# DOE Bioenergy Technologies Office (BETO) 2021 Project Peer Review

# **1.2.1.2 Biomass Size Reduction, Drying and Densification**

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Date: 03/12/2021 Feedstock Technologies Program

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# **Overview**

#### **Particle System Limitation**

a) Most herbaceous biomass and low-cost carbon resources (e.g., forest residues and municipal solid waste) lack a three-dimensional format, which create feeding, handling, and storage issues.
b) Particles during feeding and handling agglomerate, aggregate, or suffer attrition.
c) Particle system often does not scale.



Bridging of ground corn stover in storage bins and drop chute.

**Why Densification?** Converts diverse forms of **dr** biomass into a commodity like product with better feeding, handling, storage and transportation properties.

#### **Background:**

- In FY-2017, the project developed advanced preprocessing technologies (i.e., fractional milling, high-moisture pelleting, and low-temperature drying) and reduced the pellet production cost by 65% as compared to conventional method followed by the industry.
- In FY-2017, this project contributed to the BETO feedstock cost target of \$84/dry ton by reducing the preprocessing cost by 50% as compared to 2013 state of technology (SOT).



Jamming of ground corn stover in a conveyor.

# **Overview**

Several barriers (e.g., fines, particle morphology, microstructure) still exist to deploy pelleting technology for biofuels and bioproducts applications.

#### 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 the particle attrition that results in 35-38% fines (<400 microns) during grinding and pelleting of corn stover are not recovered during biochemical conversion.

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

**End of project goal (09/31/2020):** Reduce particle attrition or fines (<400 microns) in corn stover by 80% (e.g., less than 7% fines) compared to conventional pelleting process performance which is about 35-38%.

#### Relevance

- Project addresses the biomass barriers such as density, feeding, handling, storage, and quality. Also helps to produce on-spec material, with increased yields and profitability for biorefineries.
- The preprocessing solutions developed in this project can be transferred to 2nd generation biorefineries, to improve operational reliability.

# **1-Management**

- This project has gone through DOE, Merit Review in 2017 and 2020.
- Participate in BETO, Feedstock Technologies (FT) 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 closely with INL analysis team and update the particle attrition targets achieved in the project.
- Validate the processes developed in this project by peer review publications and conference presentations.

Project Team: Agricultural, mechanical, chemical, and material engineers; plant physiologist; and chemist.

#### **Critical success factors**

Optimize the grinding and pelleting process conditions and feedstock properties to minimize the particle attrition (fines <400 microns) to meet the established target of <7% in the corn stover pellet.

#### **Risks**:

- Particle attrition is an inherent characteristic in current preprocessing activities and the ability to affect it is limited without major process and technology improvements.
- Meeting the aggressive particle attrition targets (e.g., 80% reduction compared to conventional pelleting process).
- Unknown effectiveness of grinding and pelleting process variables on fines generation of corn stover fractions.

Mitigation: Conduct fundamental research and use advanced imaging techniques to understand the particle binding mechanism during pelleting.

# 2-Approach

**Hypothesis**: Particle residence time, pelleting process variables, and material properties impact the particle attrition or fines, particle morphology, and microstructure formation in pellets.



### Impact of pressure on particle binding:

- Pressure increases the contact area between the particles.
- Moisture in the biomass helps particles to agglomerate.
- Chemical reactions in the biomass results in sintering, solidification, hardening of the binder, and helps particles to agglomerate.

#### Particle binding mechanism during densification

This project specifically address barriers, such as: (a) biomass quality, (b) biomass physical state alteration, and (c) biomass material handling and transportation.

# 2-Approach

#### Pelleting system

A single pellet press is used for doing the pelleting tests.

#### **Process variables tested**

- Grinder type: (a) hammer mill (7/16-inch screen size); and (b) rotary shear grinder fitted with a 2 mm cutter
- Compressive force (kN)
- Residence time (sec.)
- Moisture content (%, w.b.)

#### **Feedstock tested**

- Hammer mill and rotary shear ground corn stover.
- Corn stover fractions (e.g., cob, stalk, husk and leaf)

#### **Methods**

ISO and ASABE standards are used to measure the unit density and particle size distribution in corn stover pellets.

#### Table 1: Particle attrition (percent fines <400 microns) targets for corn stover pellets.

	FY-17 (conventional corn stover pellet, based on	FY-18	FY19	FY-19	FY-20
	INL & NREL collaborative work)				
% Fines	35-38	21	14	10.5	<7



Single pellet press.

# **3-Impact**

- This project specifically addresses a recognized barrier (fines) in corn stover pellets, which effects the downstream conversion yields and supports the bioeconomy.
- Converts diverse forms of herbaceous, low-cost carbon resources (forest residues and municipal solid waste) into a consistent, high-quality commodity product to efficiently store, feed, handle, and transport to biorefineries.
- The outcomes specifically address biomass particle attrition issues during preprocessing, as this represents a major impediment for using pelleted feedstock for biofuels production.
- Reduced pellet production cost and production of on-spec material, increases profitability to biorefineries.
- Preprocessing technologies developed can be used by biomass processors and designers for producing biofuels, chemicals, and bioproducts.
- Helps to achieve DOE's vision of commoditization of diverse forms of biomass at a lower cost.

# **3-Impact**

## **Industry impact**

- Currently working with Fulcrum Bioenergy, which produces jet fuels from municipal solid waste (MSW) to test advanced preprocessing technologies (fractional milling, high-moisture pelleting and low-temperature drying) to convert high-moisture and low-dense MSW to pellets.
- Currently working with Lignetics Inc, a major wood pellet producer in the US on testing advanced preprocessing technologies developed in this project for wood pellet production for biopower application.
- We have recently signed an NDA with Clean Energy Systems, California, to discuss the advanced preprocessing technologies for processing of biomass feedstocks.
- Recently discussed with Trestle Energy LLC, California, on high moisture pelleting of corn stover as they feel it can have a dramatic impact
   both for densification and for the broader supply chain.
- High moisture pelleting was an R&D Award Finalist in 2018 and 2020.
- Published a book on *Biomass Densification*, 2020, Springer Nature, Switzerland, (https://www.springer.com/gp/book/9783030 628871).



Java Shankar Tumuluru

**Biomass** 

Densification

# Biomass Densification

Systems, Particle Binding, Process Conditions, Quality Attributes, Conversion Performance, and International Standards

Authors: Tumuluru, Jaya Shankar



The impact of pelleting on particle diameter of rotary shear and hammer mill grind pellets

- For hammer milled grind, particle diameter changed between 200 to 400 microns and 650 to 1700 microns after pelleting.
- For rotary shear grind, particle diameter changed between 750 to 3300 microns after pelleting.

#### **4-Progress** Comparison of rotary shear versus hammer mill grind pellets



Fines (<400 microns) in rotary shear and hammer mill grind corn stover pellets.

Unit density of rotary shear hammer mill grind corn stover pellets.

- Minimum percent fines in rotary shear grind pellets is about 5.2%, whereas hammer mill grind pellets result in about 12.4%.
- Unit density of rotary shear grind pellets is slightly higher than hammer mill grind pellets.
- Durability of the hammer mill grind pellets was slightly higher compared rotary shear grind pellets (please see additional slides).

## Grinding and pelleting studies on cob, stalk, husk, and leaf fractions



Wiley mill used for grinding studies

#### Chemical composition

- Cob has lowest ash content (about 1%), while leaf has the highest (about 8%)
- Husk has the lowest lignin content (about 12%), while stalk has the highest (about 22%).

#### **Process variable tested**

Grinder speed: 20-60 (Hz) Grind moisture content: 10-20 (%, w.b.) Screen size: 12 mm

#### Grind properties measured

- a) Percent fines (<400 microns)
- b) Particle size distribution
- c) Geometric mean particle length
- d) Energy consumption.

#### Grinding energy (kWh/ton)

Cob was in the range of 9-14 kWh/ton Stalk was in the range of 73-128 kWh/ton Husk was in the range of 68-80 kWh/ton Leaf was in the range of 47-74 kWh/ton.

#### **Particle attrition (fines <400 microns)**

- Cob was in the range of 0.9-1.4%
- Stalk was in the range of 2.7-4.1%
- Husk particle attrition values are in the range of 3.54-6.9%
- Leaf values are in the range of 4.38-8.5%.

## Trends of fines (<400 microns) in the ground corn stover fractions: cob<stalk<husk<leaf

# **4-Progress** Pelleting tests on corn stover fractions

Corn stover fractions that result in minimum fines during grinding were used for pelleting studies

#### Pelleting process variables

- Compressive force: 11-13 kN
- Preheating: 90-110 (°C)
- Grinder screen: 12 mm
- Residence time: 45 sec.



Cob, stalk, husk, and leaf fractions pellets

## Pellet properties measured

- Unit density (kg/m<sup>3</sup>)
- Particle attrition (fines <400 microns)</li>
- Particle size distribution
- Geometric mean particle length (mm)

## Pellet unit density (kg/m3)

- Maximum unit density of cob pellet: 1134 kg/m<sup>3</sup>
- Maximum unit density of stalk pellet: 1293 kg/m<sup>3</sup>
- Maximum unit density of husk pellet: 1309 kg/m<sup>3</sup>
- Maximum unit density of leaf pellet: 1315 kg/m<sup>3</sup>

#### Geometric mean particle length (mm)

Highest for cob (about 4 mm) and lowest for leaf (about 2 mm).

- Higher compressive force and preheating temperatures maximized the unit density of cob, stalk, and husk pellets
- In case of leaf, lower preheating temperature and higher compressive force are necessary to maximize unit density.

Table 2: Percent fines in the corn stover fractions after grinding and after pelleting.

Process variables	Cob	Stalk	Husk	Leaf
Compressive force	Fines (<400	Fines (<400	Fines (<400	Fines (<400
(kN)	microns) (%)	microns) (%)	microns) (%)	microns) (%)
Initial fines in the grind	0.9	2.7	3.54	4.38
CF-11kN; PHT-90°C		1.29	1.44	3.32
CF-13kN; PHT-90°C	0.52	1.97	1.25	6.98
CF-11kN; PHT-110°C	0.67	3.99	2.11	3.59
CF-13kN; PHT-110°C	0.64	3.42	3.22	2.37

• Fines in cob and husk pellets decreased after pelleting at all pelleting conditions tested

- Fines in stalk pellets increased at higher compressive force and preheating temperatures
- Higher compressive force and low preheating temperature increased the fines in leaf pellets.

Corn stover fraction pellets processed at different pelleting conditions met the established particle attrition target of <7% and can increase the sugar yields during the biochemical conversion process.

Corn stover fraction pellets with different particle attrition and chemical composition can be blended to achieve the desired particle attrition of <7% and chemical composition for the downstream conversion requirements.

## Scanning Electron Microscope, CT-scan, and Focused Ion Beam Tomography Studies on Corn Stover Fraction Pellets





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Advanced imaging techniques to understand the impact of process variables on the particle morphology in a corn stover pellet

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Advanced imaging techniques help to design pellets with desired physical properties, particle morphology, and microstructure for the downstream conversion process.



## SEM-Leaf

## Before grinding

- Leaf surface has many visible features.
- There are several "lines" of stomata (p).
- A single trichome is present (q).





## After grinding

- The "suture" pattern seen is the cell wall junction (s)
- Broken trichome is visible (t)
- Cellulose microfibril mesh is also visible (u).

## After pelleting

- Fine white and dark deposits were seen on the surface.
- Dark deposits are parallel to the direction of extrusion (v)
- Microstructure studies indicated that anatomical fractions partially retained microstructure after grinding but mostly lost after pelleting
- Pelleting creates a new microstructure.



SEM of leaf after grinding

## **CT-scan**



#### Orthogonal slices and 3D surface rendering CT-scan of cob and husk pellet.

## Table 3: Agglomerate metrics in corn stover fraction pellets.

Scan	Unit density	Surface	Total	Agglomerates/	Agglomerate
	measured	Area to	Agglomerates	cm^3	Equivalent
	(CF-13kN;	Volume			Diameter
	PHT-110°C)	(cm^-1)			(mm)
Leaf	1164	87.94	285	839.3	0.35
Cob	1190	43.82	274	836.2	0.26
Stalk	1293	79.20	303	926.7	0.38
Husk	1309	82.84	282	862.4	0.29

- Amount of surface area for a given volume was highest for leaf and lowest for cob
- Agglomerate equivalent diameter is lowest for cob and highest for stalk
- Corn stover fraction pellets produced at same process conditions has different particle morphology.
- Controlling the process conditions can help to produce pellets with desired particle morphology required for downstream conversion.

## Focused Ion Beam Tomography (FIB)



#### FIB of stalk, leaf, and cob pellet:

#### Stalk pellet

• Stalk pellet does not have definite structure, but it is very dense.

## Leaf pellet

- (a) Top volume is less dense and has tubular pores (b) bottom volume is denser
- (c) The bright area in the center of the volume which likely SiOX. **Cob pellet**
- Composed of 10–15-micron thick layers
- All the layers seem to be connected giving a bicontinuous structure.

# Table 4: Free volume in the pelletbased on watershed segmentation.

	Free volume (%)
Husk pellet	13
Cob pellet	21
Leaf pellet	8

- Free volume was highest for cob and lowest for leaf pellet
- This observation matched with unit density trends where cob has the lowest and leaf has the highest.

# Summary

- INL and NREL collaborative work identified that fines <400 microns represent a major barrier to use pelleted corn stover for biochemical conversion.
- Reduced the particle attrition from 35-38% to about <7% in pelleted corn stover and its fractions.
- Used SEM, CT-scan, and FIB to understand particle morphology and microstructure formation.
- Produced on-spec material, which can increase yields and profitability for biorefineries.
- The preprocessing solutions developed in this project can be transferred to 2nd generation biorefineries to improve operational reliability of biorefineries.

#### FY-21-23 Research work

**Objective:** Develop preprocessing solutions for high moisture low-cost carbon resources, such as forest residues and MSW biomass to meet the critical quality attributes, such as aspect ratio, porosity, moisture, and bulk density at 30%, reduced preprocessing costs compared to methods followed by the industry.

#### Mechanical preprocessing technologies that will be tested

- Rotary shear grinding
- Mechanical dewatering
- Agglomeration.

**End of the project milestone:** Optimize mechanical preprocessing systems for low-cost carbon resources to meet quality attributes, such as aspect ratio, porosity, density, and moisture content. Conduct TEA and LCA analysis to understand the cost saving and  $CO_2$  emission reduction compared to the conventional method followed by the industry.

# Quad Chart Overview (AOP Project)

### Timeline

• 10/01/2020-09/30/2023

	FY20	Active Project
DOE Funding	\$1,115,000	\$2,465,000

Project Partners (N/A)

Barriers addressed Ft-G: Feedstock Quality & Monitoring; Ft-K: Biomass Physical State Alteration, Ft-L: Biomass Material Handling and Transportation

#### **Project Goal**

Develop preprocessing solutions for high moisture lowcost carbon resources, such as forest residues and MSW biomass to meet the critical quality attributes, such as aspect ratio, porosity, moisture, and bulk density at 30%, reduced preprocessing costs compared to methods followed by the industry.

### **End of Project Milestone**

Optimize mechanical preprocessing systems for lowcost carbon resources such as forest residues and municipal solid waste to meet quality attributes, such as aspect ratio, porosity, density, and moisture content. Conduct TEA and LCA analysis to understand the cost saving and  $CO_2$  emission reduction compared to the conventional method followed by the industry.

Funding Mechanism: Feed Technologies, AOP

# **Publications and Presentations**

#### **Books published**

- **1.** Tumuluru, J. S. (2021). Biomass densification: Systems, methods, process conditions, quality attributes, and conversion performance" has been accepted by Springer for publication (In Press).
- 2. Tumuluru, J. S. (Ed.)., (2021). Woody Biomass for Bioenergy Production, Energies, MDPI publications (In press)

## **Guest Editor for Special Issues**

- 1. Energies Journal: Municipal Solid Waste Logistics and Conversion to Produce Biofuels and Bioproducts (editor: Jaya Shankar Tumuluru).
- 2. Frontiers in Energy Research: Torrefaction Pretreatment for Biomass Upgrading: Fundamentals and Technologies (Editors: Paola Brachi, Wei-Hsin Chen, Daya Ram Nhuchhen, and Jaya Shankar Tumuluru).

## Patent

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

## Awards

High moisture pelleting is an R&D 100 Award finalist in the year 2020.

Dr. Tumuluru received Idaho National Laboratory's exceptional engineering achievement award in 2020.

## **Conference Organizing Committee**

International Bioenergy & Bioproducts Conference (IBBC-2020) Conference secretary.

Moderated technical sessions for American Society of Agricultural and Biological Engineers.

Energy Systems, Vice-Chair, for American Society of Agricultural and Biological Engineers.

# **Peer Reviewed Publications**

- **1. Tumuluru, J. S.,** Yancey, N., and Kane, J., 2020, Grinding and briquetting characteristics of high moisture municipal solid waste bales. *Waste Management* (In Press).
- 2. Tumuluru, J. S., and Fillerup, E., 2020, Briquetting characteristics of woody and herbaceous biomass blends: Impact on physical properties, chemical composition, and calorific value. *Biofpr* (<u>https://onlinelibrary.wiley.com/doi/abs/10.1002/bbb.2121</u>).
- **3. Tumuluru, J. S.,** Fillerup, E., Kane, J. J., and Murrey, D., 2020, Advanced imaging techniques to understand the impact of process variables on the particle morphology in a corn stover pellet. *Chemical Engineering Research and Design*, 161, 130-145.
- 4. Herde, Z. D., Dharmasena, R., Sumanasekera, G., **Tumuluru, J. S.,** and Satyavolu, J., 2020, Impact of hydrolysis on surface area and energy storage applications of activated carbons produced from corn fiber and soy hulls. *Carbon Resources Conversion*, 3, 19-28
- 5. Aamiri, O. B., Thilakaratne, R., **Tumuluru, J. S.**, and Satyavolu, J., 2019, An 'in-situ binding' approach to produce torrefied biomass briquettes. *Bioengineering*, 6, 87.
- 6. Pandey, R., Nahar, N., **Tumuluru, J. S.**, and Pryor, S. W., 2019, Quantifying reductions in soaking in aqueous ammonia pretreatment severity and enzymatic hydrolysis conditions for corn stover pellets. *Bioresource Technology Reports*, 7, 100187.

# Presentations

- Neal Yancey and Jaya Shankar Tumuluru. 2020. Improving Forest Residue Quality Through Air Classification and Specific Gravity Separation. International Bioenergy & Bioproducts Conference (IBBC), November 2 - 4, 2020 | Atlanta, GA.
- 2. Jaya Shankar Tumuluru and Eric Fillerup. 2020. Corn stover pellet particle morphology: impact of feedstock properties and pelleting process conditions. International Bioenergy & Bioproducts Conference (IBBC), November 2 4, 2020 | Atlanta, GA
- 3. Jaya Shankar Tumuluru and Eric Fillerup. 2020. Pelleting process conditions and feedstock properties impact on the particle attrition or fines (<425 microns) and physical properties of corn stover pellets. ASABE 2020 Annual International Meeting, Monday, July 13th, 2020 Wednesday, July 15th, 2020, Virtual & On Demand.
- 4. Jaya Shankar Tumuluru. 2019. Advances in biomass mechanical preprocessing and thermal pretreatments: improvements in physical properties and chemical composition. 26th Anniversary meeting of the Bio-Environmental Polymer Society (BEPS) will be held on June 5-7, 2019 at the Clemson International Center for Automotive Research (CU-ICAR) on the campus of Clemson University in South Carolina (<u>https://cuicar.com/</u>) (Invited talk).

# **Previous Peer Review comments and Replies**

Weakness: Particle size distribution within the pellets would presumably be of interest to end users but only average particle size was reported. The fines have been reduced, but the effect on particle size distribution using larger screen sizes was not addressed.

Reply: Preference was given to other aspects of the work for this presentation over the finer details of the particle size distributions. For addressing the issue of fines, average particle size and attrition were deemed sufficient. Particle size distributions for each material will be made available in supplemental slides in the future.

Weakness: What I do not see is a discussion in future plans whether there are any go/no-go decision points, if they exist, and any discussion of a TEA or business plan for the roll out and commercialization of the technology.

Reply: Project goals were set based on purely technical concerns (driven by economics, but a technical focus). Roll out and implementation were secondary concerns to resolving the technical problems.

Weakness: If not already part of plan, consider value of aspiration or similar classification (before or after milling) of the fines prior to pelleting to reduce actual lignocellulosic material loss while still removing ash from the system.

Reply: This is in consideration with our current work addressing milling and other pre-pelleting handling. Also, implementation was a secondary concern to resolving the technical problems.

Weakness: Other challenging aspects of pellet technology are not discussed (e.g., durability & hydrophobicity). It is not clear how examinations of the internal structure of these pellets using X-ray CT, FIB, and EDS will further the stated project goal of reducing particle attrition.

Reply: Application of advanced characterization techniques allows for a better understanding of pellet structure and the impact of pelleting conditions on the pellet structure. Understanding how conditions impact structure allows other challenges (e.g., durability & hydrophobicity) to be addressed if there is a meaningful way to do so.

# **Additional Slides**

Rotary Shear grind-20% (w.b.) moisture Pellet drop test was used to 100 Hammer mill grind-20% (w.b.) moisture calcualted the durability 99.5 99 98.5 **Durability (%) Rotary shear** 98 very hammer 97.5 mill grind 97 pellets 96.5 durability 96 95.5 95 30 15 45 Residence time (sec)



7/16-inch hammer mill grind pellets

#### Surface plots

- Higher moisture in the corn stover minimized particle attrition or fines <400 microns for both hammer • mill and rotary shear grind pellets.
- In case of hammer mill grind lower residence time minimized the particle attrition •

#### **Compositional data for corn stover fractions**

Sample	% Ash	% Extractive	% Glucan	% Xylan	% Lignin	% Galactan	% Arabinan	% Acetate
Cob	1.09	4.09	31.04	29.48	20.72	1.42	3.73	3.54
Stalk	4.96	11.37	36.51	18.54	22.17	1.11	2.61	3.12
Husk	3.69	5.27	39.63	25.01	12.49	1.78	4.78	3.44
Leaf	8.04	4.56	34.33	18.42	14.17	1.42	2.92	1.42





Geometric mean particle length of particles in corn stover pellets made at different pelleting process conditions

Unit density of the corn stover fraction pellets made at different pelleting conditions.

**Note:** The properties reported are an average of three or measurements.



#### SEM of stalk before grinding



SEM of stalk after pelleting

## **SEM-Stalk**

## Before grinding

- The "flaky" tissues cover most of the surface (e)
- Vascular bundle that runs
   from the top to bottom can
   be seen (f)



## SEM of stalk after grinding

- After grinding
- Thin and fragile cell walls and large cell cavities are visible (g)
- Many plasmodesmata are visible (h)

#### After pelleting:

- There are no identifiable anatomical features remaining after pelleting
- Surface cracks and less voids are observed



## SEM of cob after grinding

## SEM-Cob

#### Cob after grinding

- Broken cell cavities in the tissues are visible (a).
- Plasmodesmata can be seen in the exposed cell cavities (b).
- Plasmodesmata are small pores that extend through the cell walls connecting adjacent cell wall cavities.
- A bacterial colony inside one of the cell cavities can be seen (C)



SEM of cob after pelleting

## Cob after pelleting

- Very few anatomical features remain in the pelleted cob.
- Few plasmodesmata after pelleting are still visible (d)
- · Less voids on the pellet surface
- Cracks are visible

## SEM-Husk



## SEM of husk before grinding



SEM of husk after pelleting

## **Before grinding**

- Hair-like structure "Tricomes" is visible (i)
- Cell wall junctions is visible (j)
- stomata is visible (k)

## After pelleting

- Some husk fibers appear to be visible in the pellet surface (o)
- Very fine white deposits are observed on the pellet surface



## SEM of husk after grinding

## After grinding

- Cell wall junctions are visible (I)
- Cellulose microfibril mesh is visible (m) (microfibrils are bundles of cellulose fibers wrapped in hemicellulose)
- A single stomata is also visible (n)