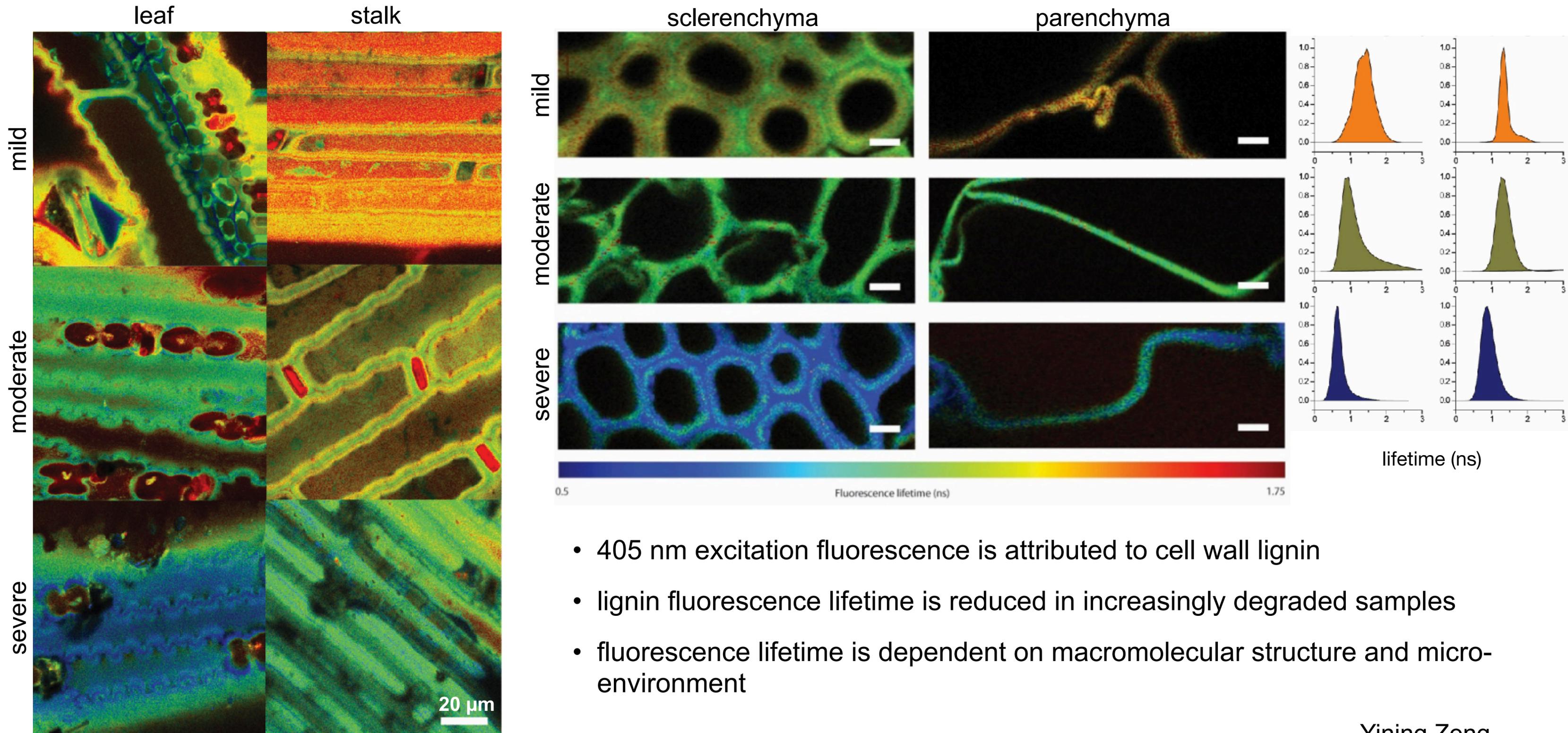
- the lignin content of all the samples is similar as ~17%
- if lignin structural modification is also taking place, the rearrangement could result in a less elastic material

- Amber Hoover



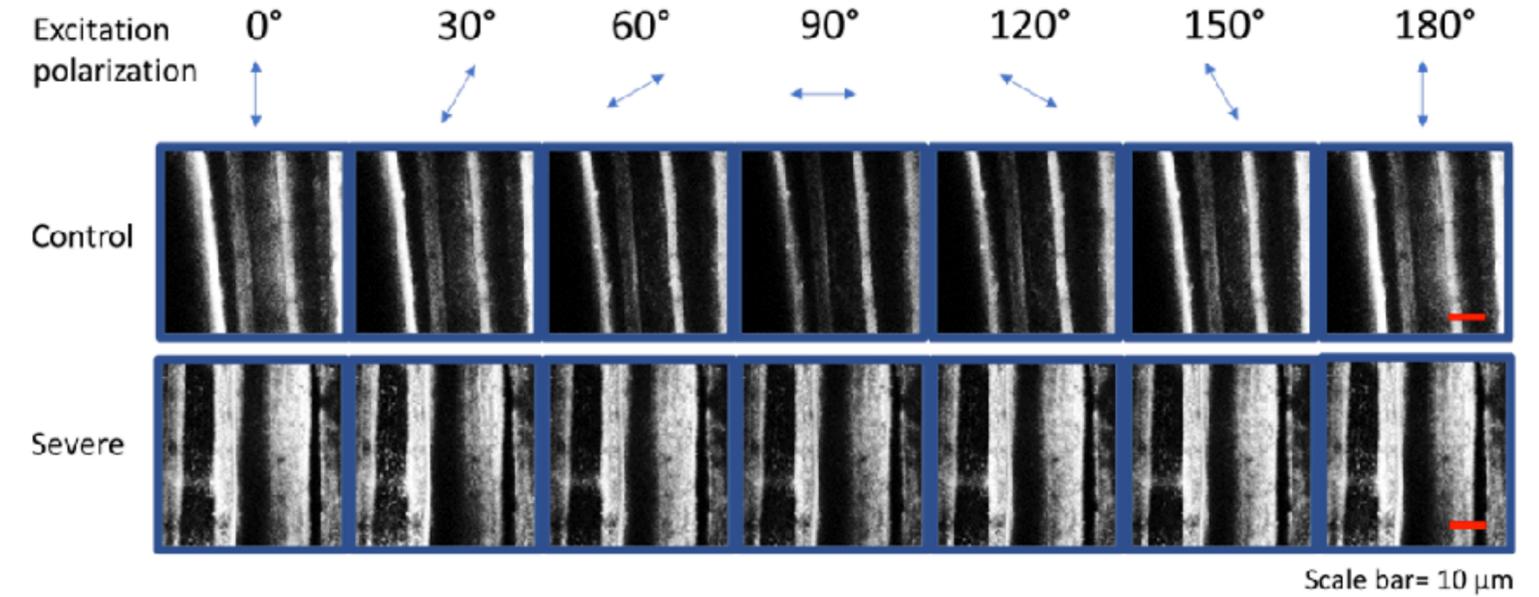
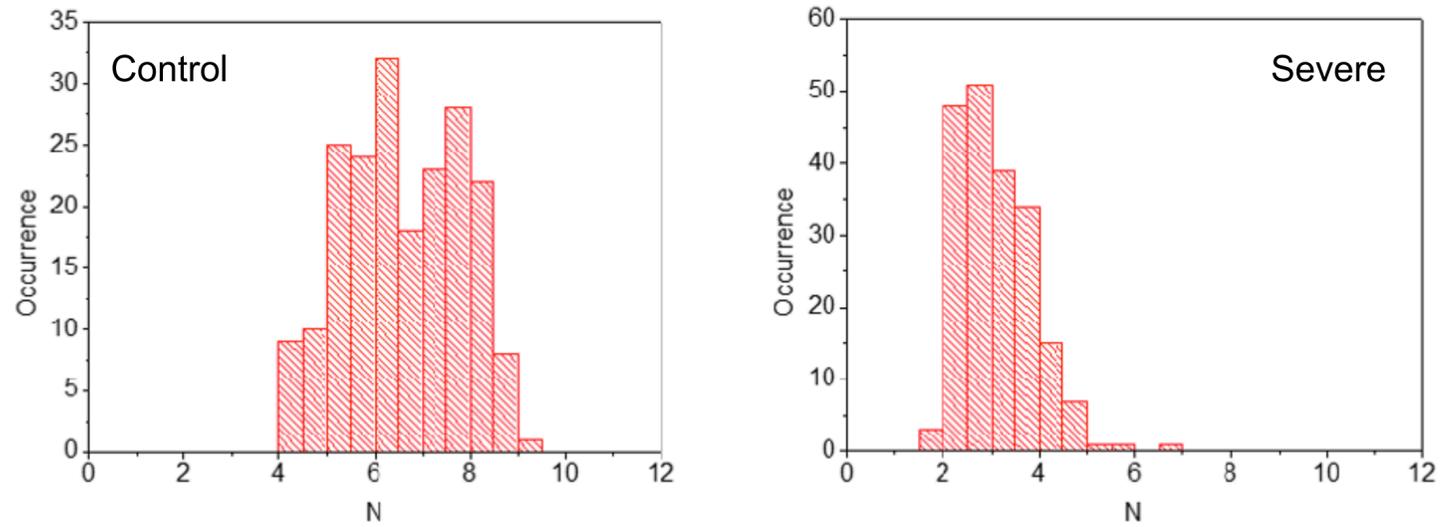
tensile forces resisted by cellulose and compressive forces resisted by lignin

Fluorescence lifetime imaging (FLIM) reveals changes in lignin structure or environment

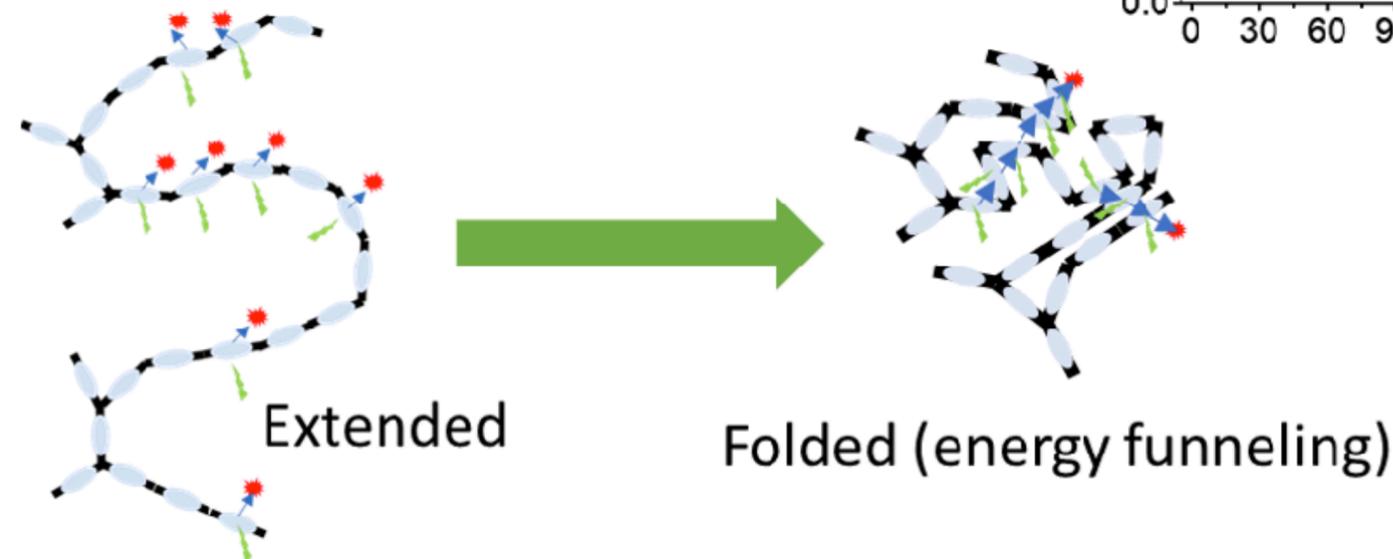
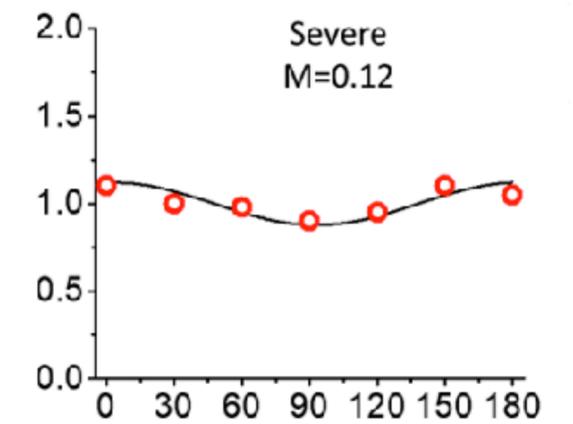
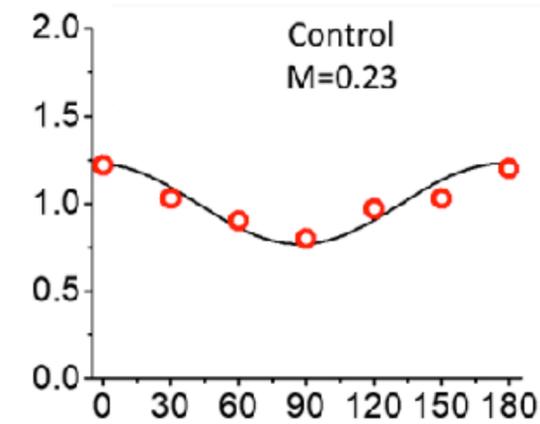


- 405 nm excitation fluorescence is attributed to cell wall lignin
- lignin fluorescence lifetime is reduced in increasingly degraded samples
- fluorescence lifetime is dependent on macromolecular structure and micro-environment

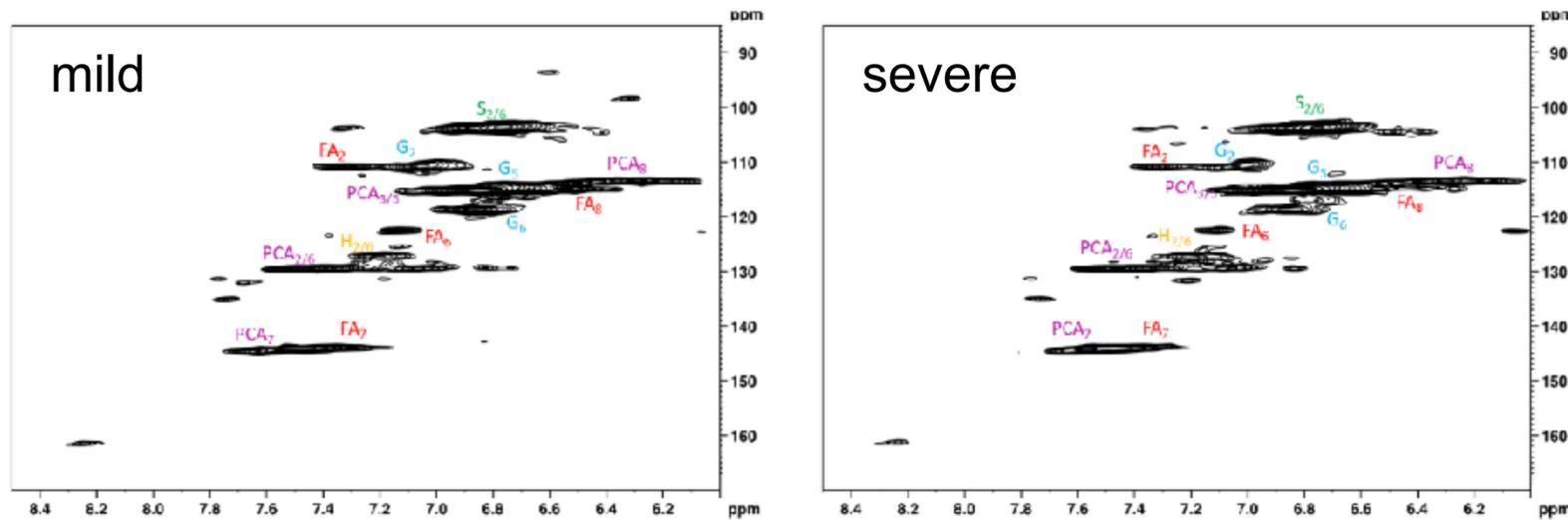
Reduction in fluorescence emitters and polarization



- number of fluorescent emitters is reduced by energy funneling
- fluorescence intensity modulation with excitation polarization
- self-heated samples show less modulation indicating less preferential orientation of lignin molecules

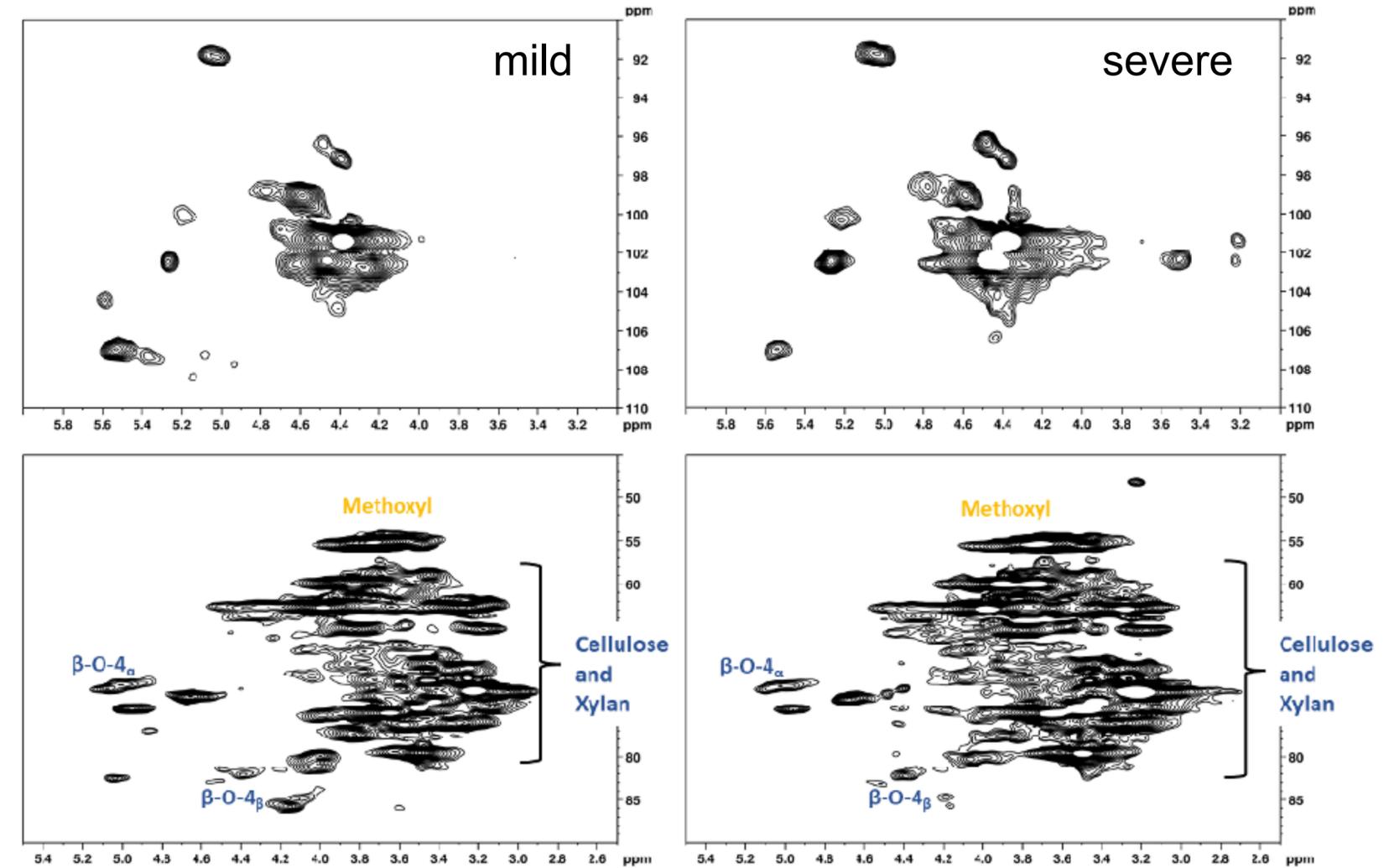


NMR confirms subtle changes in lignin structure



Aromatic Content

- S and H content do change
- **Coumarate, ferulate, and G content show some change**
- The severely degrade sample does contain some unknown peaks and peaks that disappear



Aliphatic Content

- Peaks have shifted, disappeared, or changed in intensity between the two samples.
- **β -O-4 content has dropped** in the severe sample.

Biologically degradation during storage impacts pretreatment and enzymatic hydrolysis

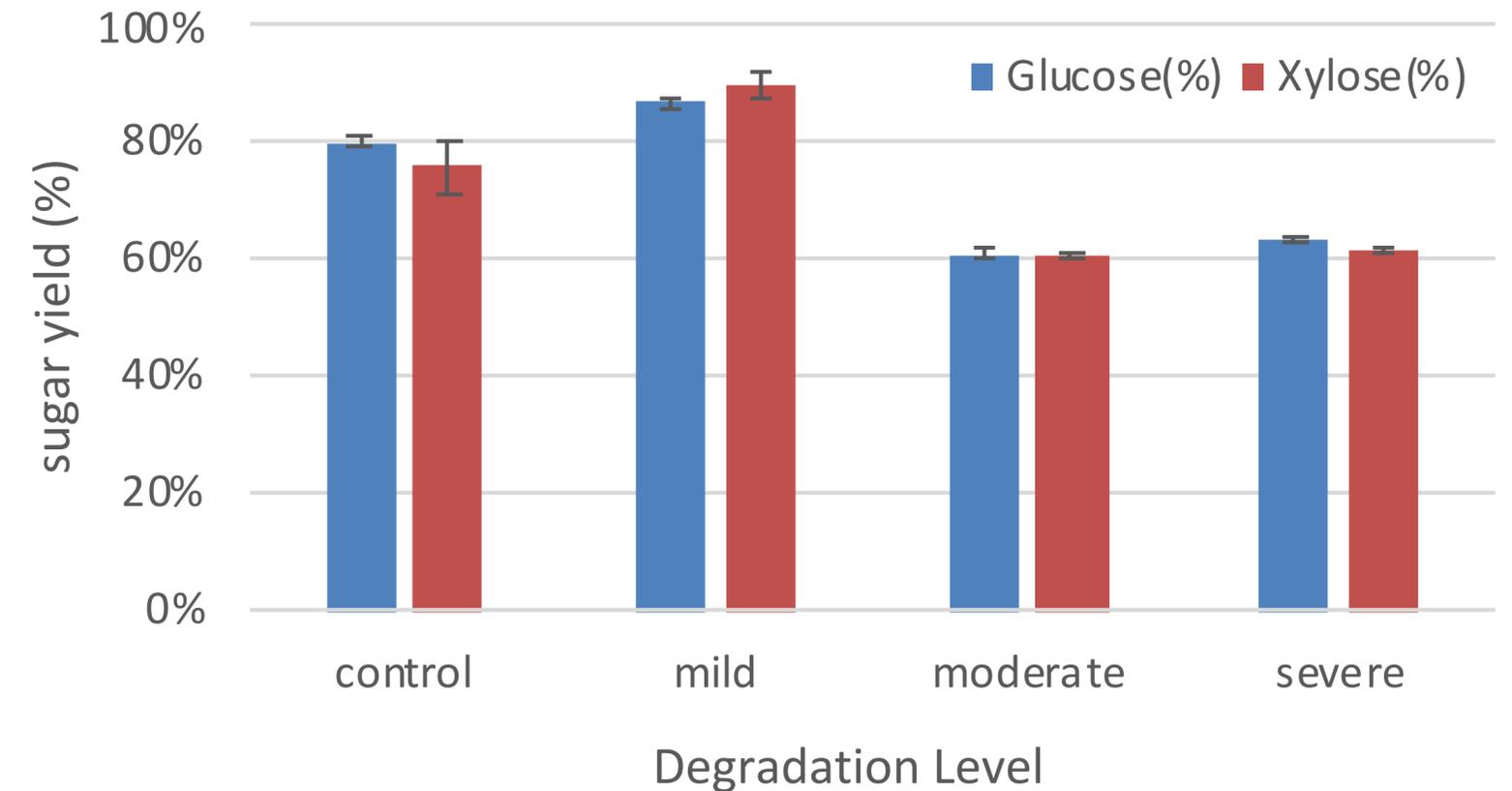
Black Liquor after DMR

Sample Description	Undiluted pH	Lignin (mg/ml)	Glucose (mg/ml)	Xylose (mg/ml)	Arabinose (mg/ml)	Lactic Acid (mg/ml)	Acetic Acid (mg/ml)
Control	10.52	2.65	0.66	2.49	1.22	0.34	3.54
Mild	11	2.32	0.72	3.69	1.81	0.42	3.55
Moderate	6.64	0.82	0.72	3.70	1.81	2.19	3.66
Severe	7.76	1.31	0.73	3.74	1.83	1.62	3.23

Feedstock after DMR

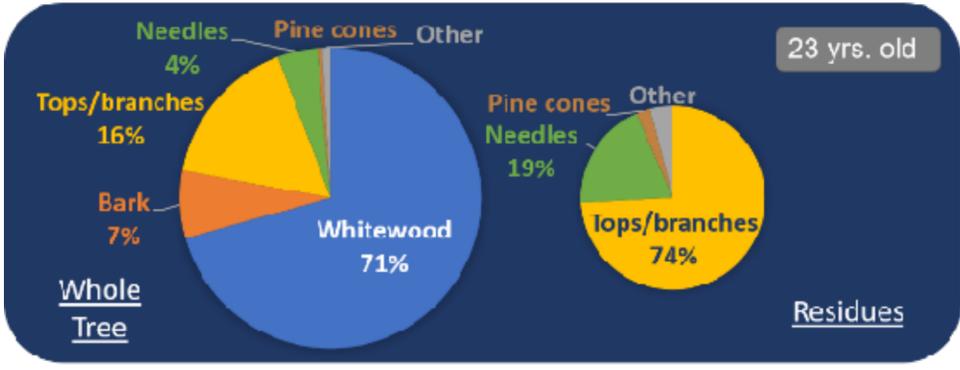
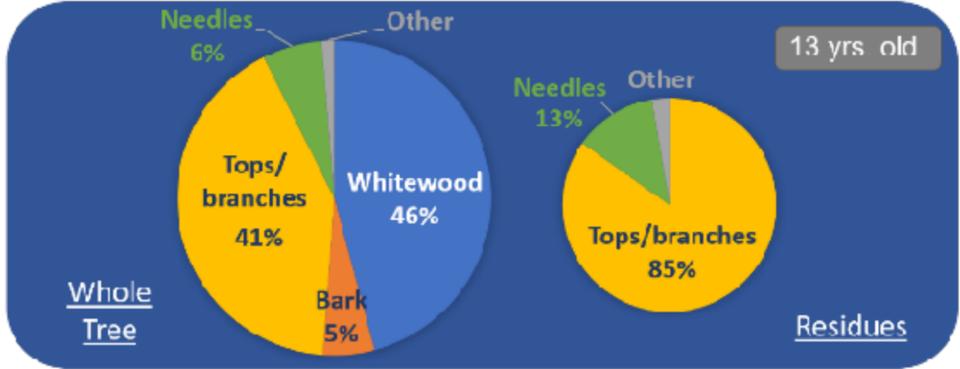
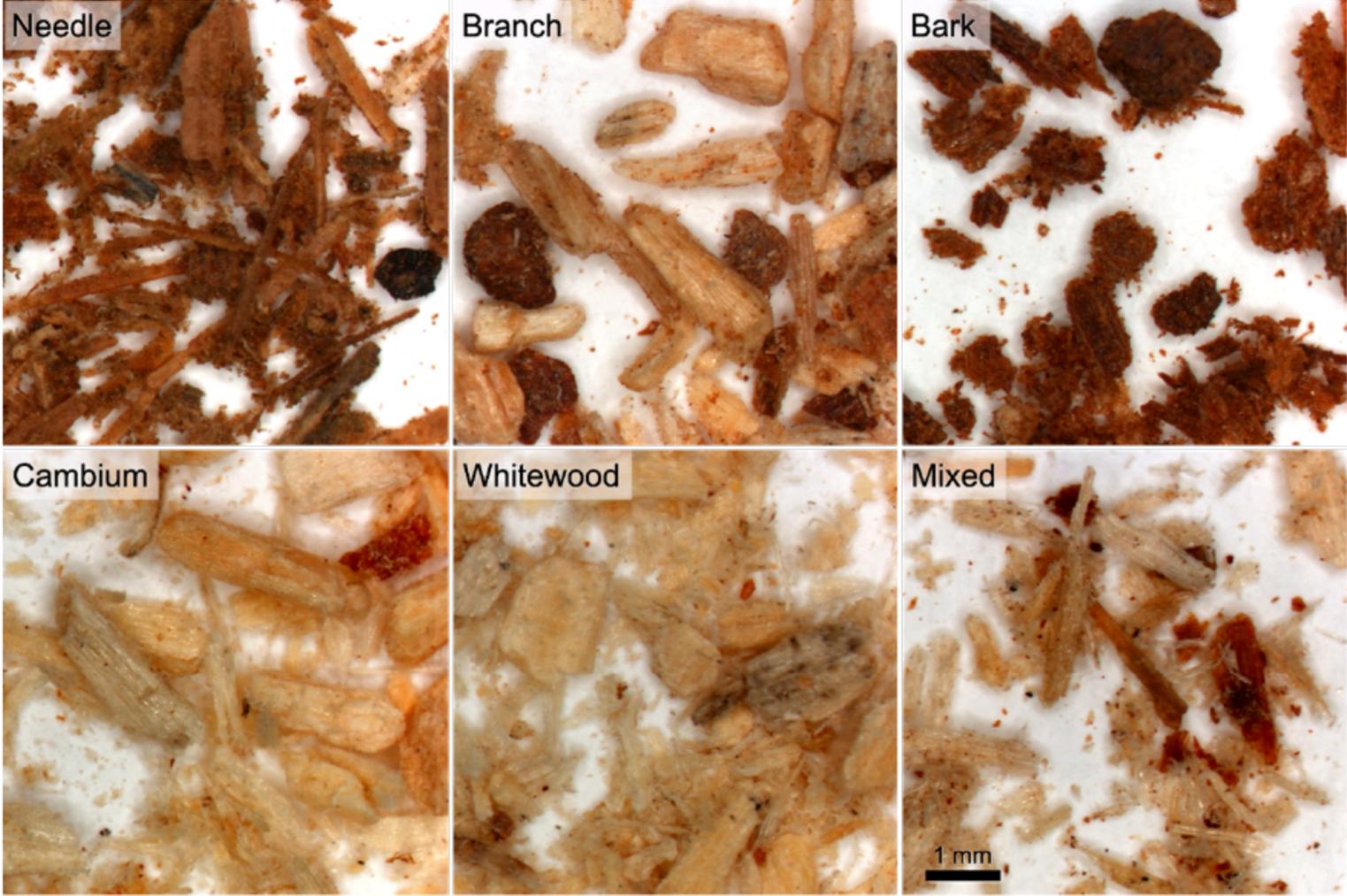
Sample Description	% Ash	% Lignin	% Glucan	% Xylan	% Galactan	% Arabinan	% Acetate	Total %
Control	5.03	13.65	52.49	22.17	1.14	2.43	0.19	97.11
Mild	2.83	11.21	54.21	23.33	1.04	2.62	0.15	95.39
Moderate	2.74	19.14	51.07	20.43	0.76	1.60	0.36	96.11
Severe	2.80	22.04	52.23	17.47	0.68	0.93	0.35	96.49

Sugar Yields after DMR and EH



- less lignin is released by DMR of degraded samples
- more acetate is left behind in DMR pretreated degraded samples
- glucose and xylose yields from enzymatic hydrolysis of degraded samples were 20-30% lower

What unique attributes do the bark and needle fractions in forestry residues contribute to yields and reliability?

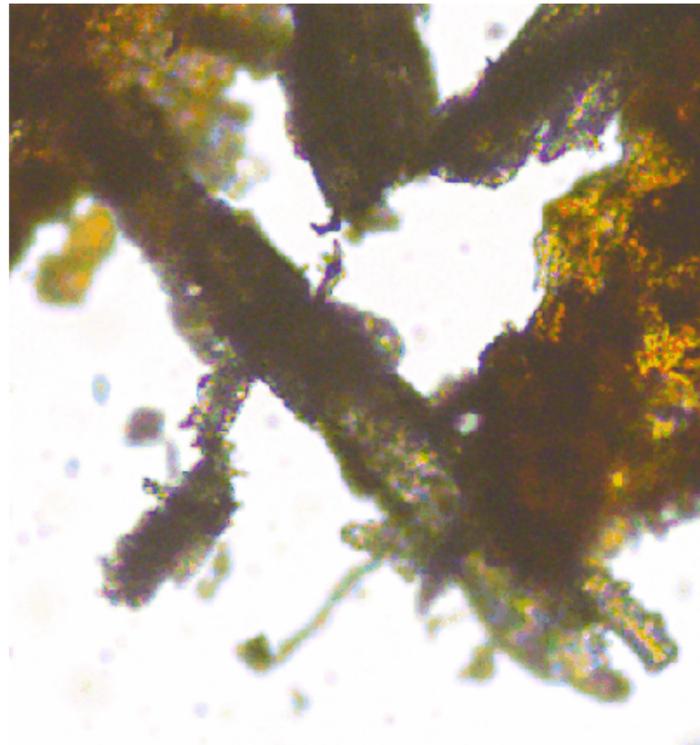


- How does tree age at harvest impact the proportion and attributes of pine tissue types?
- Focus on how different tissues impact the feed auger for catalytic fast pyrolysis.

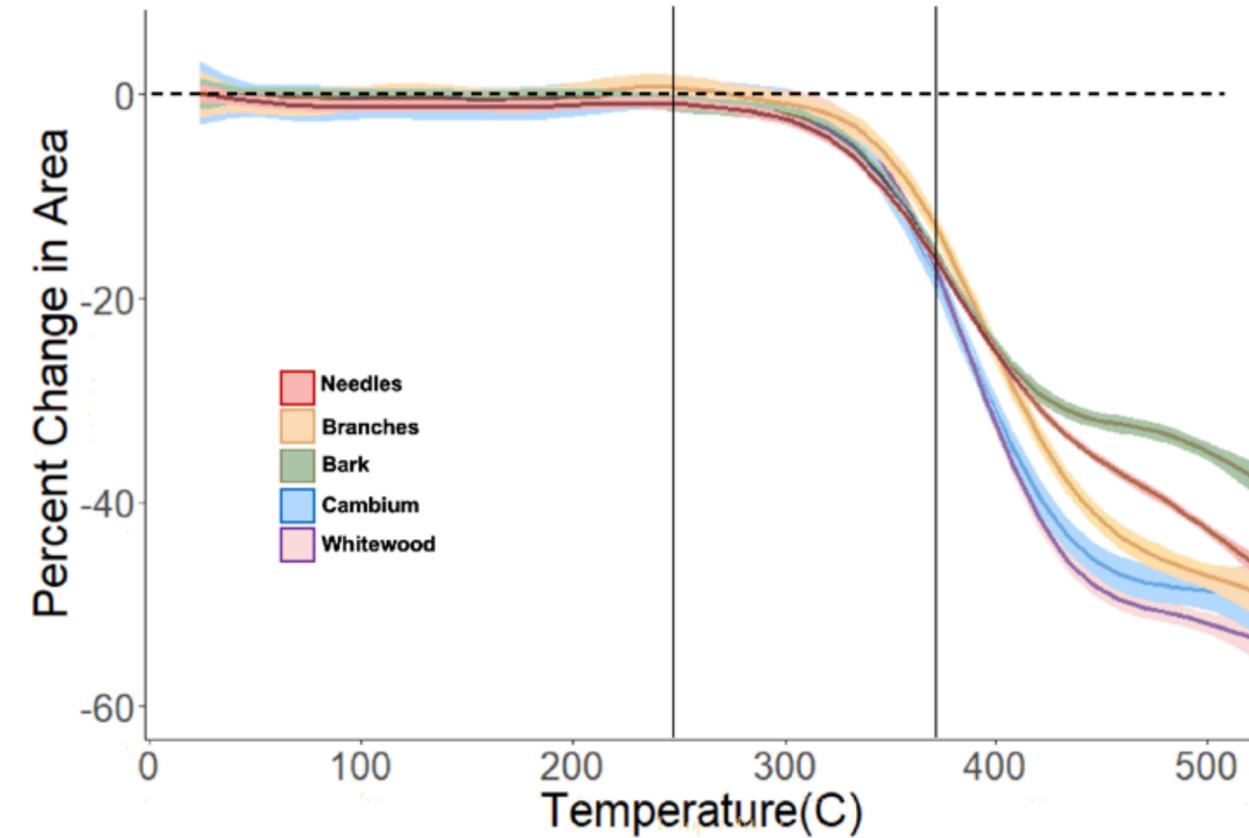
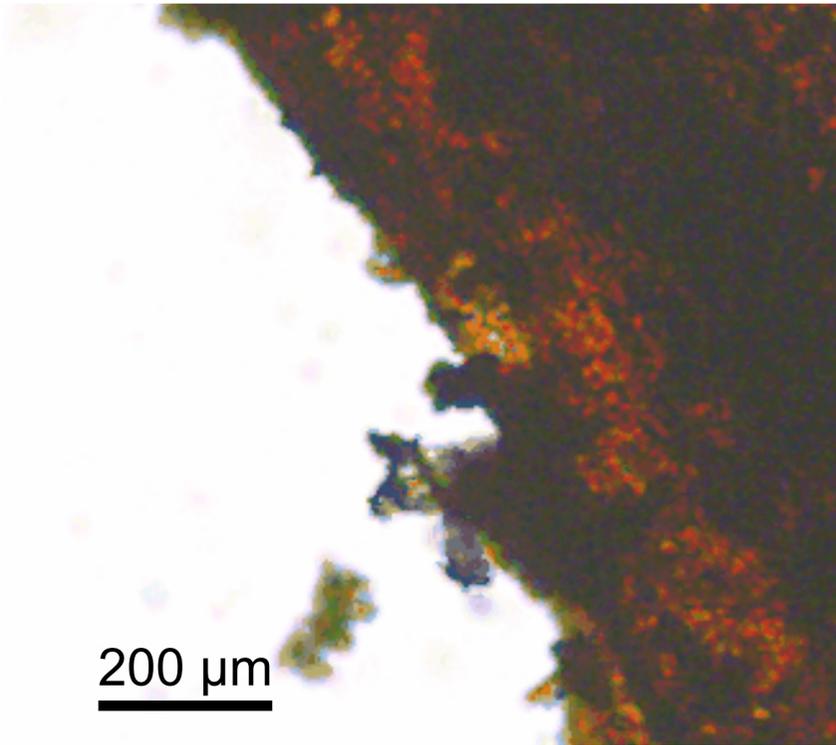


Pine particles display dynamic surfaces and volume change during heated in-situ microscopy

Needle



Bark



Needle

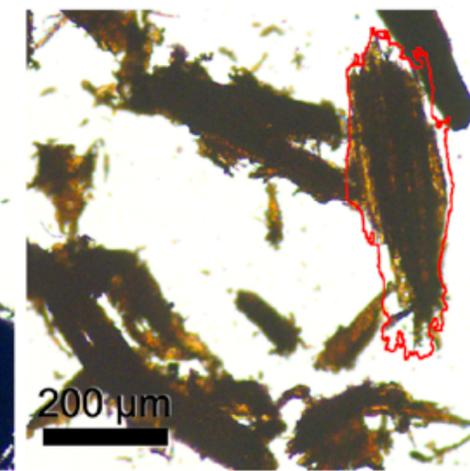
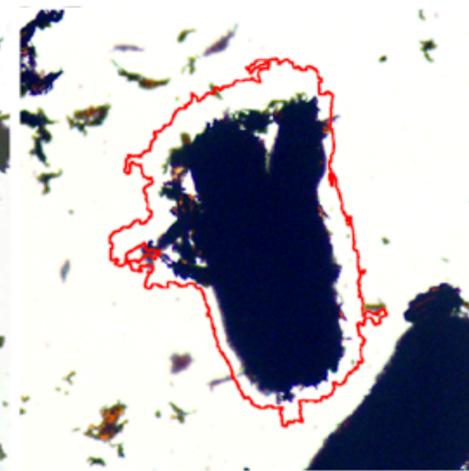
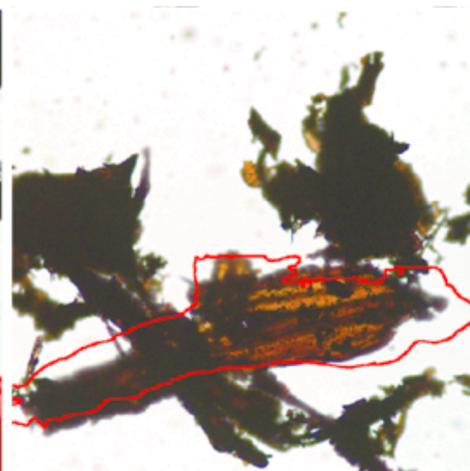
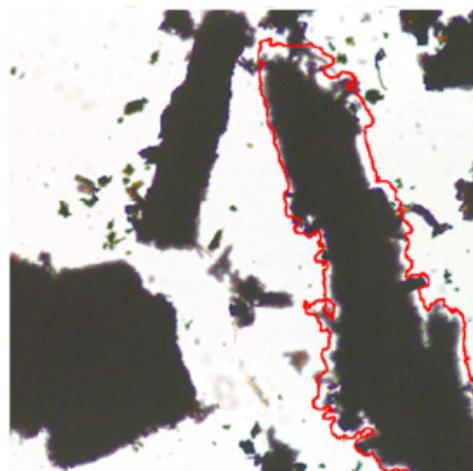
Branch

Bark

Cambium

Whitewood

375°C

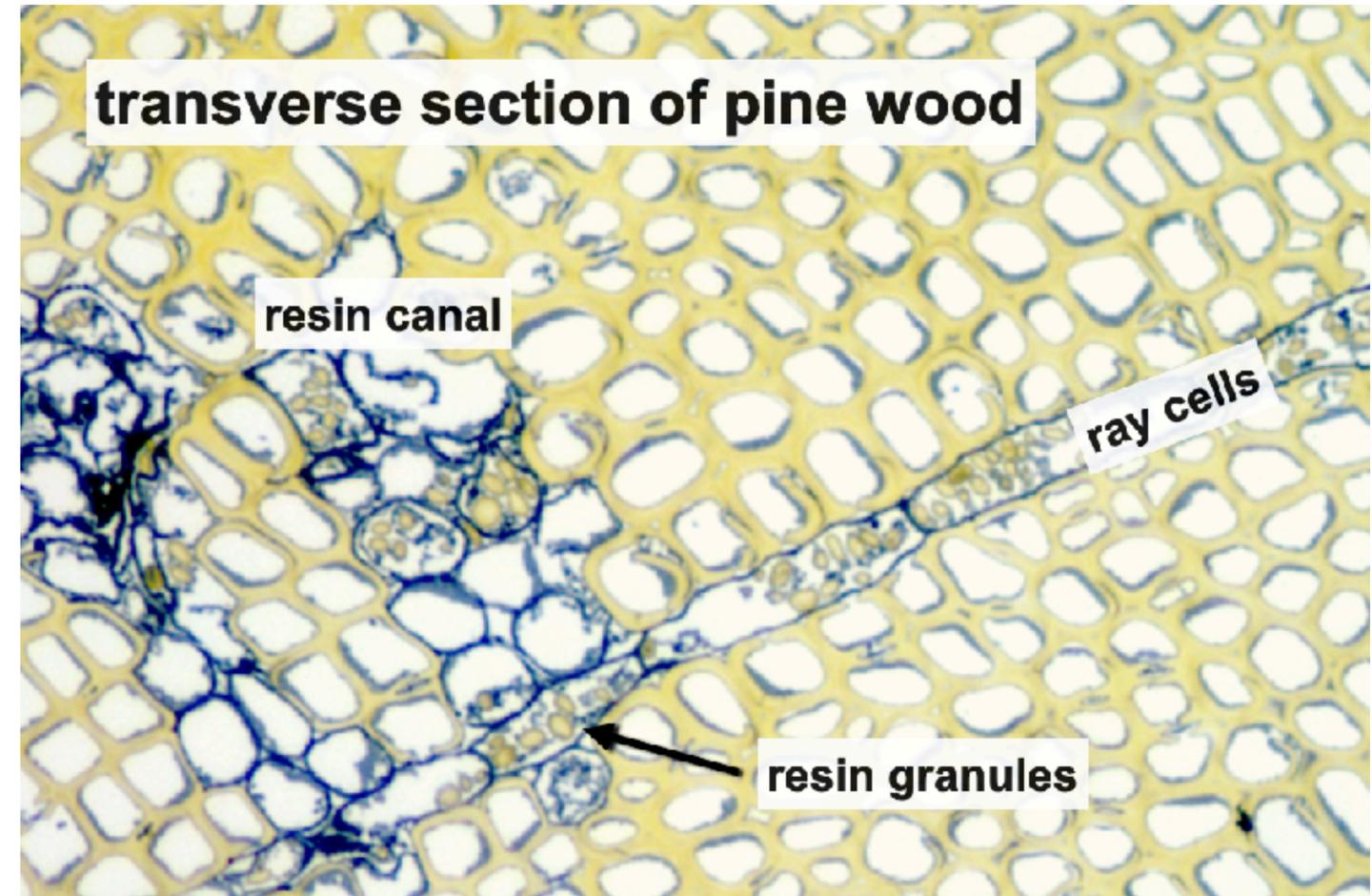
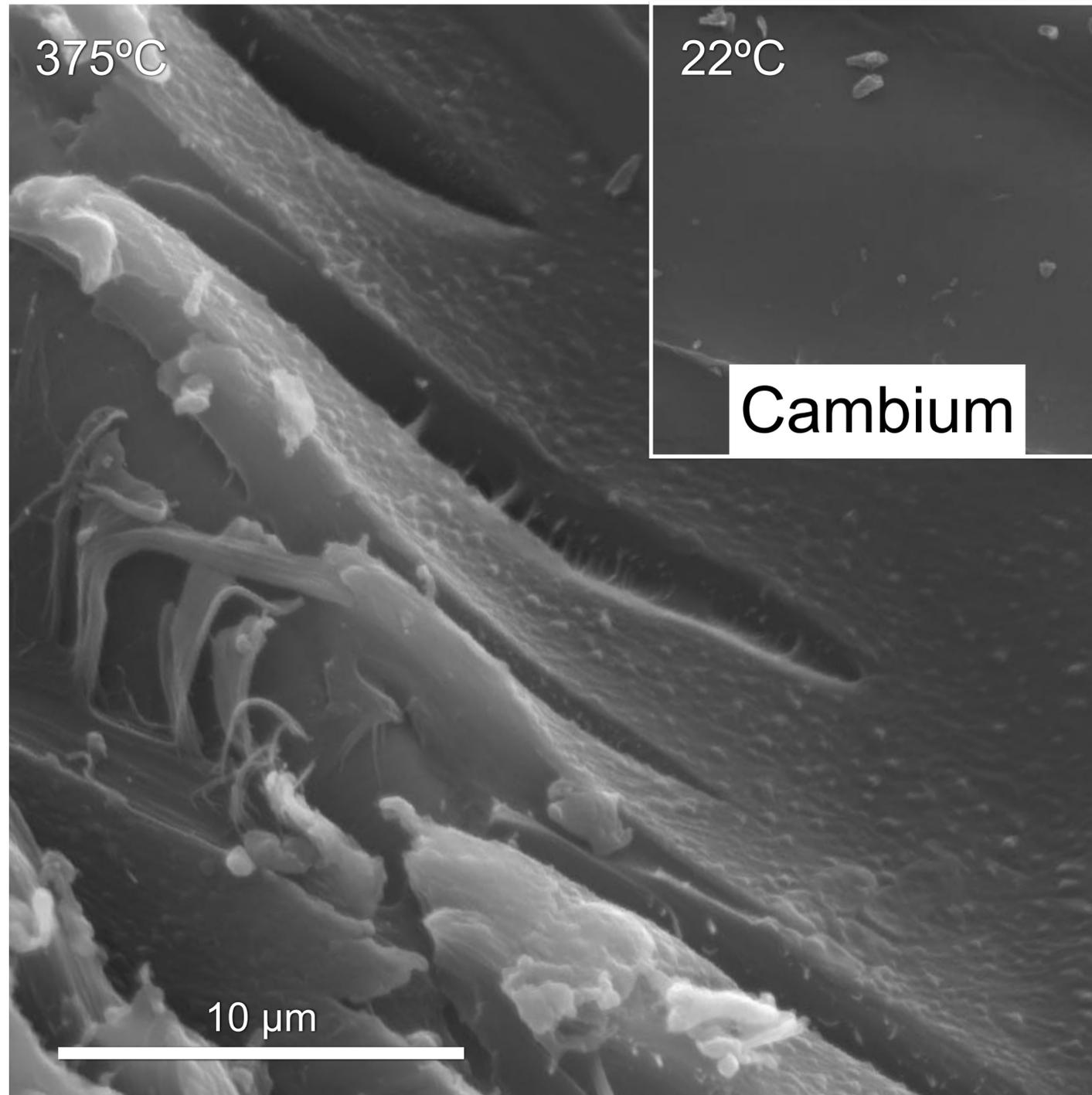


- Pine anatomical fractions change size at different rates.
- Pine particles shrink in volume, but maintain aspect ratio.

- Josie Gruber



Evidence for emerging or deposited volatiles

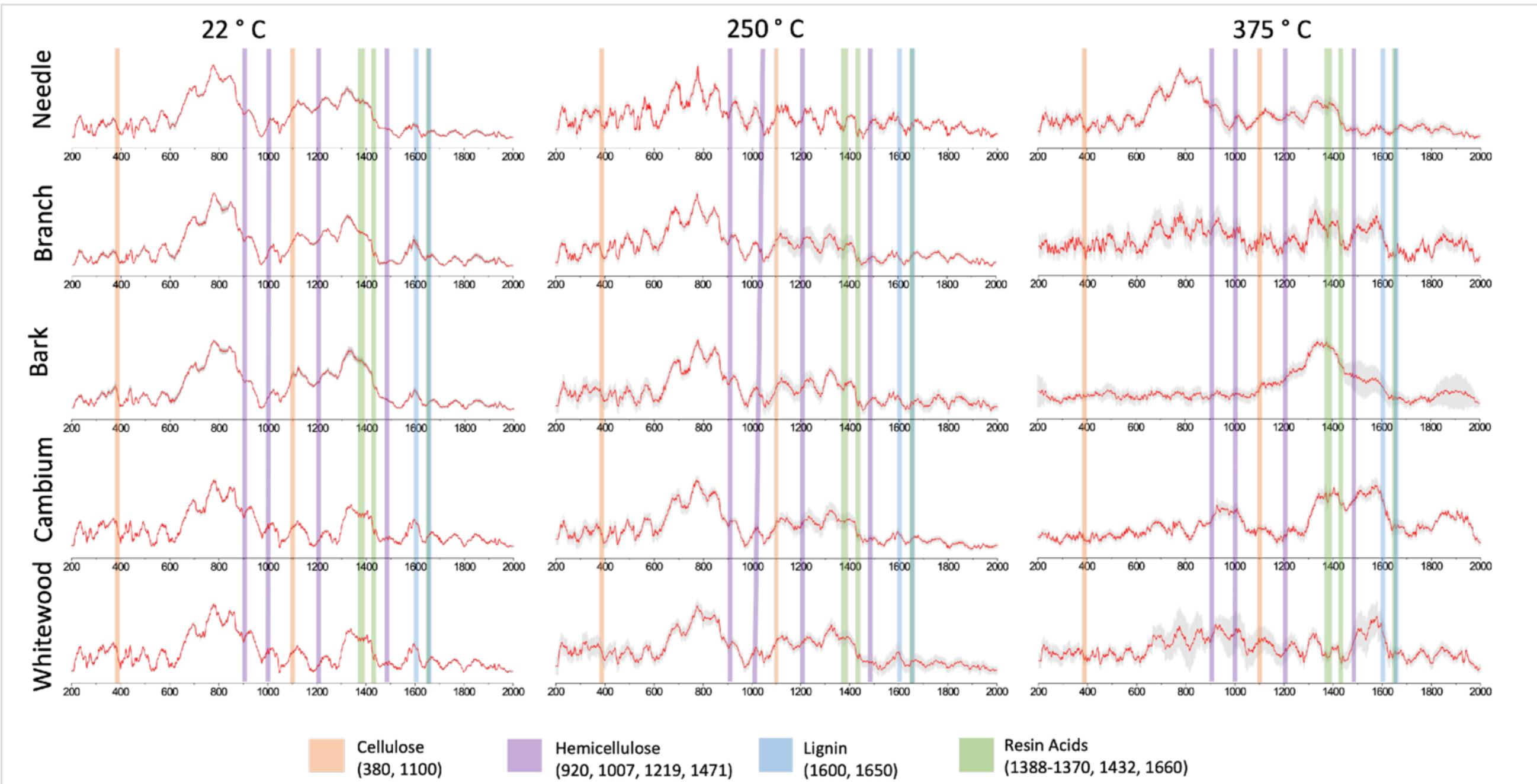


- Surface topology becomes corrugated, with fissures opening between cells and within cell walls contributing to increased roughness
- Fissures are the most prominent in cambium, and whitewood

- Josie Gruber



Pine fractions have variable surface chemistry after heating



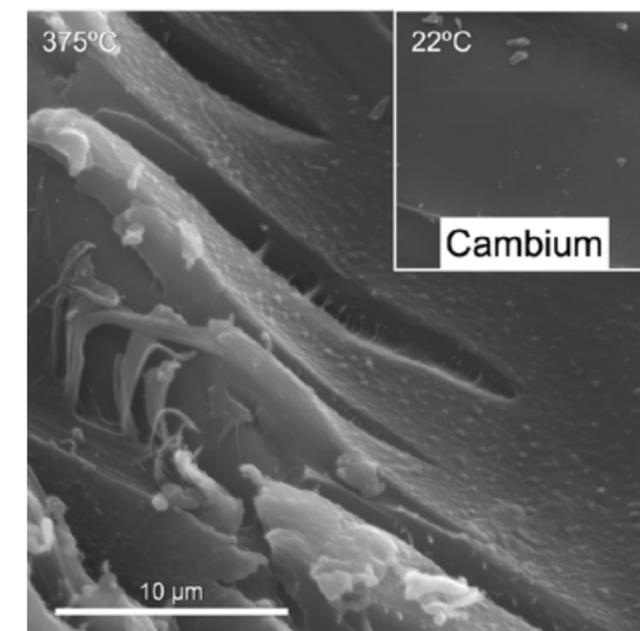
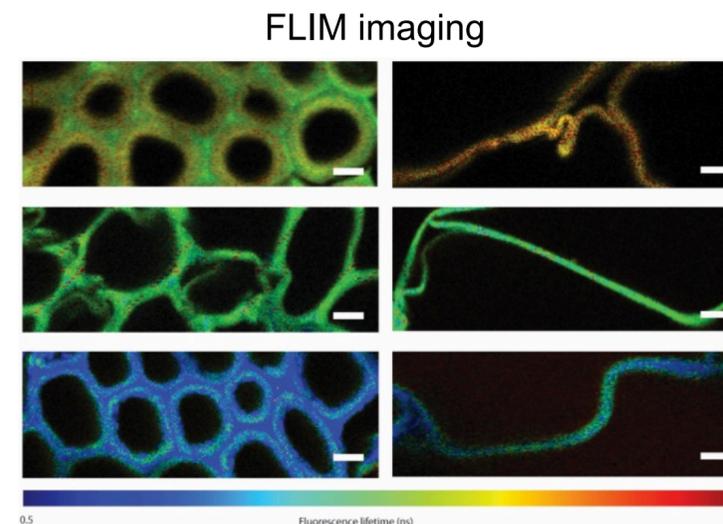
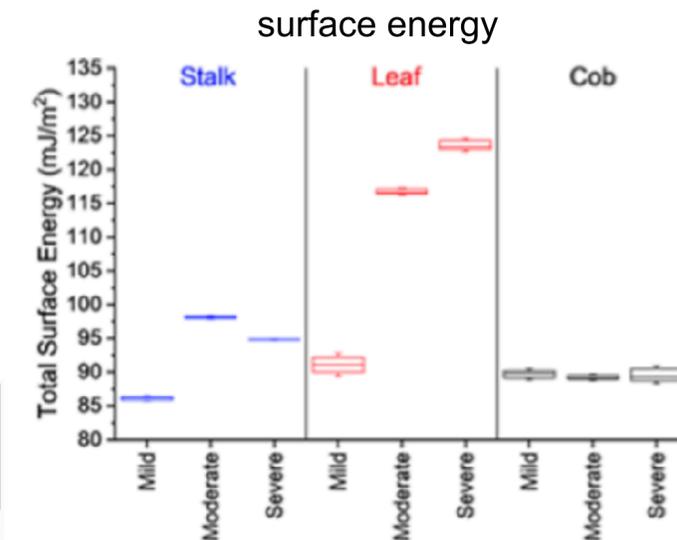
- Confocal Raman spectra average of 3 replicate measures taken per sample, with shading indicating standard error
- Bands of interest associated with cell wall macromolecules: cellulose – orange, hemicellulose – purple, lignin – blue, and resin acids – green

- Yining Zeng



Conclusions and take-home messages

- biomass particles are anisotropic and particle morphology can have a major impact of flowability
- biological degradation impacts has variable impacts on biomass surface attributes
- lignin appears to be changing in degraded corn stover with potential negative impacts on pretreatment and enzymatic saccharification
- pine residue particles respond differently to heating and may develop sticky surfaces



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Renewable Energy
BIOENERGY TECHNOLOGIES OFFICE



Speaker Contact Information



Allison E. Ray, Ph.D.

Research Excellence Lead, Science & Technology
Senior Research Scientist
Idaho National Laboratory
208-526-4554
allison.ray@inl.gov



Bryon S. Donohoe, Ph.D.

Senior Scientist
National Renewable Energy Laboratory
303-384-7773
bryon.donohoe@nrel.gov



Questions?



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