U.S. DEPARTMENT OF ENERGY | Energy Efficiency & Renewable Energy

U.S. Department of Energy (DOE) Bioenergy Technologies Office (BETO) 2019 Project Peer Review

1.2.1.2 Biomass Engineering: Size reduction, drying and densification of high moisture biomass

Technology Area Session: Feedstock Supply & Logistics

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Goal Statement

- The goal of the project from FY18-20 is to solve the particle attrition that results in fines generation during grinding and pelleting and thus enable pelleting as a viable option for production of conversion-ready cellulosic feedstocks.
- The end of project goal is to reduce particle attrition or fines in the corn stover pellet by 80% (less than 7% fines, defined as <425 microns) compared to current process performance (about 35-38% fines in the pelleted product).

Major Outcomes:

a) Production of on-spec material; reduced mass loss during biochemical and thermochemical conversion resulting in increased yields.

b) Increased profitability for biorefineries.

c) More reliable operation of the pelleting process.

d) Reduced pellet production cost.

Relevance:

a) Variable moisture and particle size, and elastic and cohesive properties of biomass is currently limiting the ability of biorefineries to operate at capacity.

b) This project converts biomass into a dense, uniform feedstock with consistent moisture and particle size with minimum fines that exhibits much better flow characteristics than raw milled biomass.

c) Reduction of fines in the pellets can help to reduce mass loss during biochemical and thermochemical conversion.

Quad Chart Overview

Timeline
- Project start date: 10/01/17
- Project end date: 09/30/20
- Percent complete: 40%

	Total Costs Pre FY17**	FY 17 Costs	FY 18 Costs	Total Planned Funding (FY 19-Project End Date)
DOE Funded	$0	$1,018,117	$790,275	$1.850M

Partners: Single lab project

Barriers addressed
- Ft-E: Variable properties of biomass material
- Ft-G: Feedstock Quality & Monitoring

Objective
- Solve the particle attrition which results in fines generation during preprocessing (grinding and pelleting).

End of Project Goal
- The end of project goal is to reduce particle attrition in corn stover by 80% (less than 7% fines, defined as <425 microns) compared to current process performance (about 35-38% fines) in a ring die pellet mill.

1 - Project Overview

History

- The new preprocessing technologies developed in this project (fractional milling, and high moisture pelleting and low temperature drying) reduced the pellet production cost of high moisture corn stover bales by 65% compared to conventional pelleting method followed by the industry (Tumuluru et al., 2017).
- Significant commercial interest for the new preprocessing technologies developed at INL.

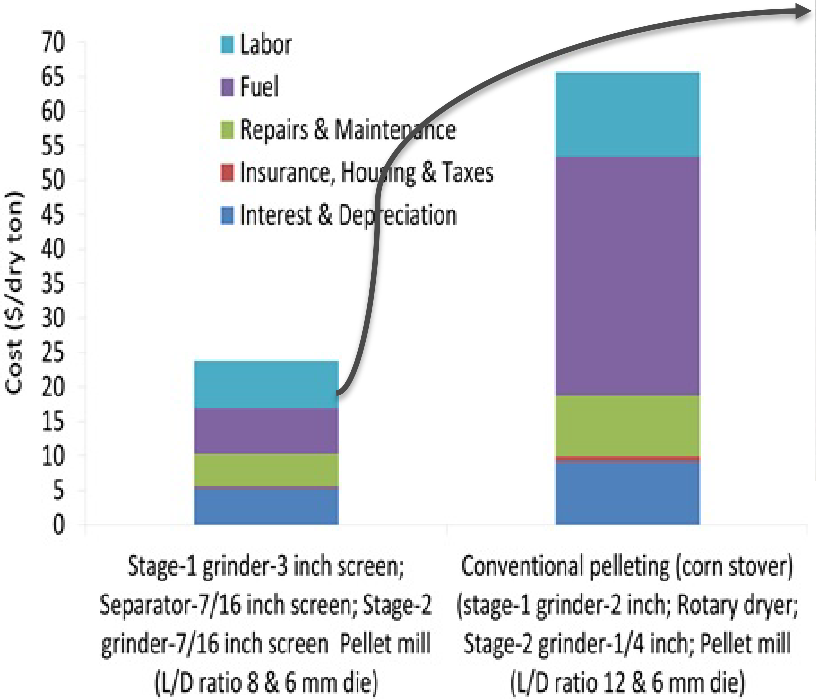

Fig. 1: Techno-economic analysis of fractional milling, high moisture pelleting tested on INL's PDU (Tumuluru et al., 2017).

Fig. 2: Picture of corn stover pellets.

Lessons learned

- Efficient moisture management is critical to reduce the preprocessing cost.
- High moisture pelleting reduces the pellet production cost significantly.
- Larger screen size (7/16th) in the stage-2 grinder reduces the grinding energy significantly.

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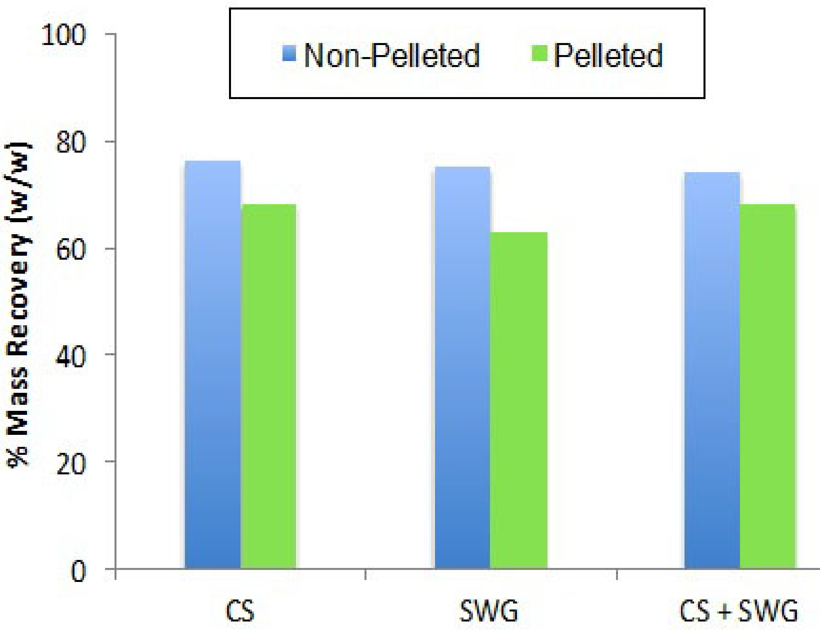
1-Project Overview (cont.)

Several barriers still exist to deploy pelleting technology to the biofuels and bioproducts market.

Challenges

- INL/NREL AOP project (FY17 WBS 2.2.1.102; Ray and Nagle 2016; Nagle et al., 2016), and communications with industrial biofuel projects, identified issues of biomass particle attrition (generation of fines) during grinding and pelleting process.
- The particle attrition that results in fines are lost during biochemical and thermochemical conversion and reduce the conversion efficiency.



Overall objective: Solve the particle attrition issue during biomass grinding and pelleting and enable pelleting as a viable option for production of on-spec material or conversion-ready cellulosic feedstocks.

End of project goal: Reduce particle attrition in corn stover by 80% (less than 7% fines, defined as <425 microns) compared to current process performance.

Note: In figure 3 the pellets are made commercially using a 1/4-inch hammer milled grind where the process conditions are not optimized for attrition.

Fig. 3: Mass recovery after deacetylation w/ 0.2% NaOH (w/w), 1% Acid (w/w) impregnation (Ray and Nagle, 2017).

2-Approach (Management)

Management approach

- DOE, Merit Review in 2017.
- Participate in BETO, Feedstock Supply & Logistics (FSL) monthly call and update the Technology Manager regarding progress of the project.
- Major milestones with definite particle attrition targets, and Go/No-Go decision points.
- Work with INL analysis team to update the state of technology costs based on the results obtained in this project.
- Validate the processes developed in this project by peer review publication.

Critical success factors

Optimize the pellet mill process conditions (preheating temperature, compressive force) and feedstock properties (moisture content and screen size of the grind) and binders to optimize particle residence time and minimize the particle attrition to meet the established targets (Table 1).

Table 1: Particle attrition targets established for the project

	FY-17 (current value in pellets produced using conventional method)	FY-18	FY19 (go-no-go)	FY-19	FY-20
Particle attrition targets (%)	35-38	21	14	10.5	7

2-Approach (Technical)

Hypothesis

Conduct fundamental research that allows proper matching of process variables with biomass material properties to optimize the biomass residence time in pellet die and improve particle binding.

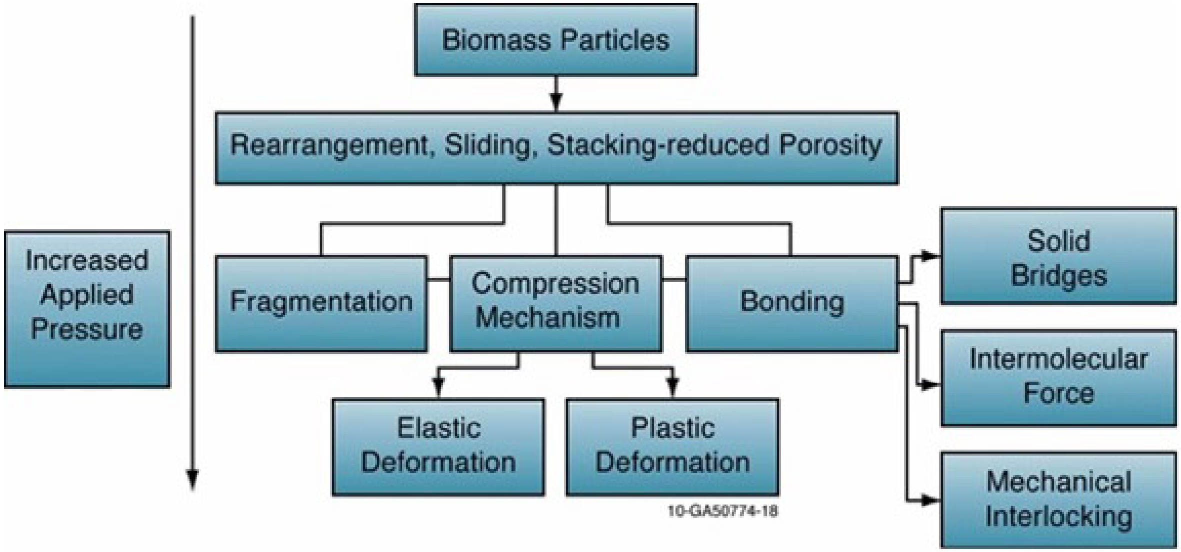


Fig. 4: Particle deformation mechanisms under compression (Tumuluru et al., 2011).

Impact of pressure on particle binding

- Pressure reduces the melting point of the particles and increases contact areas.
- Water increases the particle bonding due to interfacial forces and capillary pressures.
- Solid bridges formed during densification results in agglomeration of biomass particles.
- Solid bridges are formed due to chemical reactions, sintering, solidification, hardening of the binder, hardening of the melted substances, or crystallization of the dissolved materials.

2-Approach (Technical) (Cont.)

Experimental Approach

Understand the effect of pelleting process variables on the particle dimensions, physical properties of pellets and bonding phenomena due to mobilization of lignin and other binding agents in the biomass.

Feedstock tested: 1/4 and 7/16-inch hammer grind corn stover

Experimental plan

1st set of experiments → **Objective:** Impact of moisture content, residence time and hammer mill screen

Process variables tested

a) Feedstock moisture content (10, 15 and 20%, w.b.)
b) Hammer mill screen size (1/4 and 7/16 inch)
c) Residence time (45-150 sec)

Note: Compressive force and preheating temperature were kept constant at 7kN and 110°C

For the 2nd set of experiments optimized hammer mill screen size and residence time identified based on 1st set of experiments were used.

2nd set of experiments → **Objective:** Impact of moisture content, preheating temperature and compressive force

Process variables tested

a) Feedstock moisture content (10, 15 and 20%, w.b.)
b) Preheating temperature (70-110°C)
c) Compressive force (7-11kN)

→ **Pellet properties measured**

- Particle attrition (%)
- Particle dimensions (mm)
  - Geometric mean particle length (mm)
  - d10 (mm)
  - d50 (mm)
  - d90 (mm)
- Unit density (kg/m3)
- Durability (%)

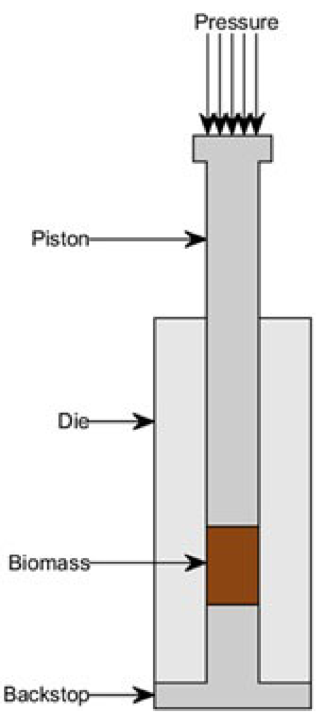


Fig. 5: Single pellet press

FY19 (go-no-go)-14% fines in the pelleted corn stover

2-Approach (Technical) (Cont.)

Methods

Particle attrition: Solid biofuels – Determination of particle-size distribution of material within pellets-ISO 17830:2016.

Unit density: Mass of each pellet divided by its volume (ASABE , 2007).

Durability: Drop test (pellet is dropped from certain height and fines generated are determined by mass loss) (Al-Widyan and Al-Jalil, 2001).

Particle size distribution: ANSI/ASAE S424.1 (1992) (Geometric mean particle length, d10, d50 and d90). (for example d50 is 50% of the material is below the measured particle size).

Experimental data analysis

a) Response surface models b) Surface plots and c) Optimization of response surface models

Pellet characterization studies

SEM, CT-scan, FIB and FTIR

Table 2: Raw material properties of 1/4 and 7/16 inch grind hammer mill corn stover

	Grind properties before pelleting				
Hammer mill Screen size	Geometric mean particle length (mm)	d10 (mm)	d50 (mm)	d90 (mm)	Attrition 425 µm (%)
1/4"	0.80	0.18	0.60	1.78	34.36
7/16"	1.20	0.26	0.91	2.87	20.24

3-Technical Accomplishments (Cont.)

Table 3: Response surface models for pellet properties.

Pellet properties	RSM model	R^2
1/4- inch grind		
Particle attrition (%)	$26.82540 - 1.41808x_1 + 0.063191x_2 + 0.057800x_1^2 + 0.000112x_2^2 - 0.003644x_1x_2$	0.71
Geometric mean particle length (mm)	$1.068261 + 0.015504x_1 - 0.001616x_2 - 0.000588x_1^2 - 0.000002x_2^2 + 0.000081x_1x_2$	0.69
7/16-inch grind		
Particle attrition (%)	$13.71364 - 0.250182x_1 + 0.0012281x_2 + 0.017750x_1^2 + 0.000327x_2^2 - 0.002139x_1x_2$	0.66
Geometric mean particle length (mm)	$2.067707 - 0.071730x_1 - 0.004429x_2 + 0.002230x_1^2 + 0.000013x_2^2 + 0.000049x_1x_2$	0.50

Note: x_1: Feedstock moisture content; x_2: Residence time (min); R^2: Coefficient of determination

Surface plots for 1/4 inch grind

Fig. 6

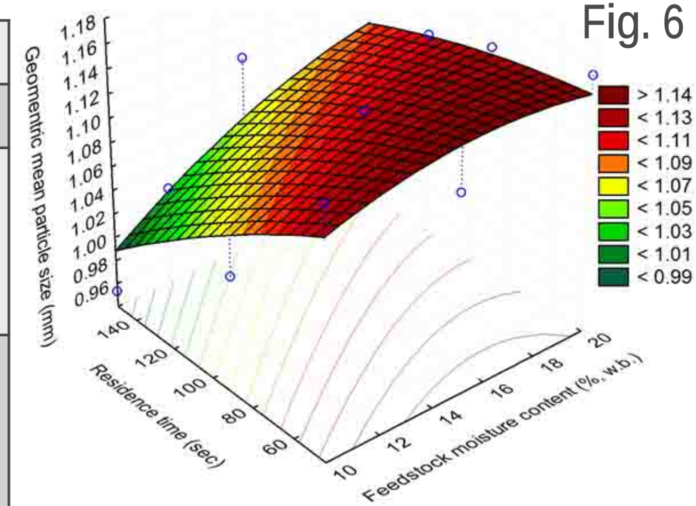
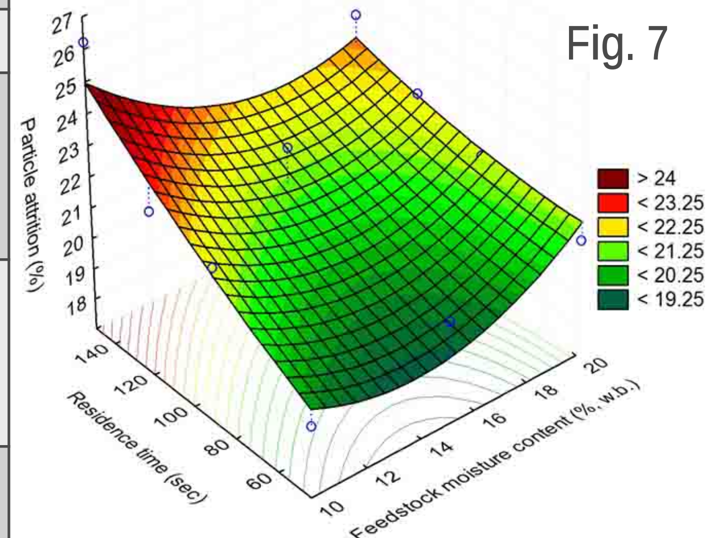


Fig. 7



Low to medium moisture and lower residence time minimized the attrition and maximized the geometric mean particle length

3-Technical Accomplishments (Cont.)

Fig. 8

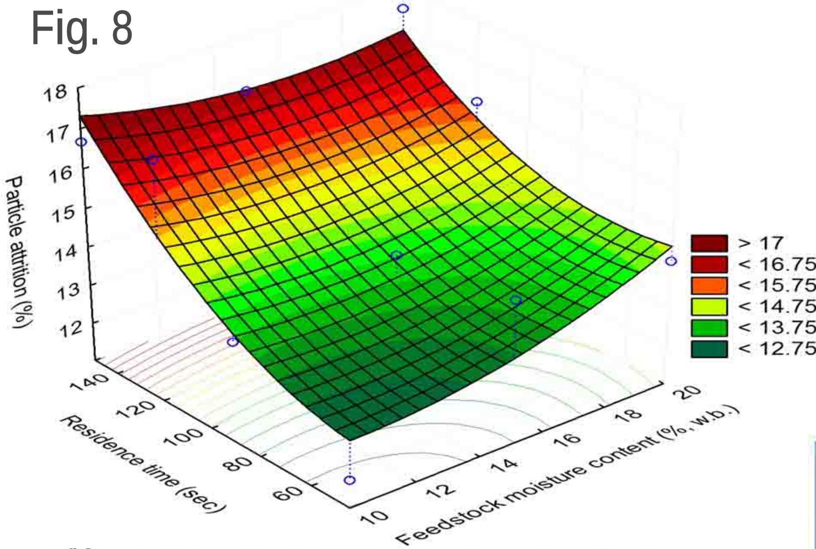
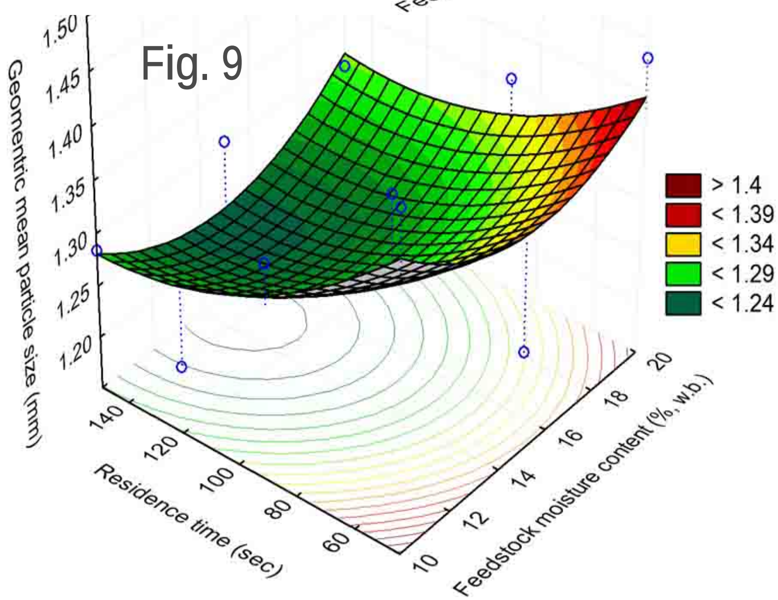


Fig. 9



Surface plots for 7/16 inch grind

- Lower moisture (10%, w.b.) and lower residence time (45 sec) minimized the particle attrition.
- Medium to higher moisture content (15-20%,w.b.) and lower residence time (45 sec) maximized the geometric mean particle length.

Optimization of response surface models

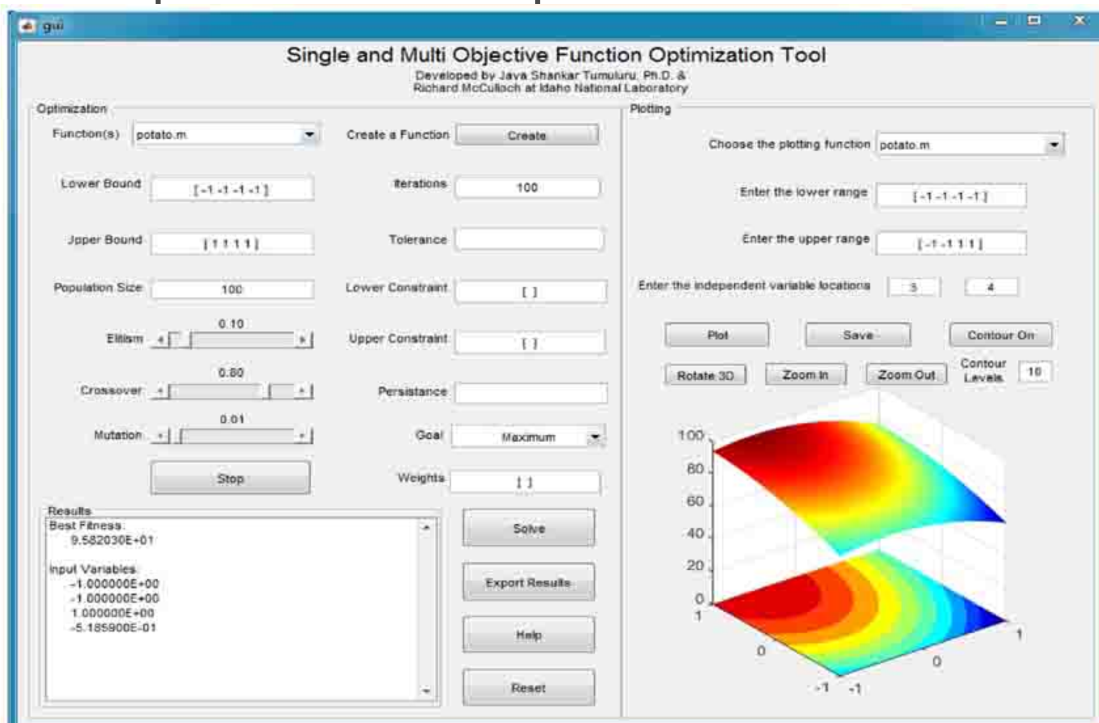


Fig. 10: Hybrid genetic algorithm (Tumuluru and McCulloch, 2016).

3-Technical Accomplishments (Cont.)

Table 4: Optimized pelleting process conditions identified using hybrid genetic algorithm

	Maximum	Minimum	Optimum process conditions	
1/4 inch grind				
			Feedstock moisture content (%, w.b.)	Residence time (sec)
Particle attrition (%)		19.07	13.80	45.11
Geometric mean particle length (mm)	1.147		16.28	45.41
d10	0.295		13.73	45.04
d50	0.805		14.67	45.31
d90	2.662		19.61	67.95
Unit density (kg/m3)	1286.83		19.88	149.87
7/16th inch grind				
			Feedstock moisture content (%, w.b.)	Residence time (sec)
Particle attrition (%)		12.75	10.34	45.22
Geometric mean particle length (mm)	1.417		10.19	45.10
d10	0.370		10.10	45.22
d50	1.058		19.93	45.13
d90	3.080		19.63	45.24
Unit density (kg/m3)	1282.51		11.55	149.82

3-Technical Accomplishments (Cont.)

Table 5: Response surface models based on central composite design for 7/16 inch grind

Pellet properties	RSM model	R^2
Particle attrition (%)	$65.44824 - 0.604109x_1 - 6.39153x_2 - 0.376453x_3 + 0.011764x_1^2 + 0.372273x_2^2 + 0.002685x_3^2 + 0.20000x_1x_2 - 0.008188x_2x_3 - 0.002x_3x_1$	0.66
Geometric mean particle length (mm)	$-0.982904 + 0.014619x_1 + 0.246677x_2 + 0.023630x_3 - 0.000032x_1^2 - 0.013792x_2^2 - 0.000149x_3^2 - 0.000932x_1x_2 + 0.000249x_2x_3 + 0.000075x_3x_1$	0.73
Note: x_1: Feedstock moisture content; x_2: Compressive force (kN); x_3: preheating temperature (°C)		

Fig. 11

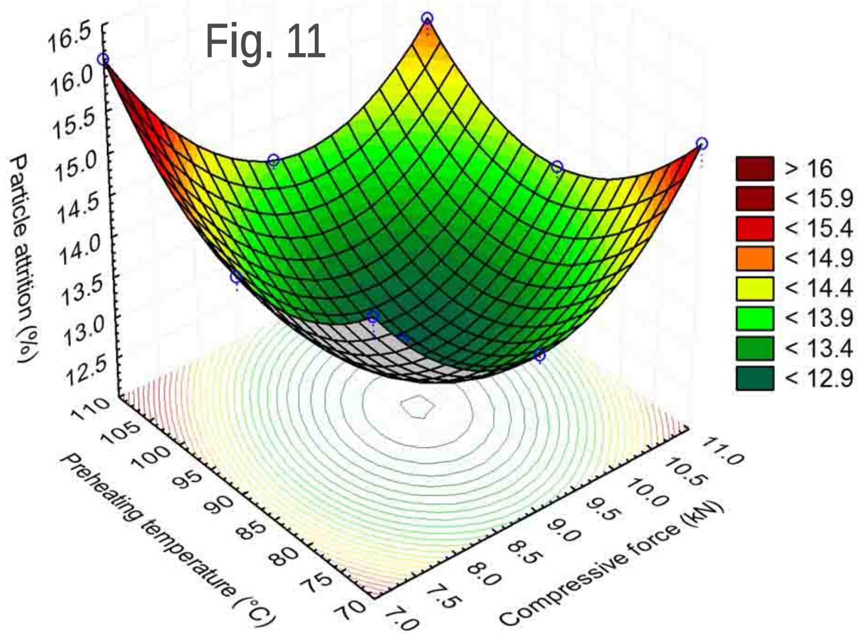
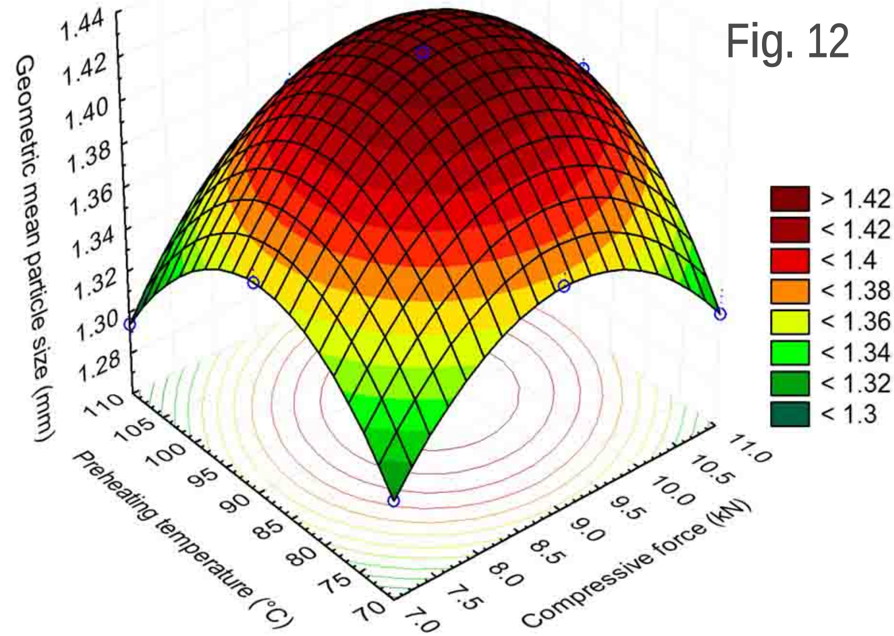


Fig. 12



Surface plots clearly indicate that particle attrition decreases when the mean particle size increases.

3-Technical Accomplishments (Cont.)

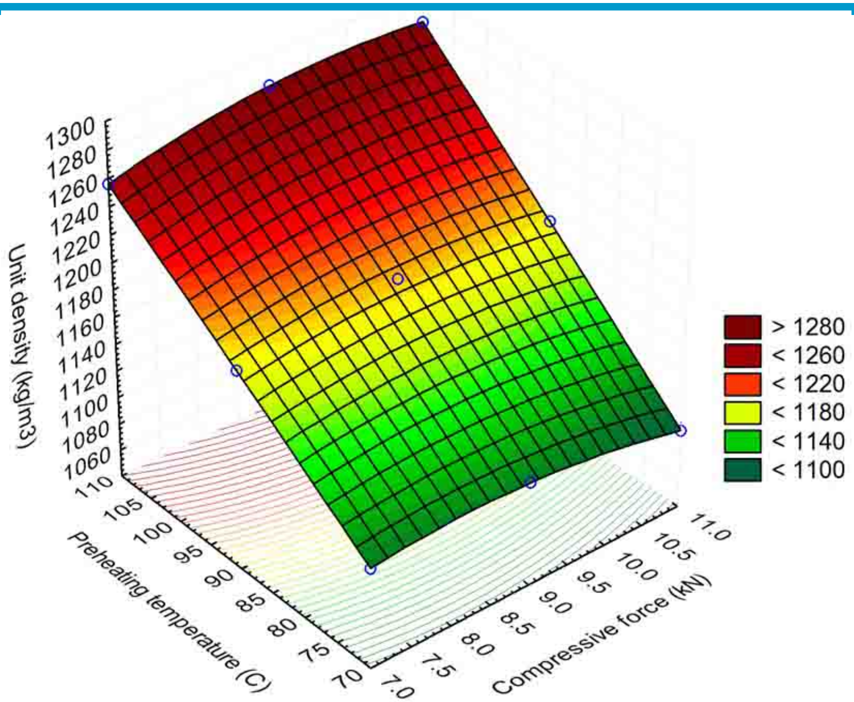


Fig. 13: Impact of preheating temperature and compressive force on pellet unit density

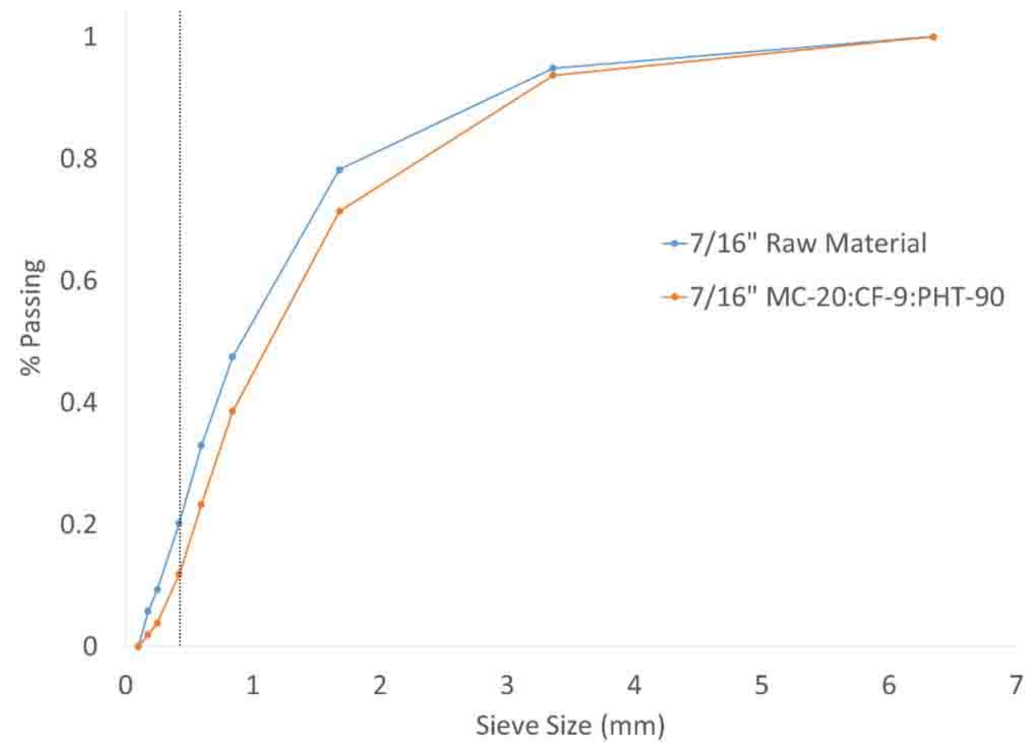


Fig. 14: Comparison of percent passing of raw and pelleted biomass for 7/16 inch hammermill grind at optimized process conditions

Higher preheating temperature increased the unit density and durability of the pellets

3-Technical Accomplishments (Cont.)

Table 6: Optimized process conditions identified using hybrid genetic algorithm for 7/16-inch grind

	Maximum	Minimum	Optimum process conditions		
7/16 inch grind					
			Feedstock moisture content (%, w.b.)	Compressive force (kN)	Preheating temperature (°C)
Particle attrition (%)		11.94	19.93	9.00	91.23
Geometric mean particle length (mm)	1.487		19.82	9.15	91.89
d10 (mm)	0.373		19.98	8.89	89.81
d50 (mm)	1.127		19.97	8.99	92.57
d90 (mm)	3.151		19.96	9.62	90.89
Unit density (kg/m3)	1296.31		10.16	10.83	109.97
Durability (%)	99.81		10.22	8.765	109.92

Table 7: Particle attrition established and achieved

	FY-17	FY-18	
Particle attrition targets	Current fines in the pellets	Target (%)	Achieved (%)
1/4-inch grind	35-38	21	19
7/16-inch		21	11.94

3-Technical Accomplishments (Cont.)

Pellet characterization studies

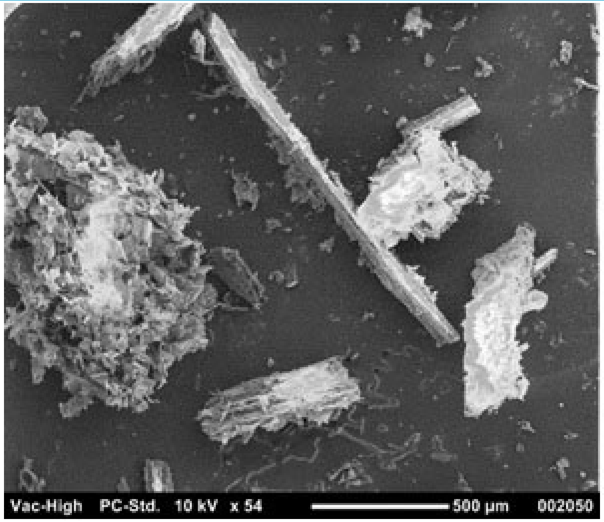


Fig. 15: SEM of ground corn stover (1/4 inch ground corn stover)

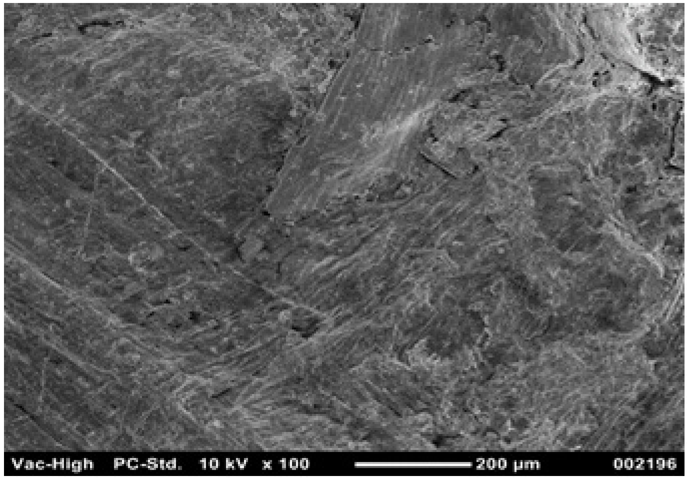


Fig. 16: Internal surface of pellets (1/4 inch grind, MC-10:CF-9:RT45)

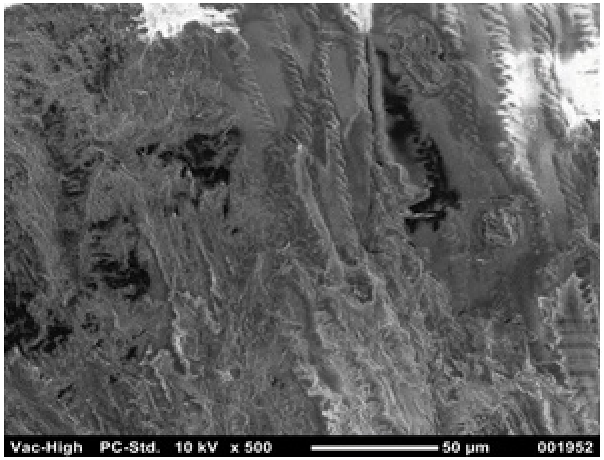


Fig. 17: SEM shows still intact cell wall post pelleting (7/16 inch grind, MC-20:CF-9:RT120)

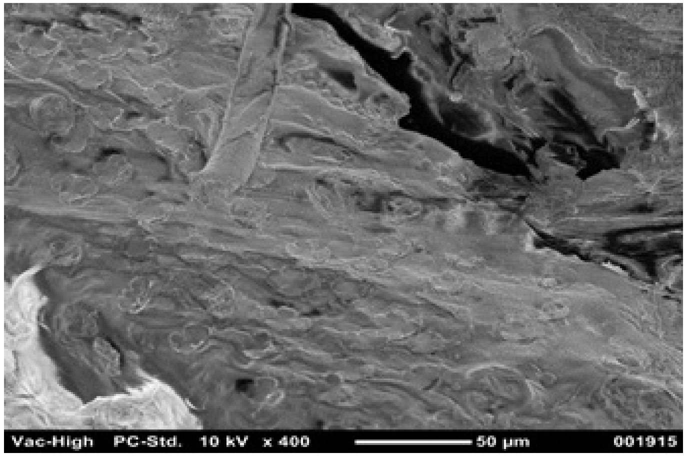
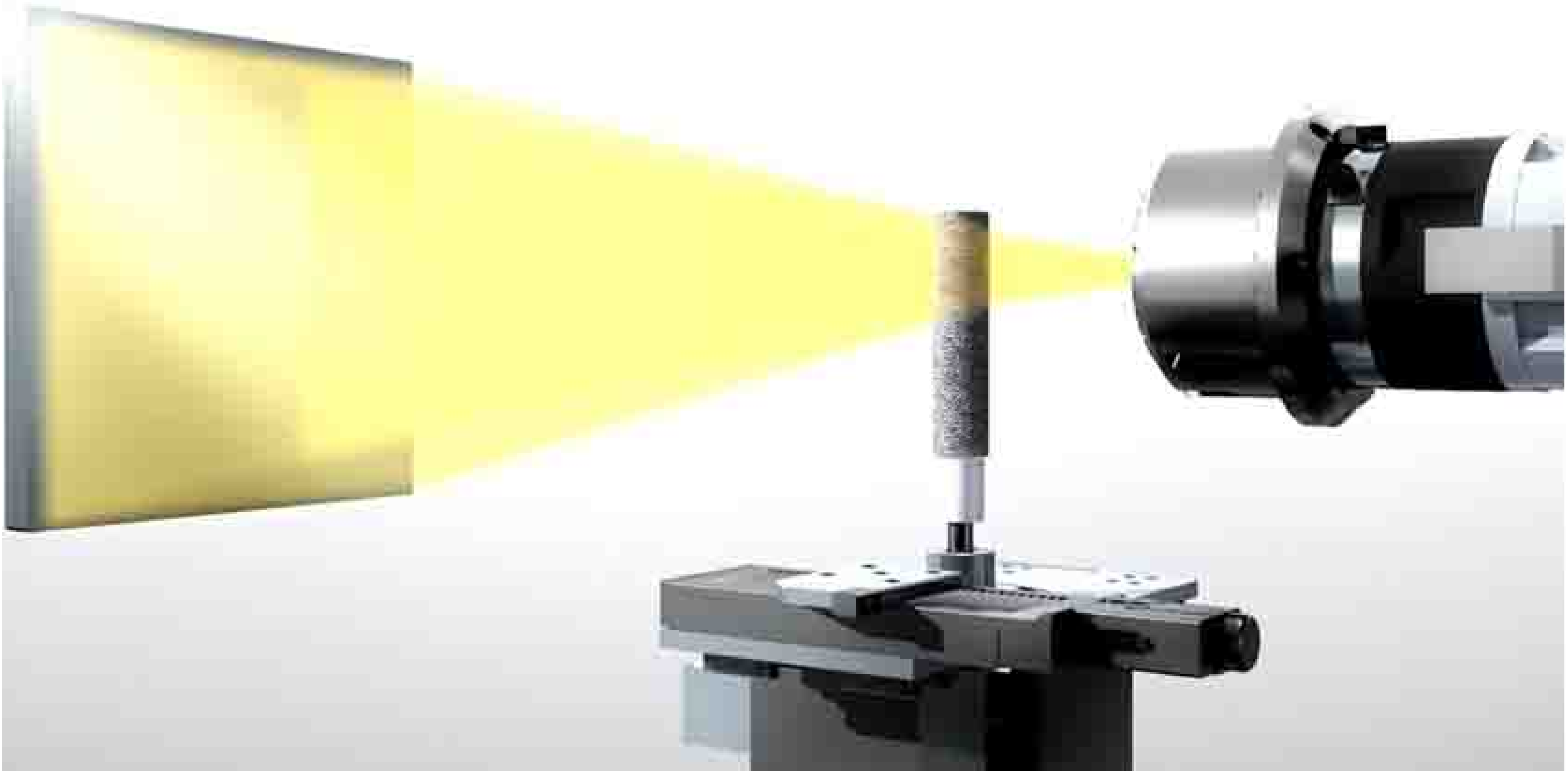


Fig. 18: SEM shows resilience of stomata and villi during pelleting (7/16 inch grind, MC-10:CF-9:RT45)

- Raw material showed ordered fibrous packets of plant cells and disordered clusters of lamellae
- Pelleting resulted in cellular structure disruption, but stomata and villi were still visible
- Stomata guard cells tend to have high lignin content, their remaining intact after the pelleting process, indicates only a partial mobilization of all the lignin during pelleting.

3-Technical Accomplishments (Cont.)

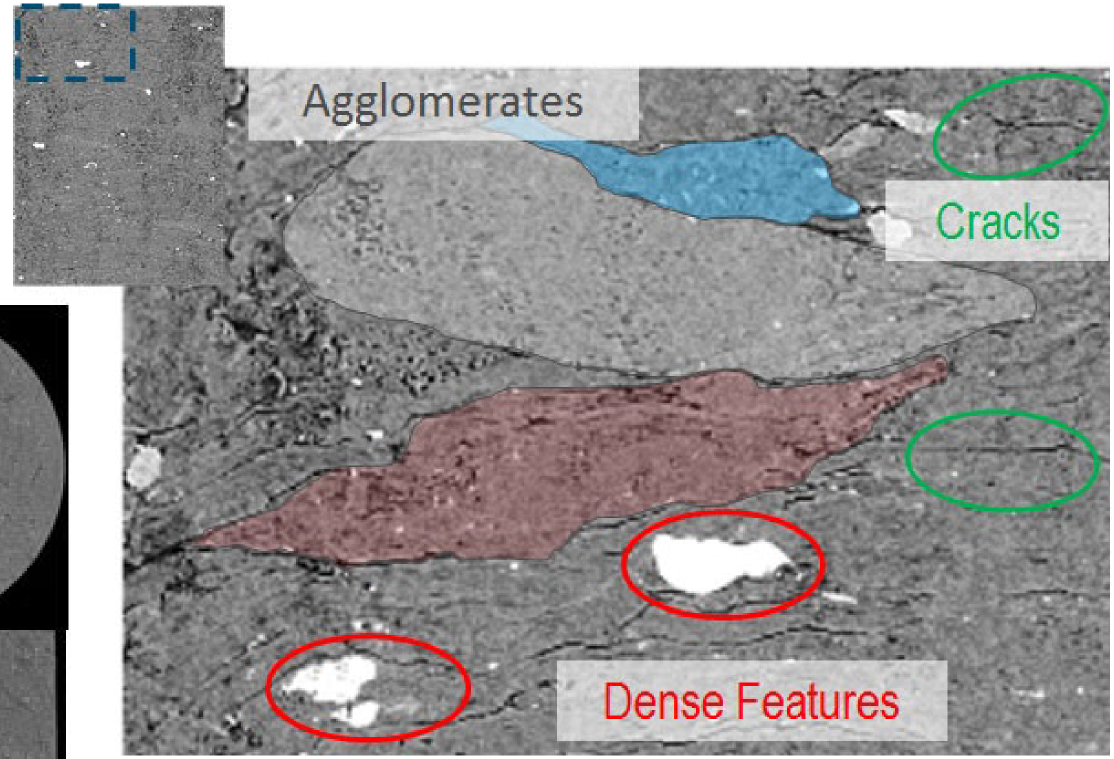
A Traditional X-Ray CT setup



3-Technical Accomplishments (Cont.)

- X-ray CT provides 3D non-destructive images of pellets
- Enables spatial and morphological characterization without destroying pellet

What We Can See in Pellets?



Primary Features Extracted:

1. Cracks
2. Dense Features
3. Agglomerates

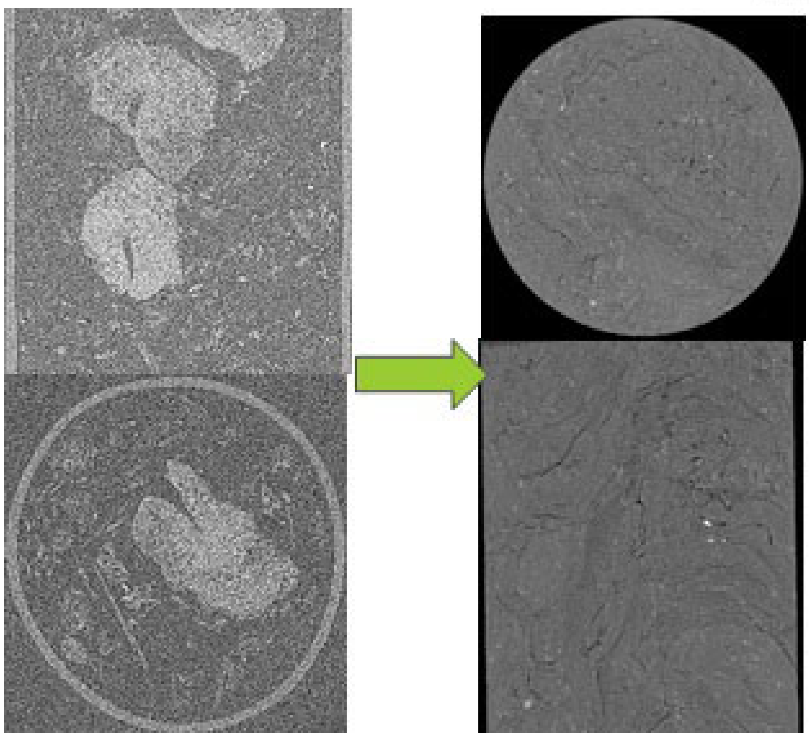


Fig. 19: CT-scan of raw and pelleted corn stover

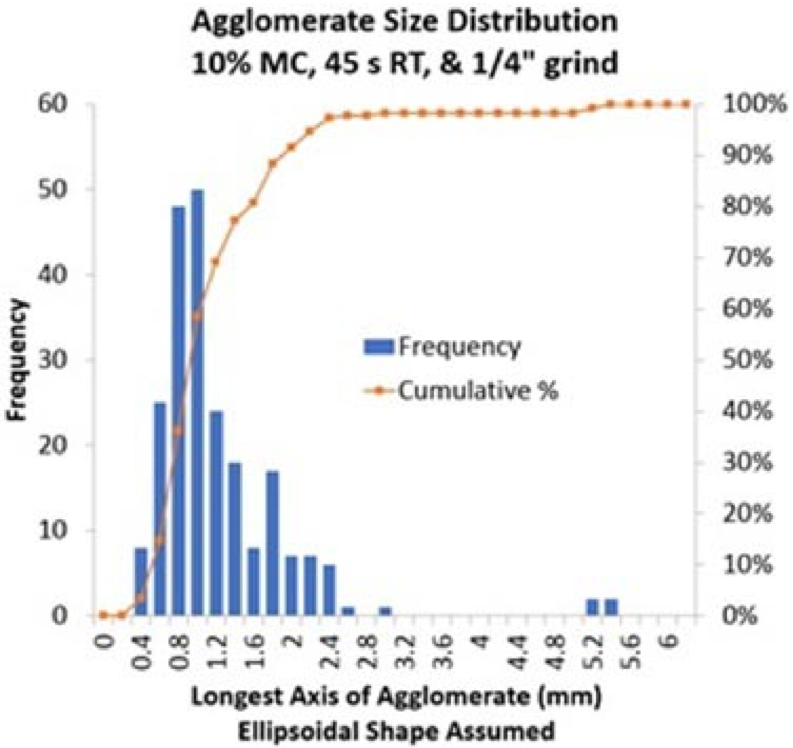
3-Technical Accomplishments (Cont.)

Example of Results for:

Residence Time: 45s

Moisture Content: 10% (w.b.)

Grind Size: 1/4 inch



- # of agglomerates: 224
- Avg. Longest Axis Length: 1.27 mm
- Geometric mean particle length calculated using Rotop: 1.17 mm

Agglomerate extraction methodology

1. Isolate inter-agglomerate cracks with connectivity algorithm
2. Use surfaces as seed point for Euclidean distance transform
3. Use watershed method to fill in borders of agglomerates between cracks.

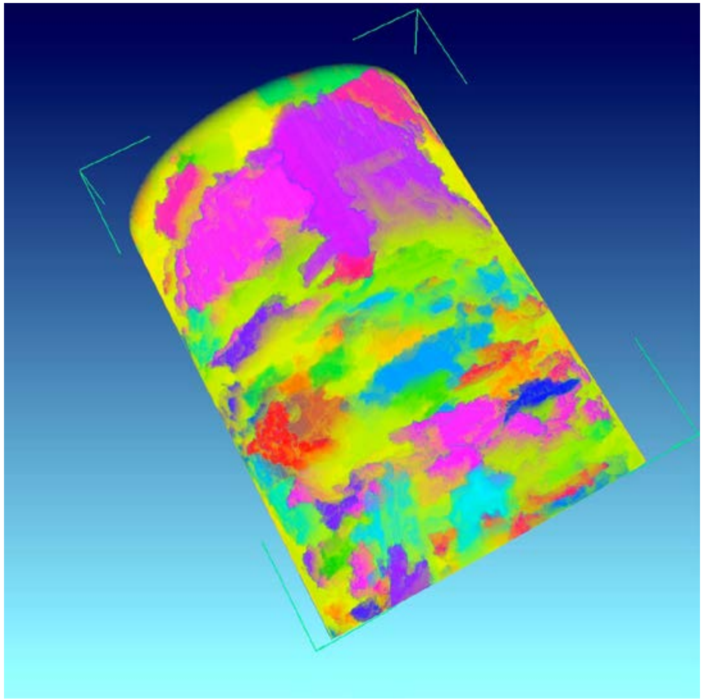


Fig. 20: 3D view of the pellet with agglomerate distribution

3-Technical Accomplishments (Cont.)

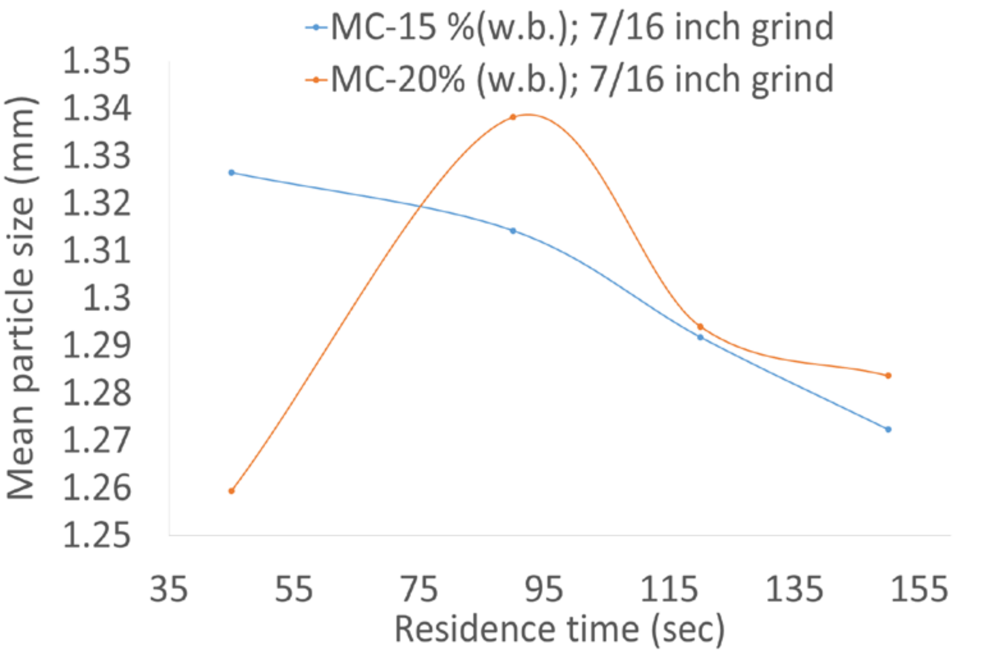


Fig. 21: Effect of moisture and retention time on the mean particle size

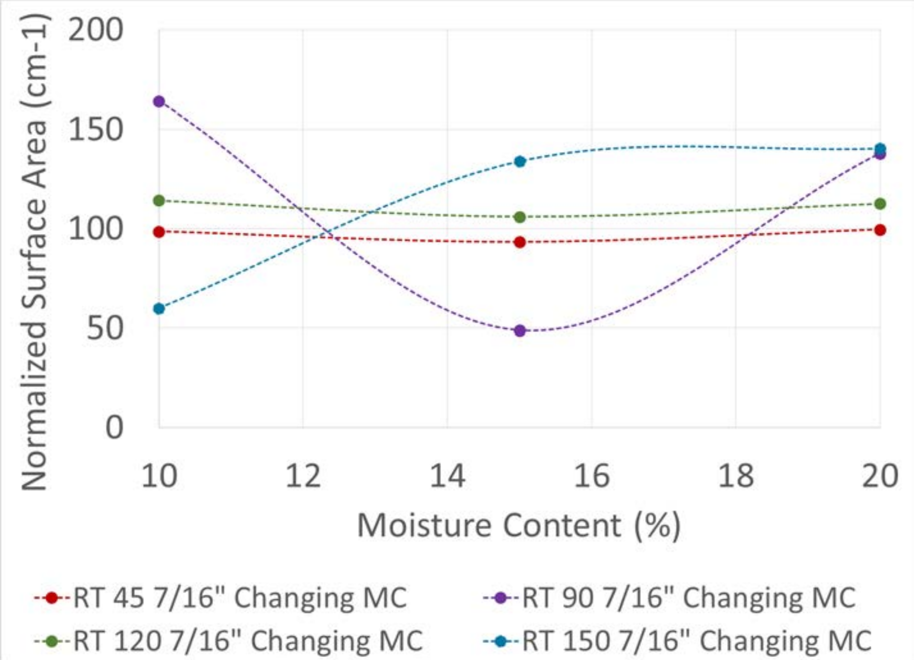


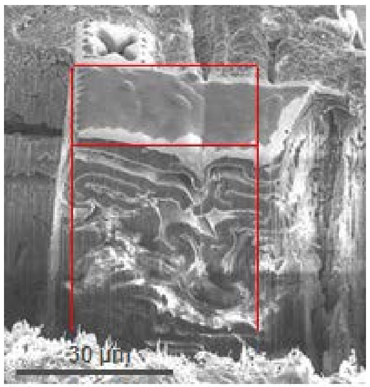
Fig. 22: Effect of moisture and retention time on the surface area

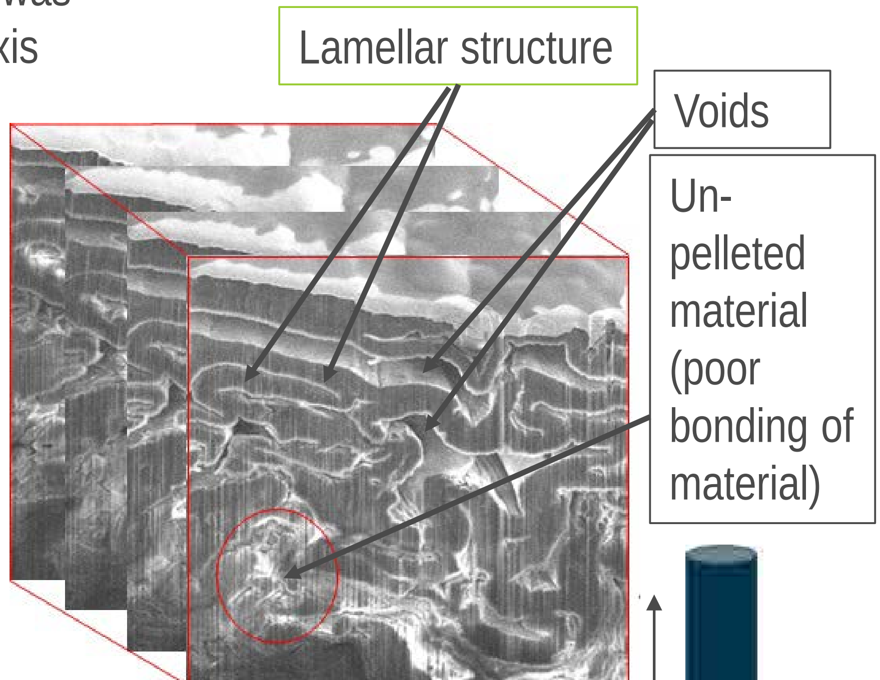
- Mean particle size decreased with increase in residence time at 15 % (w.b.) moisture content.
- Residence time and moisture content have a significant effect on the surface area of the agglomerates.
- Using the CT-scan data we can design the pelleting process to meet the particle surface area required for the conversion process.

3-Technical Accomplishments (Cont.)

Focused Ion Beam (FIB) Tomography of Corn Stover Pellet

Serial sectioning: Pellet was sectioned along the Z axis





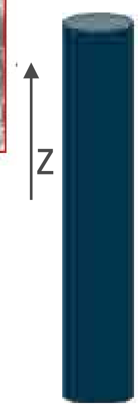


Fig. 23: Focused Ion Beam image of Corn Stover Pellet

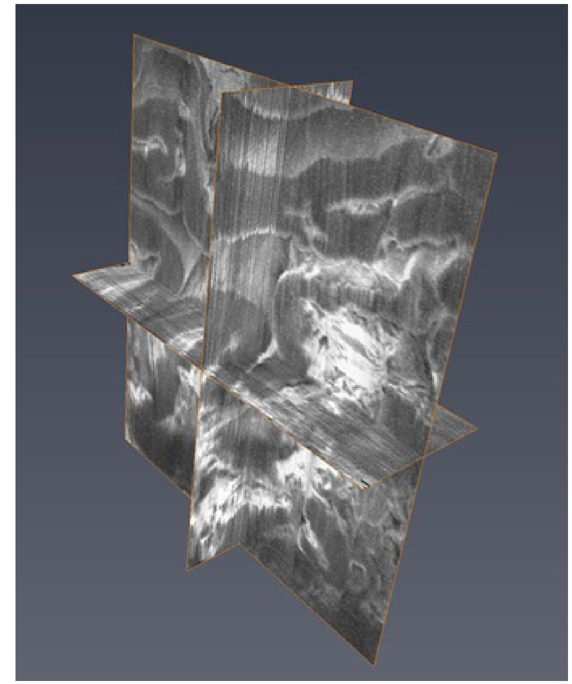


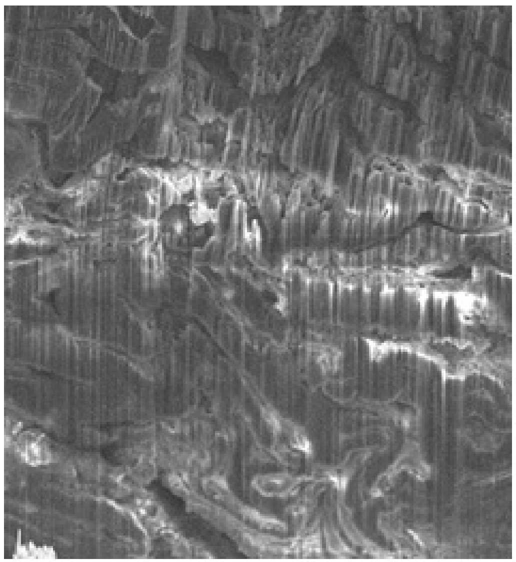
Fig. 24: 3D Visualization of pellet using FIB images

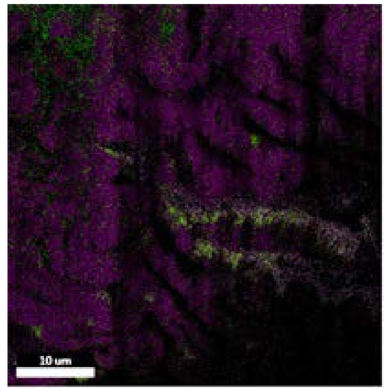
- A lamellar structure is observed.
- Lamella are ~10 µm thick.

Future work is focused on quantification of total void volume and identification of microstructure and how the pelleting process variables impact them.

3-Technical Accomplishments (Cont.)

Energy-dispersive X-ray spectroscopy (EDS) Mapping





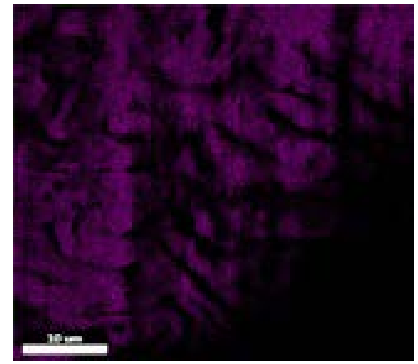


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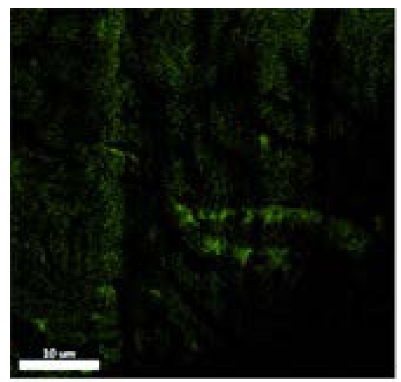
Fig. 25: EDS mapping of Corn Stover Pellet



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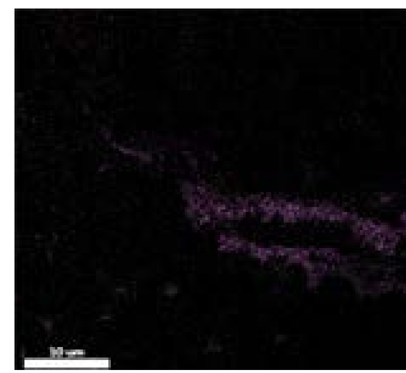


Si

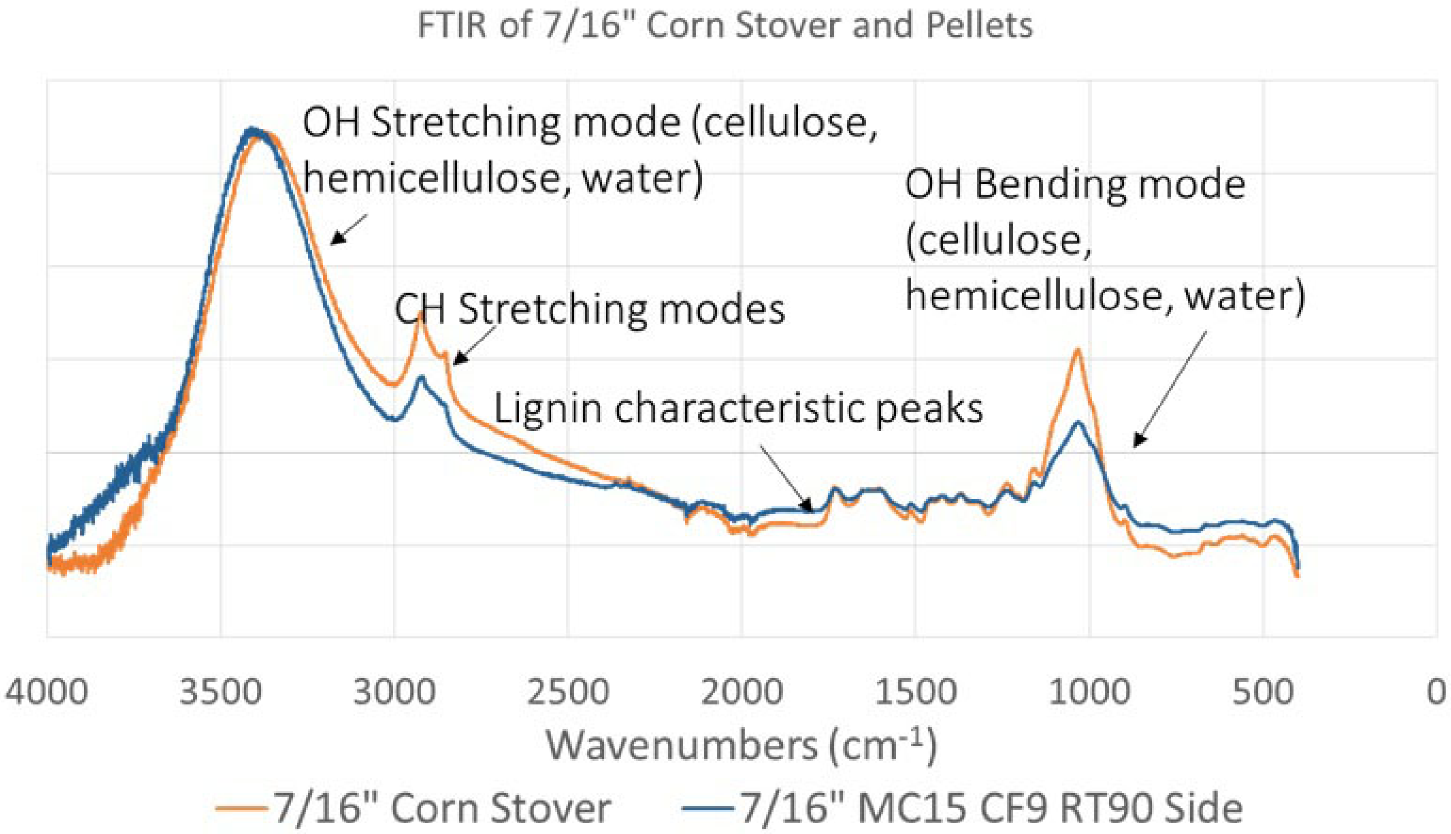
Areas of high oxygen and silicon were observed in the pellet

Future work is focused on

- Quantification of carbon, silicon and oxygen distribution in the pellet
- How the process conditions can influence the inclusions in quality or chemistry.

3-Technical Accomplishments (Cont.)

FTIR analysis



Small changes in lignin peaks, indicating not all lignin is mobilized during pelleting process corroborates with our SEM observations.

Fig. 26: FTIR of raw and pelleted corn stover

Note: Collected data using a Bruker Vertex 70 FTIR spectrophotometer using Platinum ATR with diamond window

4 - Relevance

Impact

- This project addresses a recognized barrier (feedstock quality) which effects the yields during biochemical and thermochemical conversions, and thus supports the bioeconomy.
- The outcomes specifically address biomass particle attrition issues during preprocessing as it is a major impediment for using pelleted feedstock for biofuels production.
- Helps to achieve DOE vision of commoditization of the biomass at lower cost.
- Converts diverse forms of biomass into consistent, high-quality commodity products, helps to efficiently handle, store and transport the biomass to biorefineries.
- Reduced pellet production cost, production of on-spec material, increased convertibility and increased profitability for biorefineries.
- The preprocessing solutions developed in this project can be transferred to 2nd generation biorefineries, which can help to improve operational reliability and potentially make pelleting a more cost-effective option in the United States.
- High moisture pelleting developed in this project is 2018 R & D 100 Award finalist.

Output

- Particle attrition process models developed in this project can be used by the industry for commercial-scale implementation to reduce the fines during pelleting.
- Processes developed in this project can be used by biomass processors and designers for producing biofuels, chemicals, and bioproducts.

5 – Future Work (Cont.)

- Test the pelleting process conditions (compression pressure, die diameter, feed rate, and steam conditioning) and feedstock properties (moisture content and screen size) on corn stover particle behavior in a laboratory scale flat die and ring die pellet mill.
- Impact of fines removal from the hammer milled corn stover on the particle binding behavior during pelleting. Fines generated during hammer milling are high in ash content (Table 8).

Table 8: Ash content in the fines generated during hammer milling of corn stover

Sieve Size (µm)	Total Ash in fines (1/4-inch grind) ("as received basis")	Total Ash in fines (7/16-inch grind) ("as received basis")
Raw material	8.99	6.73
400	6.37	4.05
250	5.48	3.31
212	4.53	2.56
150	4.01	2.17
106	3.57	1.55
45	3.07	1.11
Pan	1.98	0.28

- Fines removal from the biomass significantly reduces the ash content.
- We can remove more than 50% of total ash in corn stover when we screen 250 micron material from the grind.

- Effect of grinder type (hammer mill versus rotary shear) on the quality and fines particles in the pelleted product.
- Effect of natural and chemical binders on particle agglomeration behavior.

5 – Future Work (Cont.)

- Optimize the pelleting and feedstock process conditions, feed rate and binders or additives to minimize the fines to <7% in the pellets in a continuous pelleting system (laboratory scale flat die and ring die pellet mill). Current value of fines (<425 microns) in pellets is about 35-38 % by weight.

Table 9: Particle attrition targets

	FY19 (go-no-go)	FY-19	FY-20
Particle attrition targets (%)	14	10.5	7

- Characterize the pellets produced using CT-Scan, FTIR, BET, solid state NMR and Focused Ion Beam (FIB) to understand the relationship between process variables and feedstock properties on the particle binding behavior and chemical bonds formed during pelleting process.
- Understand the pellet breakage mechanism during crumbling of pellets.

Summary

Overview

- Fractional milling, high moisture pelleting and low temperature drying technologies developed in this project reduced corn stover pellet production cost by 65%.
- Major barrier identified by INL and NREL collaborative work to use pelleted feedstock is particle attrition (fines generation) during preprocessing. Fines result in mass loss during conversion and reduce yields.

Approach

- Understand the effect of pelleting process variables (compressive force, preheating temperature, residence time) and feedstock properties (moisture content and screen size of the grind) on the particle agglomeration behavior.

Technical Accomplishments

- Reduced the particle attrition to about 12% in pelleted corn stover by optimizing compressive force, preheating temperature residence time, moisture content and screen size of the grind.
- Used SEM, CT-scan and FIB to understand particle behavior during pelleting.

Relevance

- Addresses the biomass quality barriers, production of on-spec material, increased yields and profitability for biorefineries.
- The preprocessing solutions developed in this project can be transferred to 2nd generation biorefineries, to improve operational reliability and to make pelleting a cost-effective option in the U.S.

Future work

- Understand the effect of a) grinder type (hammer mill versus rotary shear) b) fines separation (typically fines are rich in ash content) c) natural and chemical binders on the particle behavior in a laboratory scale pellet mill.

References used in this presentation

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Tumuluru, J.S., Wright, C.T., Hess, J. R. and Kenney, K. L. 2011. A review of biomass densification systems to develop uniform feedstock commodities for bioenergy application, 5, (6), 683–707.

Additional slides

3-Technical Accomplishments

Table 4: Models for the particle attrition, particle dimensions and unit density for 1/4 inch grind.

Pellet properties	Equation	R^2
Particle attrition (%)	$26.82540 - 1.41808x_1 + 0.063191x_2 + 0.057800x_1^2 + 0.000112x_2^2 - 0.003644x_1x_2$	0.71
Unit density (kg/m3)	$1296.465 - 0.428504x_1 - 0.784300x_2 + 0.0450x_1^2 + 0.004780x_2^2 - 0.002928x_1x_2$	0.66
Geometric mean particle length (mm)	$1.068261 + 0.015504x_1 - 0.001616x_2 - 0.000588x_1^2 - 0.000002x_2^2 + 0.000081x_1x_2$	0.69
d10	$0.222295 + 0.012995x_1 - 0.000497x_2 - 0.000519x_1^2 - 0.000001x_2^2 + 0.000027x_1x_2$	0.75
d50	$0.78078 + 0.008783x_1 - 0.001300x_2 - 0.000390x_1^2 - 0.000001x_2^2 + 0.000060x_1x_2$	0.68
d90	$2.626540 - 0.023625x_1 - 0.001822x_2 + 0.001143x_1^2 - 0.000013x_2^2 + 0.000183x_1x_2$	0.68
Note: x_1: Feedstock moisture content; x_2: Residence time (min); R^2: Coefficient of determination		

3-Technical Accomplishments

Table 5: Models for the particle attrition, particle dimensions and unit density for 7/16 inch grind.

Pellet properties	Equation	R^2
Particle attrition (%)	$13.71364 - 0.250182x_1 + 0.0012281x_2 + 0.017750x_1^2 + 0.000327x_2^2 - 0.002139x_1x_2$	0.66
Unit density (kg/m3)	$1251.91 + 1.883645x_1 - 0.017462x_2 - 0.065000x_1^2 + 0.001094x_2^2 - 0.002555x_1x_2$	0.93
Geometric particle size (mm)	$2.067707 - 0.071730x_1 - 0.004429x_2 + 0.002230x_1^2 + 0.000013x_2^2 + 0.000049x_1x_2$	0.50
d10	$0.497983 - 0.011946x_1 - 0.001100x_2 + 0.000185x_1^2 + 0.0000001x_2^2 + 0.000052x_1x_2$	0.51
d50	$1.622038 - 0.055892x_1 - 0.004958x_2 + 0.001741x_1^2 + 0.000017x_2^2 + 0.000053x_1x_2$	0.44
d90	$4.122628 - 0.161192x_1 - 0.003200x_2 + 0.006232x_1^2 + 0.000023x_2^2 - 0.000204x_1x_2$	0.66
Note: x_1: Feedstock moisture content; x_2: Residence time (min); R^2: Coefficient of determination)		

3-Technical Accomplishments

Table 6: Response surface models based on central composite design.

Pellet properties	Equation	R^2
Particle attrition (%)	$65.44824 - 0.604109x_1 - 6.39153x_2 - 0.376453x_3 + 0.011764x_1^2 + 0.372273x_2^2 + 0.002685x_3^2 + 0.20000x_1x_2 - 0.008188x_2x_3 - 0.002x_3x_1$	0.66
Unit density (kg/m3)	$920.1341 - 4.79773x_1 + 53.72727x_2 - 1.34977x_3 - 0.310909x_1^2 - 2.69318x_2^2 - 0.003182x_3^2 - 1.72500x_1x_2 + 0.2375x_2x_3 + 0.255x_3x_1$	0.94
Geometric mean particle length (mm)	$-0.982904 + 0.014619x_1 + 0.246677x_2 + 0.023630x_3 - 0.000032x_1^2 - 0.013792x_2^2 - 0.000149x_3^2 - 0.000932x_1x_2 + 0.000249x_2x_3 + 0.000075x_3x_1$	0.73
d10	$-0.330004 + 0.007121x_1 + 0.091413x_2 + 0.004548x_3 - 0.000153x_1^2 - 0.005491x_2^2 - 0.000034x_3^2 - 0.000173x_1x_2 + 0.0001222x_2x_3 + 0.000024x_3x_1$	0.62
d50	$-1.31316 + 0.007467x_1 + 0.305535x_2 + 0.018110x_3 + 0.000329x_1^2 - 0.015928x_2^2 - 0.000111x_3^2 - 0.001409x_1x_2 + 0.000134x_2x_3 + 0.000063x_3x_1$	0.76
d90	$-0.012932 + 0.037206x_1 + 0.155979x_2 + 0.040650x_3 - 0.000136x_1^2 - 0.008548x_2^2 - 0.000234x_3^2 - 0.000879x_1x_2 + 0.000278x_2x_3 - 0.000038x_3x_1$	0.65
Durability (%)	$96.65539 - 0.274850x_1 + 1.005906x_2 - 0.012716x_3 + 0.004499x_1^2 - 0.058667x_2^2 + 0.000081x_3^2 + 0.0006x_1x_2 + 0.000133x_2x_3 + 0.001273x_3x_1$	0.73
Note: x_1: Feedstock moisture content; x_2: Compressive force (kN); x_3: preheating temperature (°C)		

3-Technical Accomplishments

CT-Scan analysis
Agglomerate Extraction Methodology

Definition of Two types of Cracks:

- **Inter-**: those that exist between agglomerates
- **Intra-**: those that exist within agglomerates

Key Assumptions for Agglomerate Extraction:

1. Inter-agglomerate cracks define boundaries between agglomerations
2. Agglomerates may have some intimate contact with an adjacent agglomerate, but enough cracking exists between agglomerates that all inter-agglomerate cracks are part of a larger crack network.

Methodology for Extraction:

1. Isolate inter-agglomerate cracks with connectivity algorithm
2. Use surfaces as seed point for Euclidean distance transform

   -Think of cracks in this transformed space as mountain ridgelines and the space between as artificial valleys
3. Use watershed method to fill in borders of agglomerates between cracks.

   -A sophisticated version of "connect the dots". Helps to connect the dots between cracks isolating solid material within
4. With borders established assign indices to interior voxel (3D pixels) and perform further desired analysis on individual agglomerates

Publications, and presentations for 1.2.1.2 project are given in the following slides

Publications and presentations

Books published

1. Editor: **Tumuluru, J.S** (Ed.) (2017). Biomass Volume estimation and valorization for energy. InTech, ISBN 978-953-51-2938-7, Print ISBN 978-953-51-2937-0, 503 Pages 514.
2. Editor: **Tumuluru, J.S** (Ed.). (2018). Biomass Preprocessing and Pretreatments for Production of Biofuels. Boca Raton: CRC Press. ISBN 9781498765473 - CAT# K29080, 458 Pages.

Guest Editor for special issues

1. Energies Journal: Special Issue: Woody Biomass for Bioenergy Production.
2. Bioengineering Journal: Special Issue: Advances in Biomass Size Reduction.

Patent

1. **Jaya Shankar Tumuluru**. Systems and methods of forming densified biomass. US14324902 (Pending).

Book chapters

1. **Jaya Shankar Tumuluru**. 2018. Why biomass preprocessing and pretreatments. In Book: Tumuluru, J. (Ed.). (2018). Biomass Preprocessing and Pretreatments for Production of Biofuels. Boca Raton: CRC Press., ISBN 9781498765473 - CAT# K29080, 2018.

Publications and presentations

Book chapters

2. **Jaya Shankar Tumuluru** and Neal Yancey. 2018. Conventional and Advanced Mechanical Preprocessing Methods for Biomass: Performance Quality Attributes and Cost Analysis. In Book: Tumuluru, J. (Ed.). (2018). Biomass Preprocessing and Pretreatments for Production of Biofuels. Boca Raton: CRC Press., ISBN 9781498765473 - CAT# K29080, 2018.
3. Amit Khanchi, Bhavna Sharma, Ashokkumar Sharma, Ajay Kumar, **Jaya Shankar Tumuluru** and Stuart Birrell. 2018. Effects of Mechanical Preprocessing Technologies on Gasification Performance and Economic Value of Syngas. In Book: Tumuluru, J. (Ed.). (2018). Biomass Preprocessing and Pretreatments for Production of Biofuels. Boca Raton: CRC Press., ISBN 9781498765473 - CAT# K29080, 2018.
4. **Jaya Shankar Tumuluru**. 2018. Thermal Pretreatment of Biomass to make it Suitable for Biopower Application. In Book: Tumuluru, J. (Ed.). (2018). Biomass Preprocessing and Pretreatments for Production of Biofuels. Boca Raton: CRC Press., ISBN 9781498765473 - CAT# K29080, 2018.
5. Ankita Juneja, Deepak Kumar and **Jaya Shankar Tumuluru**. 2018. Hydrothermal Liquefaction—A Promising Technology for High Moisture Biomass Conversion. In Book: Tumuluru, J. (Ed.). (2018). Biomass Preprocessing and Pretreatments for Production of Biofuels. Boca Raton: CRC Press., ISBN 9781498765473 - CAT# K29080, 2018.

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Publications and presentations

Book chapters

1. C. Luke Williams, Rachel M. Emerson and **Jaya Shankar Tumuluru** (2017). Biomass Compositional Analysis for Conversion to Renewable Fuels and Chemicals, Biomass Volume Estimation and Valorization for Energy, Editor: Dr. Jaya Shankar Tumuluru, InTech, ISBN 978-953-51-2938-7, Print ISBN 978-953-51-2937-0, DOI: 10.5772/65777.

Peer Reviewed Publications

1. (Invited) **Jaya Shankar Tumuluru**. 2018.Effect of Moisture Content and Hammer Mill Screen Size on the Briquetting Characteristics of Woody and Herbaceous Biomass, KONA Powder and Particle Journal, 36, 241-251.
2. **Jaya Shankar Tumuluru**. 2018. Effect of pellet die diameter on density and durability of pellets made from high moisture woody and herbaceous biomass. Carbon Resources Conversion, 1, 44-54.
3. Edmunds CW, Reyes Molina EA, André N, Hamilton C, Park S, Fasina O, Adhikari S, Kelley SS, **Tumuluru JS**, Rials TG and Labbé N. 2018. Blended Feedstocks for Thermochemical Conversion: Biomass Characterization and Bio-Oil Production From Switchgrass-Pine Residues Blends. Front. Energy Res. 6:79.
4. **Tumuluru, J.S.**, Heikkila, D.J. 2019. Biomass Grinding Process Optimization Using Response Surface Methodology and a Hybrid Genetic Algorithm. *Bioengineering*, *6*, 12.

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Publications and presentations

Milestones

1. Tumuluru, J. S., Yancey, N., Conner, C.C., Egan, E., Dee, M., Landon, C. and Scouten, D. 2017. PDU scale demonstration of integrated processing of biomass at >25% moisture content which includes both fractional milling and high moisture densification and further drying to show at least 50% reduction on preprocessing cost compared to the FY13 herbaceous biomass SOT and meet the pellet quality targets (bulk density >35 lb/ft3 and durability >97.5%) (BETO annual milestone).

2. Tumuluru, J. S., Conner, C.C., Dee, M. and Scouten, D. 2017. Demonstrate technical feasibility of pelleting high moisture switchgrass and pine blends and identify process conditions to achieve pellet quality requirements of bulk density >30 lb/ft3 (>480 kg/m3), and durability > 95% in a flat die pellet mill. This will involve analyzing the experimental data developed to identify the statistical significance of the process variables, evaluating the pellet quality attributes; optimizing the response surface models for pellet properties (pellet moisture content, bulk density, and durability) and specific energy consumption to meet the quality targets (BETO annual milestone).

Presentations

1. Jaya Shankar Tumuluru, Eric Fillerup, and Josh Kane. 2019. Particle attrition during pelleting of corn stover: impact of moisture content, residence time and particle size. 9th International Granulation Workshop, 26th - 28th June 2019, Lausanne, Switzerland (accepted for oral presentation).

2. (Session keynote speaker) Jaya Shankar Tumuluru. 2018. Advances in biomass preprocessing, pretreatments and control systems. 15th International Symposium on Bioplastics, Biocomposites and Biorefining (ISBBB 2018), Circular Economy for Bioproducts Innovation, July 24 - 27, 2018 || Guelph, Ontario, Canada.

3. Jaya Shankar Tumuluru and Neal Yancey. 2018. Size reduction and pelleting of corn stover: Impact of moisture content, length to diameter ratio and screen size on pellet properties and energy consumption. 2018 International Biomass Conference and Expo, April, 16-18, Cobb Galleria Center, Atlanta Georgia.

Publications and presentations

3. Jaya Shankar Tumuluru, Molly O'Brien and Neal Yancey. 2018. Optimizing the biomass grinding process using response surface methodology and hybrid genetic algorithm. American Society of Agricultural and Biological Engineers Annual Meeting, paper number: 1800746, Detroit, Michigan, USA, July, 30-1st August, 2018.
4. Neal Yancey, Jaya Tumuluru, William Smith. 2018. Using Air Separation to Control Particle Size and Reduce Total Ash Content During Corn Stover Processing. American Society of Agricultural and Biological Engineers Annual Meeting, paper number: 1801149, Detroit, Michigan, USA, July, 30-1st August, 2018.
5. Neal Yancey, Jaya Tumuluru, Patrick Bonebright, Quang Nguyen. 2018. Automation in Controlling Particle Size in Corn Stover Grinding. American Society of Agricultural and Biological Engineers Annual Meeting, paper number: 1801156, Detroit, Michigan, USA, July, 30-1st August, 2018.
6. Jaya Shankar Tumuluru, and Kristen Baker. 2018. Thin layer drying characteristics of high moisture woody and herbaceous biomass pellets in a laboratory scale grain dryer. American Society of Agricultural and Biological Engineers Annual Meeting, paper number: 1800744, Detroit, Michigan, USA, July, 30-1st August, 2018.
7. Jaya Shankar Tumuluru, Craig C Conner, and Ty Dansie. 2017. Impact on moisture content, particle size on the physical properties and energy consumption of corn stover, switchgrass, and lodgepole pine blend briquettes. American Society of Agricultural and Biological Engineers Annual Meeting, Spokane, Washington, USA, July, 17-19.

Publications and presentations

Presentations

8. (Invited) Jaya Shankar Tumuluru and Richard J. Hess. 2017. Biomass preprocessing to produce conversion ready feedstocks. 3rd Brazilian Bioenergy Science and Technology Conference (BBEST 2017), October, 17-19, Campos do Jordao, Brazil.
9. (Invited) Jaya Shankar Tumuluru. 2017. Advances in mechanical preprocessing of high moisture biomass: Impact on quality, cost, and performance. 3rd Brazilian Bioenergy Science and Technology Conference (BBEST 2017), October, 17-19, Campos do Jordao, Brazil.
10. (INVITED) Jaya Shankar Tumuluru. 2017. Advances in mechanical preprocessing of biomass: performance, quality attributes, and cost analysis". Presented to Ministry of Agriculture, Food and Rural Affairs and other provincial as well as federal departments staff, Guelph Ontario, on 2nd June 2017.

Awards

- Dr. Tumuluru got 2018 Asian American Engineer of the year Award
- High moisture pelleting process developed by Dr. Tumuluru was 2018 R & D 100 Award finalist.

Publications and presentations

PHD thesis external examiner

Dr. Tumuluru acted as external examiner for two PhD thesis in 2018.

- North-West University, Potchefstroom, Zambia.
- Vellore Institute of Technology, Vellore, India.

Commercialization

Fractional milling and high moisture pelleting process got interest from industries

a) Fulcrum Bioenergy Inc was selected for DOE, FCIC-DFO award for demonstrating of fractional milling, high moisture pelleting and low temperature drying of municipal solid waste, if successful the technology will be implemented at Fulcrum preprocessing facility.

b) Submitted a technology commercialization funding proposal with Lignetics Inc., which is a major pellet producer in the United States to test fractional milling, high moisture pelleting low temperature drying of biomass.

c) Cogent Technologies (testing high moisture pelleting of MSW for plasma gasification).

Responses to Previous Reviewers' Comments

Comment: Main goal is to reduce the cost of preprocessing biomass by 50% of the 2013 SOT. This seems a little generous. Is there an updated SOT that might be more relevant for the future? Update target after 2017 goal.

Nice project with clear technical and management approach.

Lab scale to pilot to commercial scale to meet quality and cost targets. It is unclear the significance of the bulk density and durability requirements. Please explain their significance in terms of cost adjustment.

Reply: Qualitatively there is a benefit to pellet biomass as higher density and durability will improve the flow, handling and storage characteristics. Logistics models developed by INL has related bulk density to transportation cost. But we are developing reliability models which can relate the pellet properties such as bulk density and durability to operational reliability and operational reliability to cost. Since peer review we completed development of operational reliability models which will be further developed to understand the significance of pellet properties in terms of cost.

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Responses to Previous Reviewers' Comments

Comment: My overall impressions of this work is that it provides predictive relationships that enable pelleting of higher moisture biomass while minimizing energy use and cost. Publically available information supports the industry and saves them costly trial and error. Examination of the potential for pelleting high moisture biomass is part of the BETO strategy of developing pathways for handling feedstocks as they are available from the field. This project has made considerable progress on evaluating the state of the technology for pelletizing high moisture feedstock. Significant energy savings over conventional pelleting operations were identified, but further validation of the claimed energy requirement in grinding high moisture feedstock using large screen-size is needed. The ability to storing high moisture pellets without mold/fungal growth will need to be demonstrated. The project worked with reconditioned materials at 30% moisture. Real world situations will result in 30-50% moisture material, so this project should expand the range of material (type, moisture level, quality) considered. This project addresses BETO's goals, and should be continued.

Responses to Previous Reviewers' Comments

Reply: This project has looked in understanding the effect of bigger screens in stage-1 and 2 grinders. The results indicated that bigger screen size of 3 inch in stage-1 and bigger screen size in stage-2 (7/16 inch) has reduced the grinding energy by about 65% compared to conventional stage-1 and stage-2 grinding process fitted with 2 inch and 1/4 inch screens. This will be verified in the integrated demonstration of the process at commercial scale, which is scheduled at the end of the project.

High moisture pelleting process tested in this project makes biomass drying optional. If the pellets has to be stored for short time and has to be transported to short distances drying can be avoided. In case if the pellets has to be stored for long durations and has to be transported to long distances then low temperature drying technologies such as grain or belt dryer can be used to reduce the final moisture content of the pellets to <9% for safe storage.

The studies on high moisture pelleting was done for both reconditioned material as well as using high moisture bales. The trends observed for both the material in terms of product properties and energy consumption were similar.Our integrated design report deal with biomass bales at 50% moisture content (Kenney et al., 2013). The storage task (W.B.S: 1.2.1.1) work is focused on finding cost-effective means to deliver corn stover bales to biorefinery at a maximum moisture content of 30%, via storage conditions that either actively or passively reduce the moisture content over time in storage. Therefore preprocessing has to deal with biomass bales at 30% moisture content.

Responses to Previous Reviewers' Comments

Comments: Lots of results and progress made to meet price reduction goals. However, this is an existing process used in forestry therefore it doesn't seem like new technology. Please clearly explain the limits to cost reduction if new technology is not developed and this framework continues. Reduced grinding energy by 65% by increasing screen size to 7/16" and adding fractional milling. Cost target also met.

The results show only test moisture content up to 30%. Lots of bales greater than 1 year old will have moisture content greater than 30%. These higher moisture content bales need to be addressed. Why stop at 30% moisture content? Nice scale up from lab to 1ton per hour facility. Interesting results on pre-heating temperature of 40-60°C to help activate biomass. It seems relevant to know if this result hold true for other herbaceous biomass or only corn stover.

Reply: Pelleting is not a new technology, but we developed a new method which operates at different process parameters, and order of unit operations to produce pellets. This new process was demonstrated at pilot scale (1 ton/h). Technoeconomic analysis of the process indicated that the cost of pelleting is reduced by 62 % compared to current technology used in forestry industry.

Our integrated design report deals with biomass bales at 50% moisture content (Kenney et al., 2013). The storage task (W.B.S: 1.2.1.1) work is focused on finding cost-effective means to deliver corn stover bales to biorefinery at a maximum moisture content of 30%, via storage conditions that either actively or passively reduce the moisture content over time in storage. Therefore preprocessing has to deal with biomass bales at 30% moisture content.

Responses to Previous Reviewers' Comments

Comment: The trade-offs of pelletization should be clearly define to establish the viability of the concept on different conversion processes and biomass types. The benefits are dependent on the conversion process and the type of biomass.
Reply: We have done testing of pellets in fast pyrolysis and biochemical conversion. This testing has identified that the fines generated drying pelleting can be a problem. Future work is aimed to address this issue which will involve additional testing to understand the viability of the concept on different conversion processes and biomass types.

Comment: The project accomplishments are highly relevant to BETO's goals and objectives related to preprocessing of agricultural residues and energy crops. The project success has also attracted additional funding sources from other BETO projects. However, it would be helpful for reviewers' to distinguish the accomplishments from this project, while addressing how the issues not relevant to this project are addressed in other BETO projects.
Reply: Integrated design report published includes from harvesting of biomass to insertion of biomass to throat of conversion facility. This can be found in the following reference Kenney et al. (2013). The major accomplishments of this project is reduction of the preprocessing cost by 50% compared to current state of technology as explained in the design report to support the DOE feedstock cost of $84/dry ton. Cost reduction of unit operations are being addressed by other projects in the INL portfolio.

Responses to Previous Reviewers' Comments

Comment: The future works are well outlined to meet the BETO technical objectives. The team should further engage with Wood Pellet Institutes to diversify and sell the high moisture densification concepts to seek feedback and increase market penetration for commercialization. The overall energy savings and consumption due to high moisture should be clearly addressed to pitch the concept to the bioenergy industries. The issues related to storage of high moisture pellets should be further addressed.

Reply: BETO has selected high moisture pelleting process to fund under Energy I-Corps program. The main aim of this program is to talk with industries and understand the value proposition of the process developed. In this program we have interviewed about 86 companies which includes wood pellet producers, biorefineries (both thermochemical chemical and biochemical conversion) and feed industries. Many industries has shown keen interest in this process and has indicated that they are interested in collaborating to test their product. We are working with a local company in Idaho Falls in turning their high moisture alfalfa into pellets.