Nanofibrillated Cellulose Fibers:

Where Size Matters in Opening New Markets to Nanofiber Usage

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Nanofibrillated Cellulose Fibers:
Opening New Markets to Nanofiber Usage

- Introduction
- Regenerated / Natural Cellulose – Structure and Nanofibrillation
- Commercial Production of Cellulose Nanofibrillated Fibers
- Applications / Performance of Nanofibrillated Fibers
- Summary

Conventional Nanofiber Definition / Polymeric Fibers

- Fibers with Diameters less than About 0.5 microns

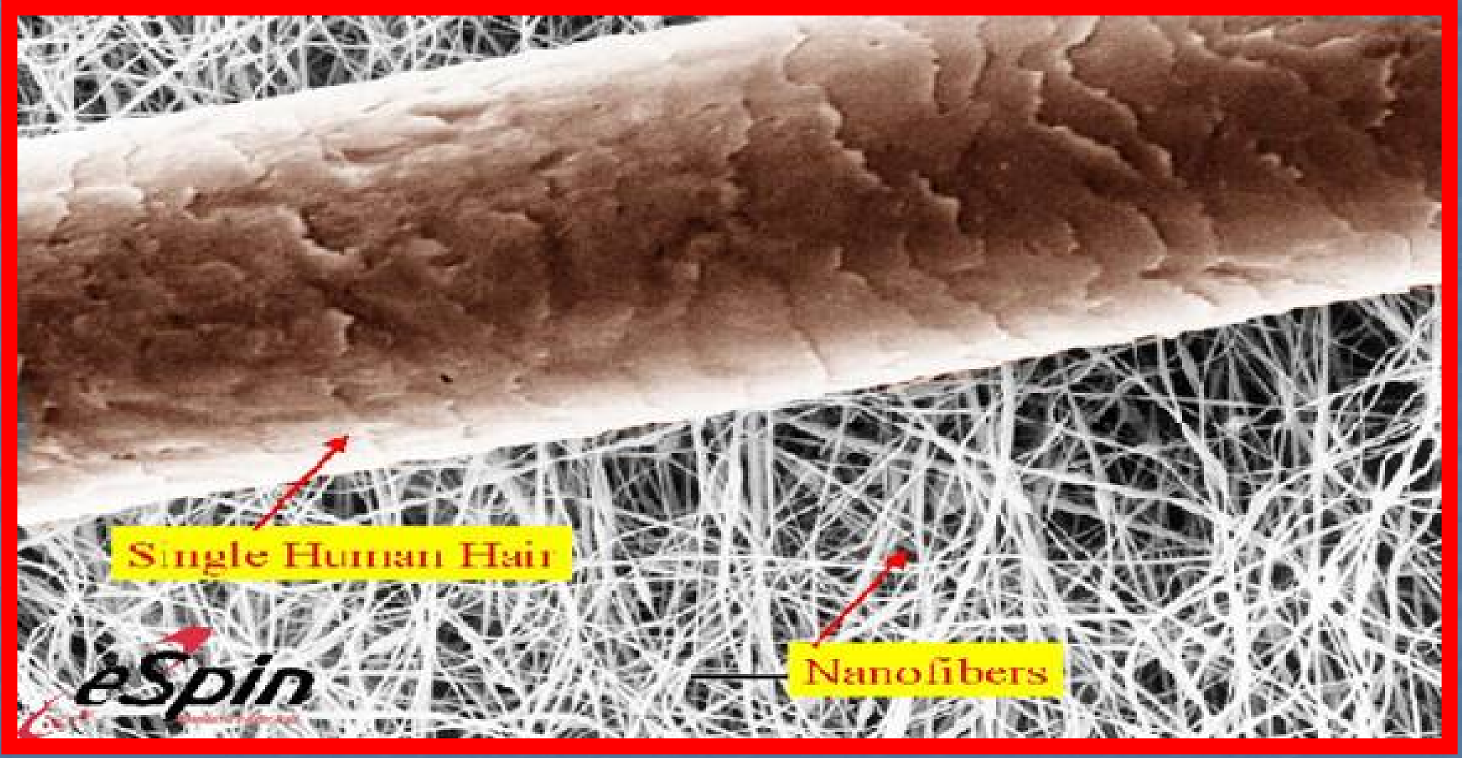
  (Consensus Not Universal with Definitions Down to < 10 Nanometers and Up to 1 Micron)

- Typical Polymeric Nanofibers Currently Produced Have Diameters Between 50 and 300 Nanometers

- Presentation Focused on Cellulosic Nanofibers Created By Nanofibrillation Technology and Produced on Commercial Scale

- Alternative "Low Cost" Nanofibers for Range of Paper Making and Other Engineered Applications

Relative Size of Nanofibers Compared to Human Hair
(20,000 to 30,000 nm)



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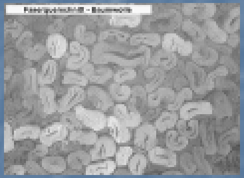
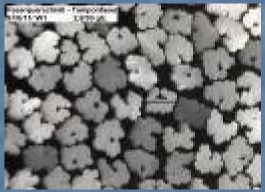
Nanofibrillation of Cellulosic Fibers (1)

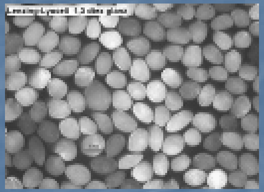
- **Natural Cellulosic Fibers**



  - Wood / Non-Wood
    - Grow with Microfibrillar Structure
    - Micro / Nano Fibrils Can be Generated Under Special Fibrillation Processing

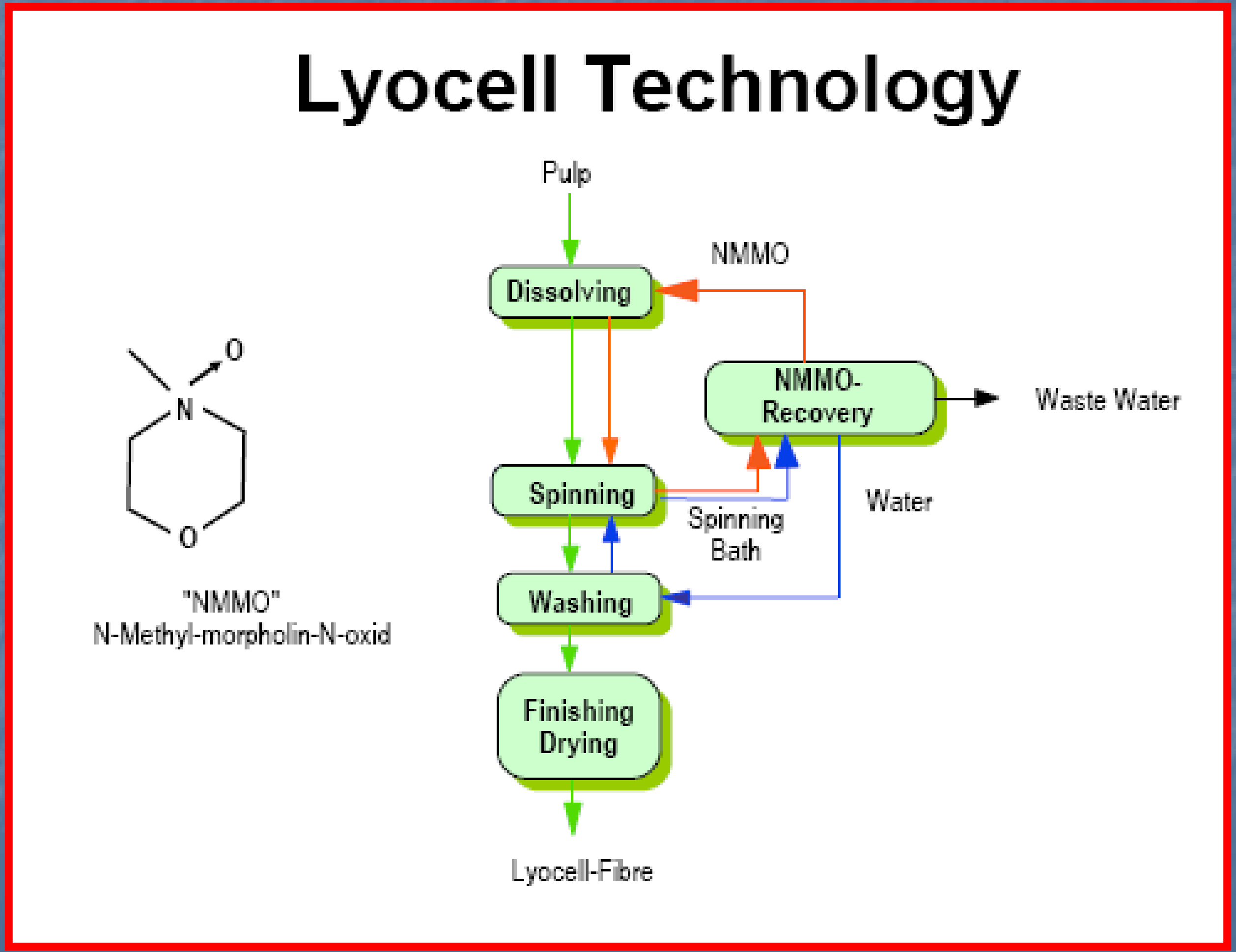
- **Regenerated Cellulose**





  - Rayon / Viscose
    - More Amorphous Structure –Generally Abrades into short lengths
    - Higher Modulus Polynosic Rayon (Tufcel) had High Fibrillation Levels
  - Lyocell
    - High Crystallinity with microfibrillar structure / similar to lyotropic rods – low lateral bonding between crystalline regions

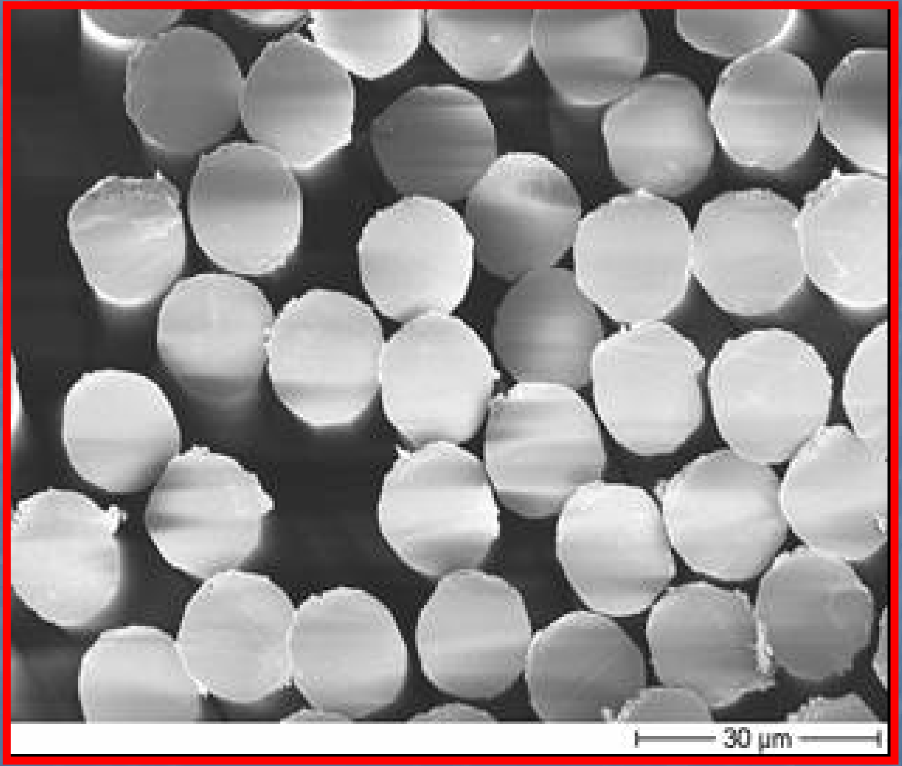
Production of Regenerated Cellulose Fibers

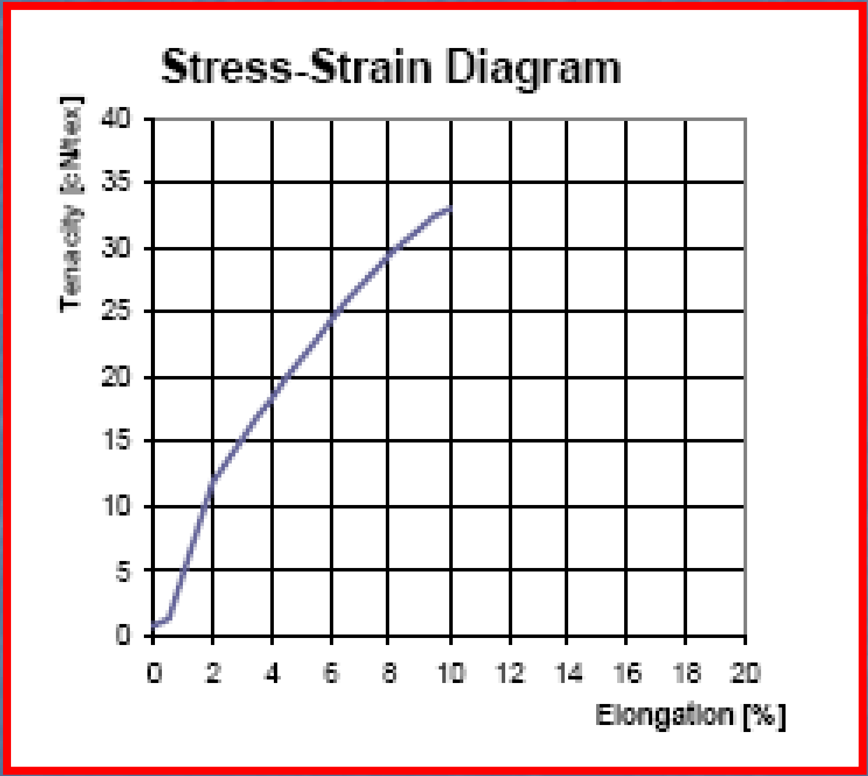


Production of Lyocell Fibers
Physical vs. Chemical Process

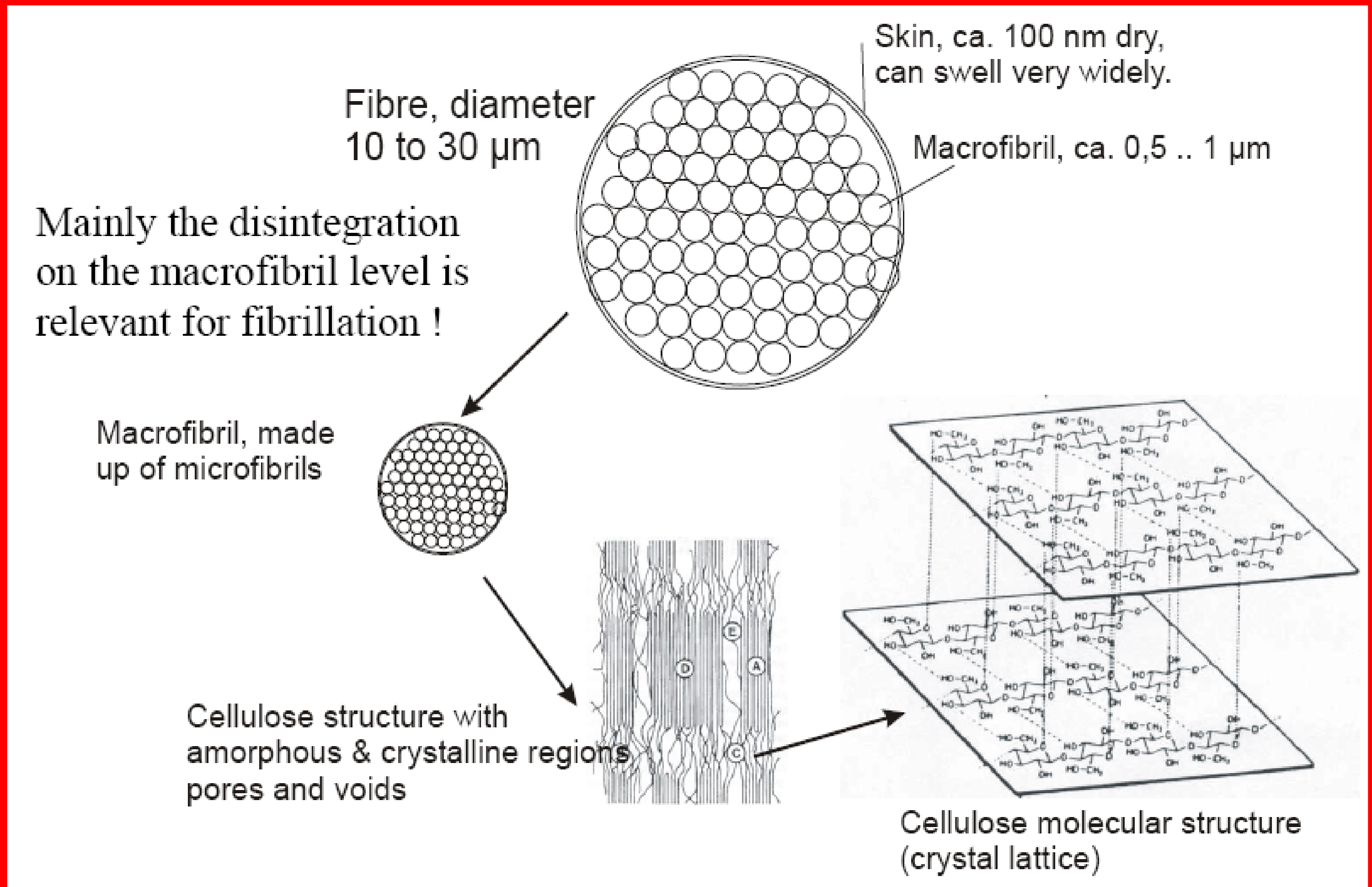
- Purified Dissolving Wood Pulp
- Amine Oxide Solvent
- Extruded (Spun) Through Spinnerets
- Continuous Filaments / Circular Cross-Section (Cut to Short – 0.5 to 8 mm-Lengths)
- Long Chain Molecules / Highly Crystalline (> 60 %) Structure

Lyocell Fiber –As Spun



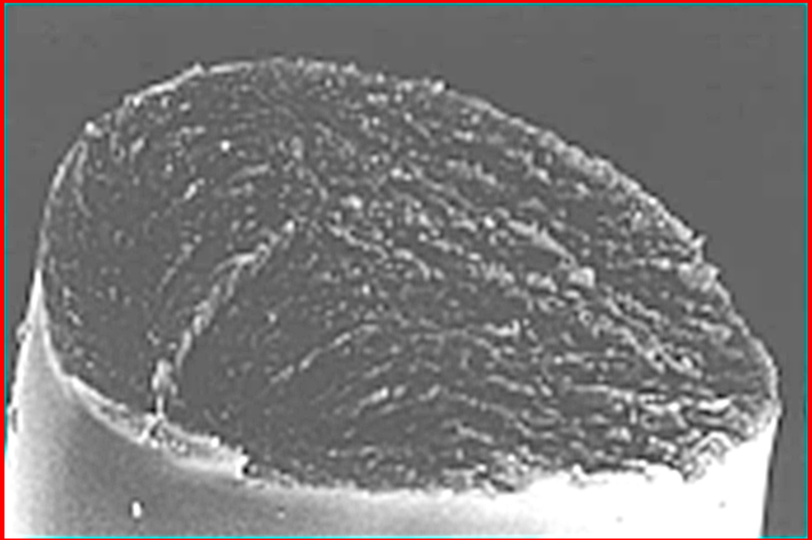


Proposed Structure at Different Dimensional Levels of Lyocell Fiber (2)



Lyocell Fiber Morphology (3) - The Ultimate "Islands in the Sea"

Structure Element	Size	Approximate number of fibrils per fibre cross-section
Nanofibril	10 nm	1,330,000
Microfibril	0.15 µm	5902
Macrofibril	0.75 µm	236
Complete fibre diameter	13 µm	(1)



Fibrillation /Splitting Mechanism to Create Lyocell Nanofibers

- Initial peeling of fibrils (macro bundles) along the fiber length of individual fibers, induced by mechanical stress, special processing / treatments

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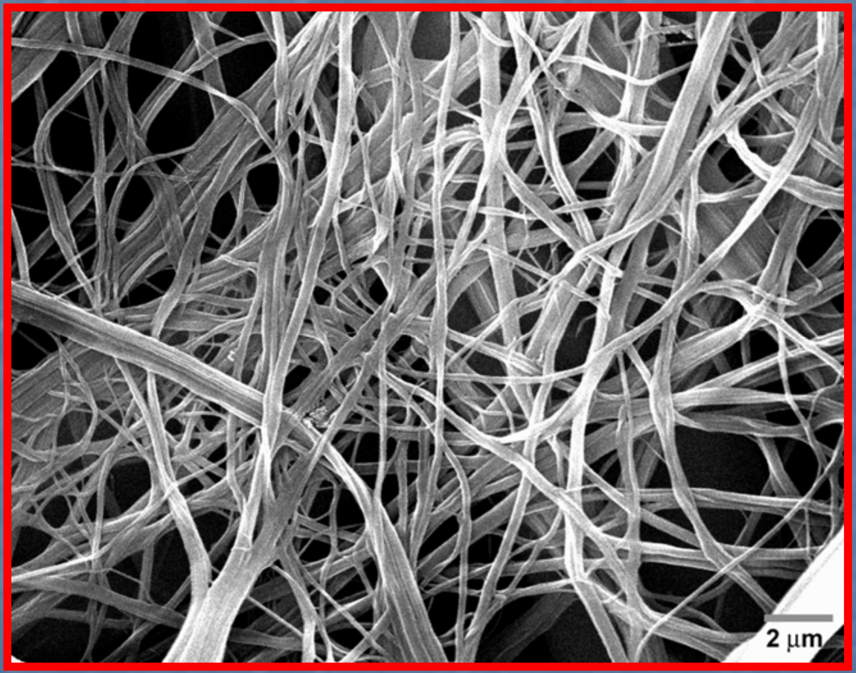


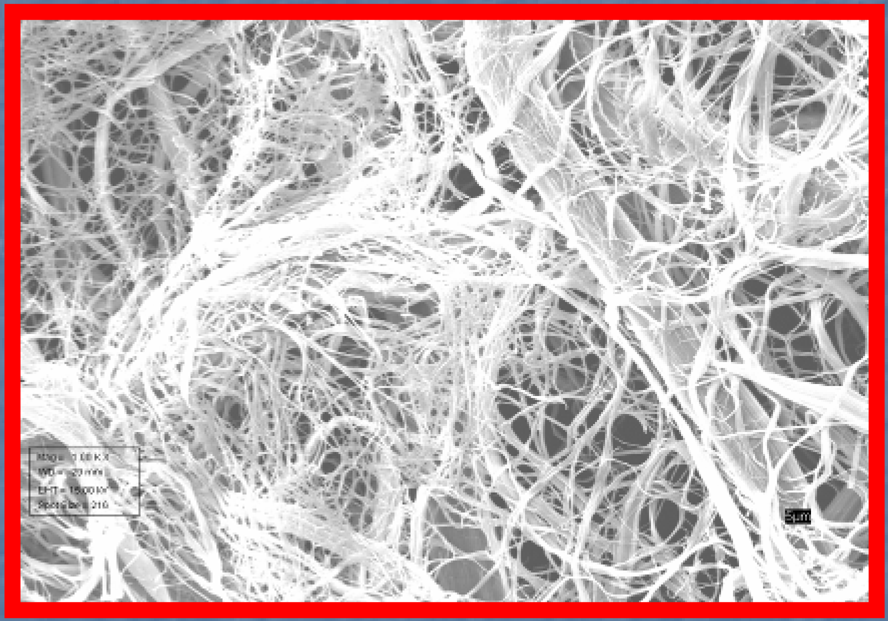
- Continued Splitting Into Microfibrils / With Final Nanofibrillated Fiber Exhibiting Range of Fibril Diameters
- High Aspect Ratio Fibrils (Est. > 1000:1)

Lyocell Nanofibrillated Fiber Structure

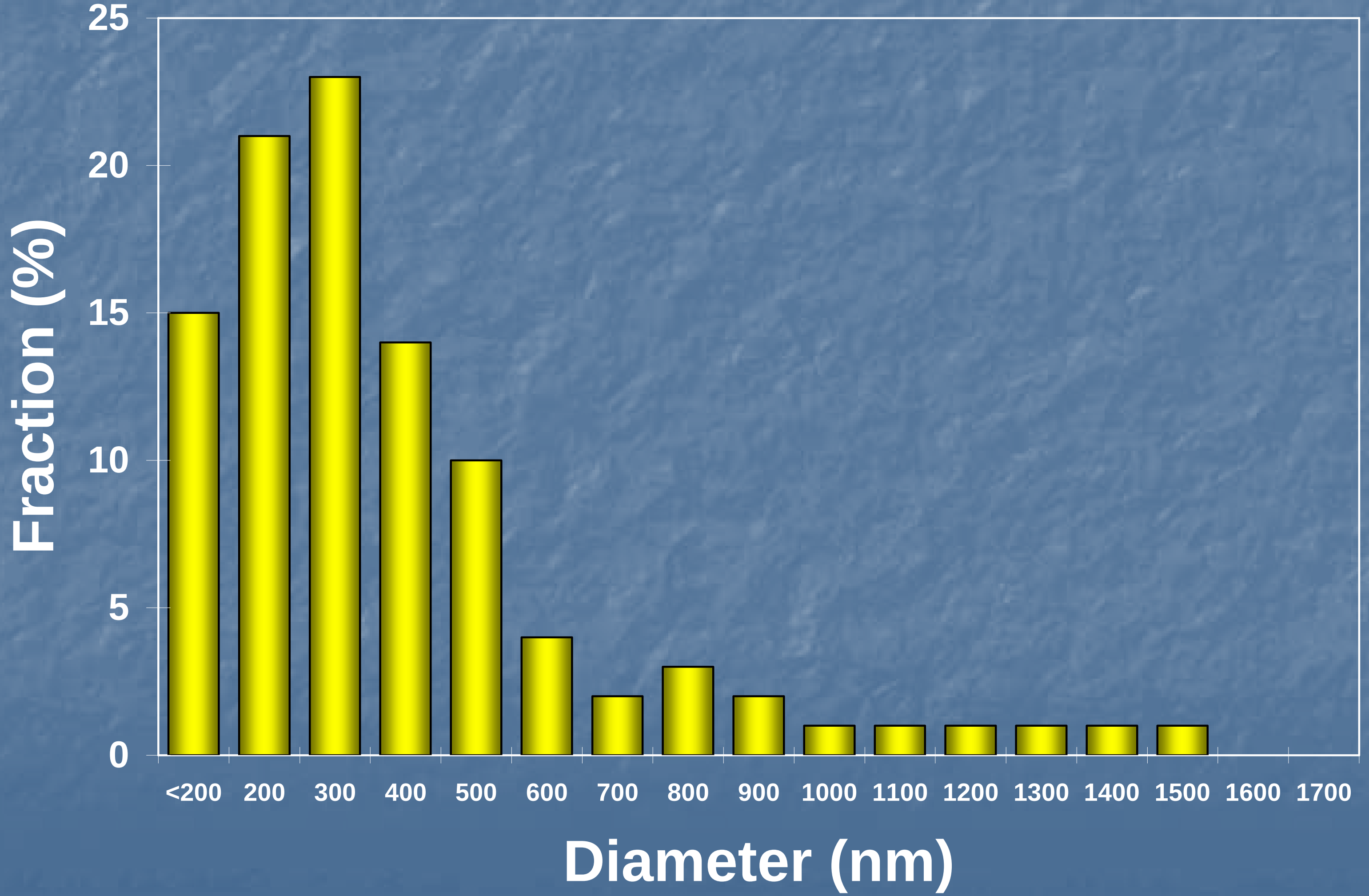
- Large majority (number average) of fibril diameters are between 0.05 and 0.5 microns, with typical average of about 0.3 micron

- Very small number fraction of 2 -5 micron diameters

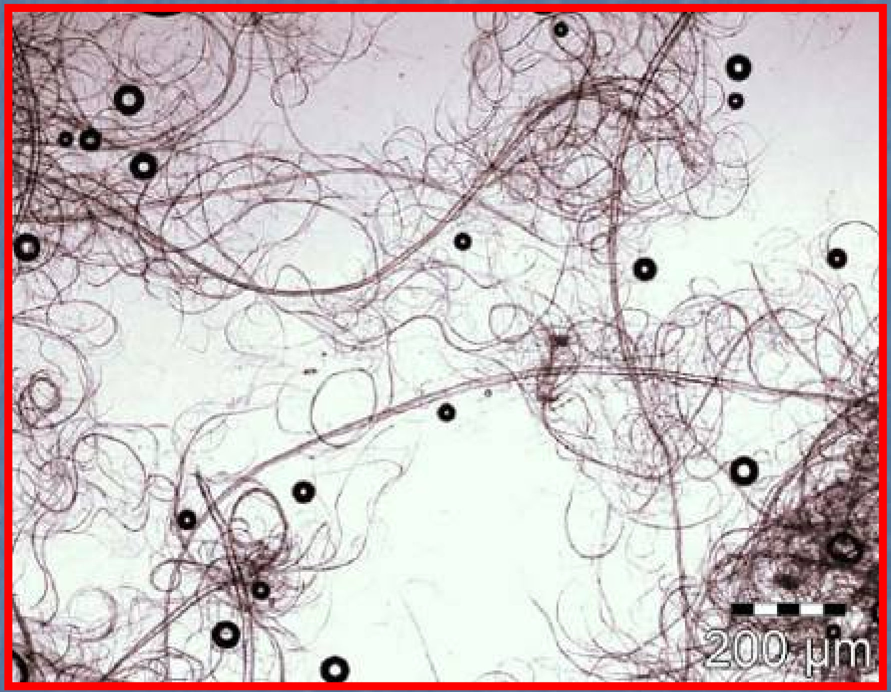
  - Effective as a “bridge or scaffold fiber” in coating applications
  - < Other Typical Fibers In Paper Formulation

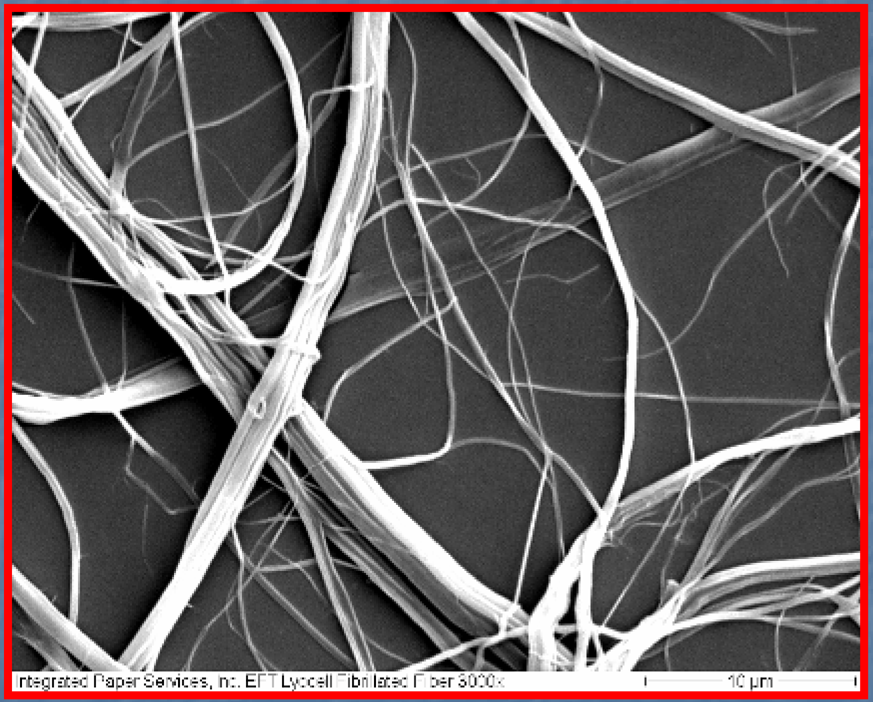


Nanofibrillated Lyocell Fibril Diameter Distribution



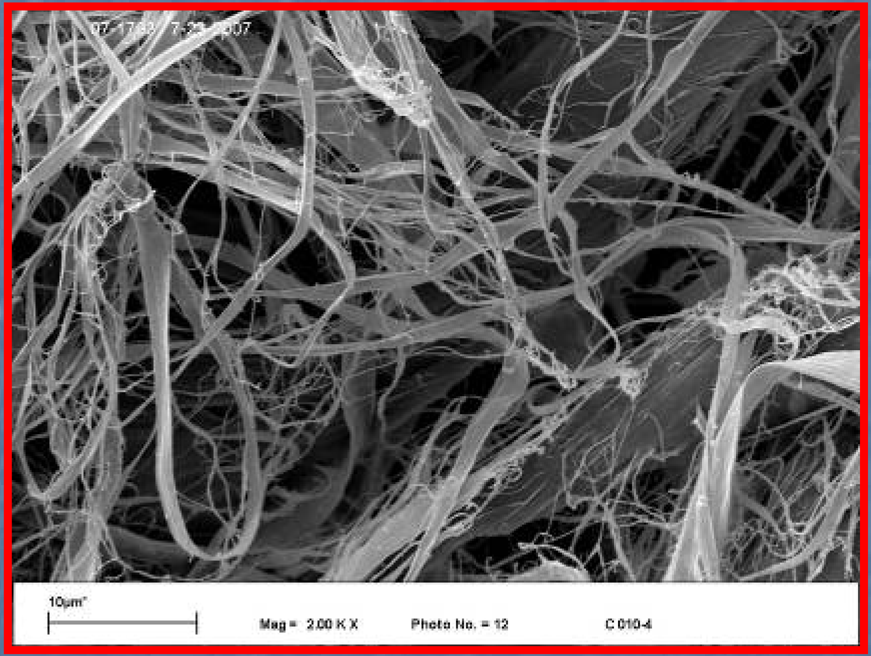
Nanofibrillated Lyocell Exhibits Very High Aspect Ratio Fibrils



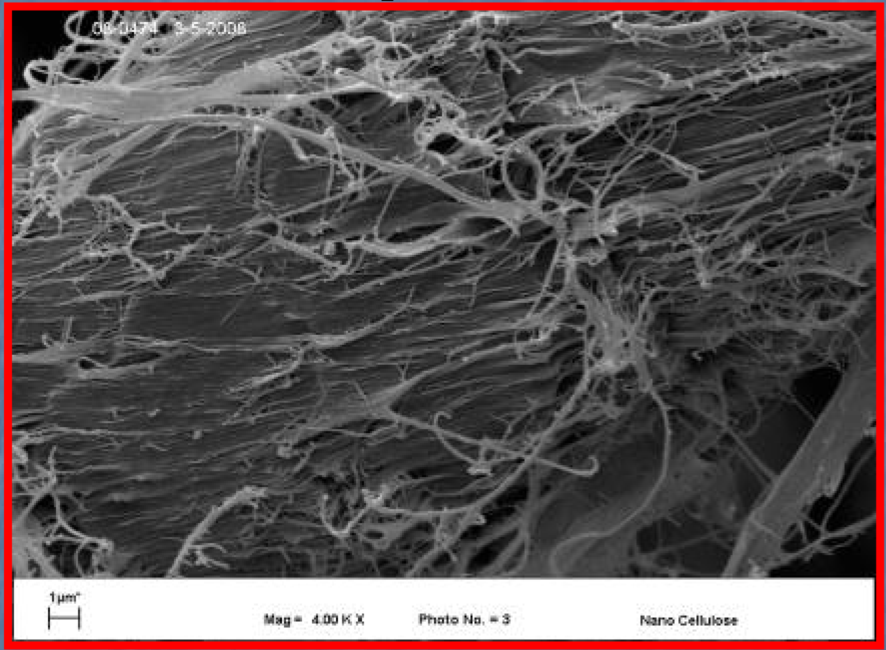


Other Wood /Non-Wood Nanofibrillation Process Developments

Cotton

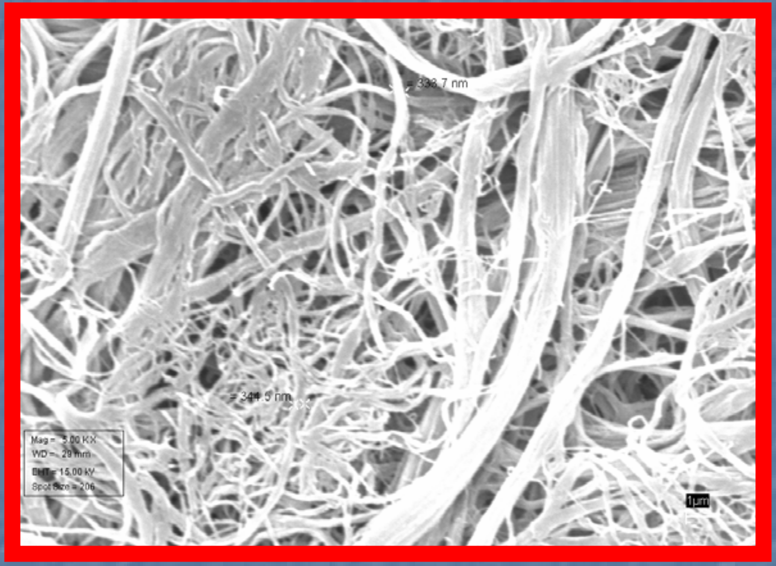


Wood Pulp



Synthetic Nanofibrillated Fiber Developments

- **Acrylic / PAN Conventional**



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Production of Lyocell Nanofibrillated Fibers

- Various Processes Described for Fibrillation In Literature Using Short-Cut Fibers

- EFT Proprietary Process Developed for Nanofibrillation

  - Fiber / Fibril Length Controlled by Length of Starting Short-Cut Fiber

  - Nanofibrillation Process Less Energy Intensive / Very High Number Average Submicron Fibril Diameters

  - Commercial Scale

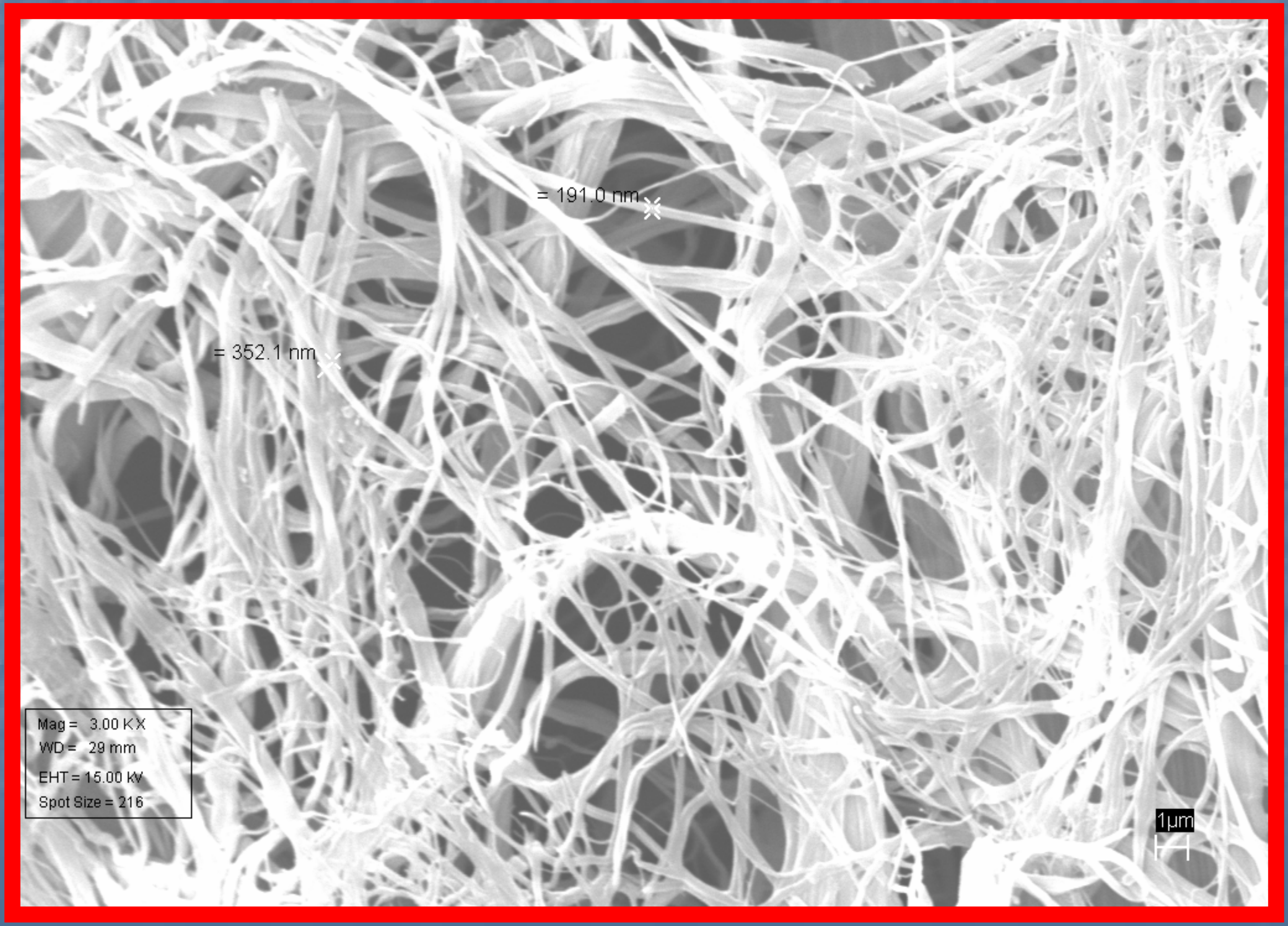
  - Economic Alternative to Other Sources of Nanofibers

Processes Being Developed for Nanofiber Production

Technique	Fiber Size (nm)	Throughput
Electrospinning	~100-300	0.3 g/hole/hour
Meltblowing	300+	0.5 g/hole/hour
Bicomponent spinning/separation	300+	0.5 g/hole/min with multiple rows of holes
Nanofibrillation	50-500	+1500 g/min/reactor

- **Nanofibrillation Process Provides a 1000X Improvement in Throughput Compared to Other Processes**

New Process Lyocell Nanofibrillated Fiber -Production



Features of New Process Nanofibrillated Fibers

- Wide Range of Nanofibrillation Level (CSF) and Fiber Length Possible
  - CSF 200 to Zero *(and Much Less)*
  - Fiber Length 1 to 8 mm
- Products Provided In Various Forms
  - Wet Slurry (2-3% Consistency)
  - Dewatered (~10% Solids)
  - Wet Lap /Crumb (~ 20 % Solids)
  - Dry Lap (~ 80 % Solids) Developmental

Features of Cellulosic Nanofibrillated Fibers

- Cellulose Nanofibrillated Fiber Supply
  - Renewable / Sustainable Sources
  - Biodegradable
  - FDA Approved Fiber Grades
- Low Cost / High Value Compared with Other Nanofiber Technologies
- Current Development Scale & Commercial-Scale Production In Place (tonnes/day production scale)

Features of New Nanofibrillated Fiber Technology

- **Hydrophillic and Hydrophobic Chemistry**
  - **Manmade and Natural Cellulose**
  - **Cellulose Blends**
  - **Acrylic (PAN)**
- **Compatible with High Speed Papermaking / Wet Laid Nonwoven Technology**

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Current Applications of Nanofibers / Wet-Laid Papers

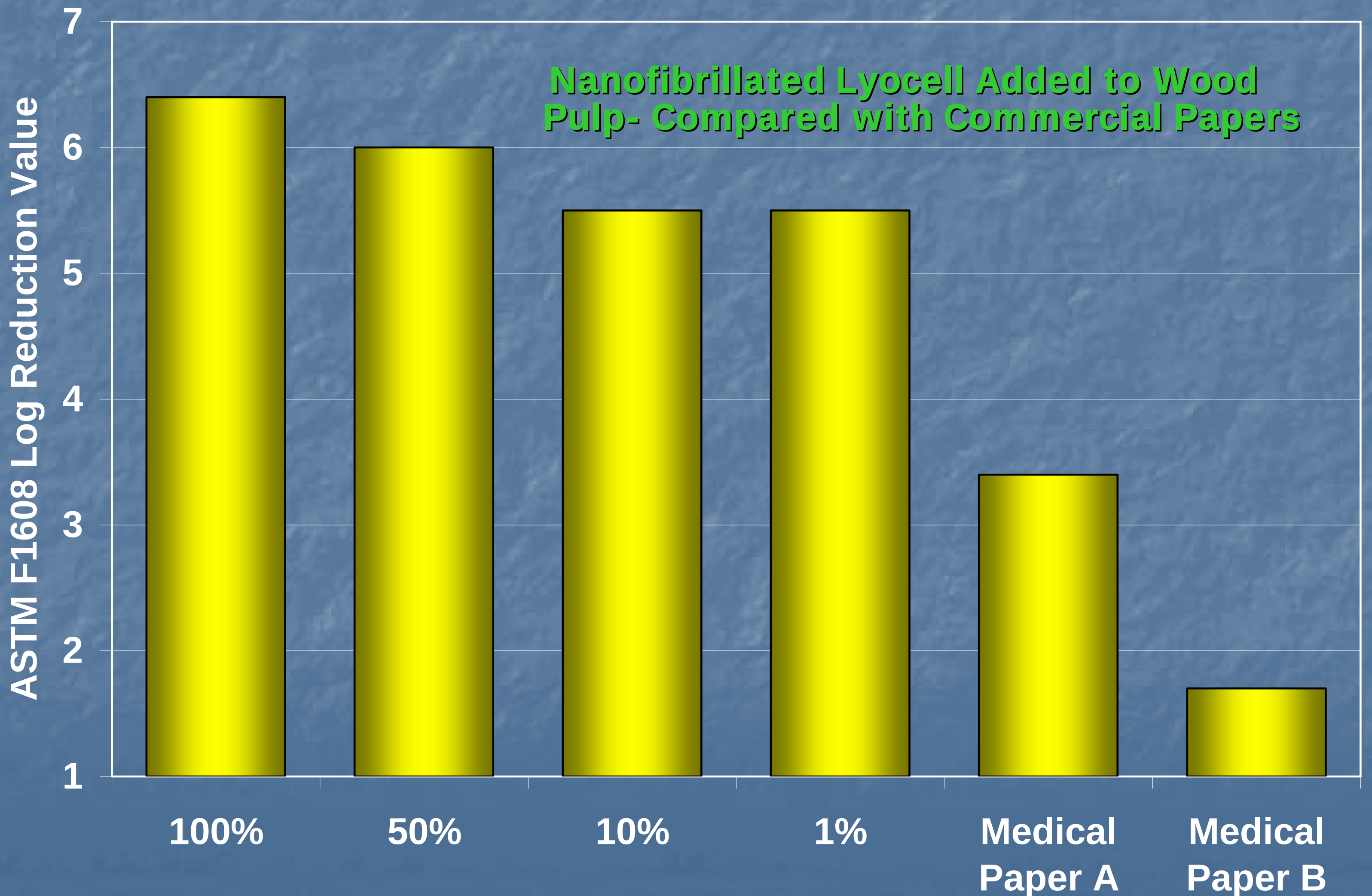
- Air Filtration
- Water / Liquid Filtration
- Protective Clothing
- Medical Barriers
- Clean Room Wipes

Cost and Low Productivity Limiting Development /Use in These and Other Applications

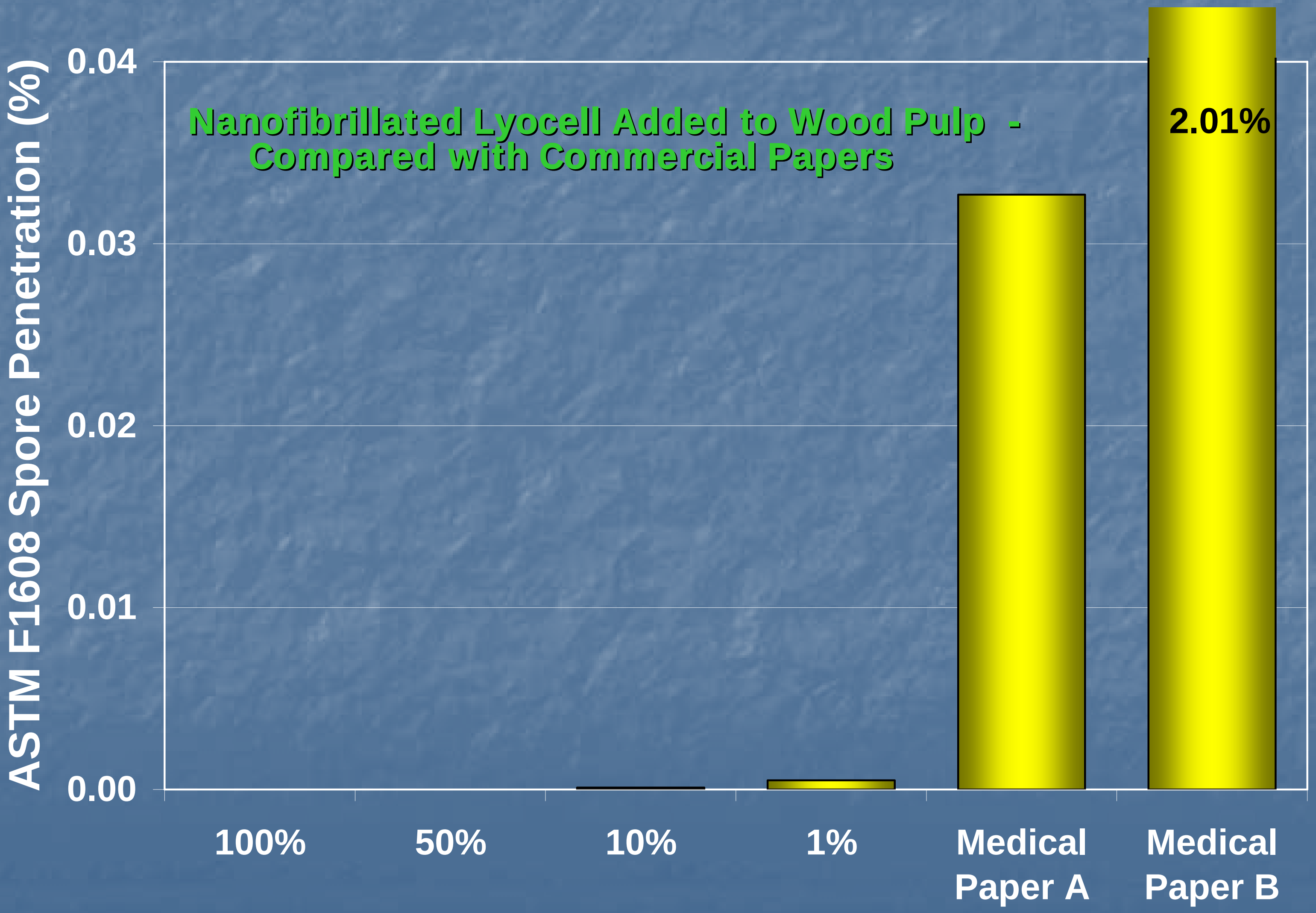
Cellulosic Nanofibrillated Fibers In Papermaking / Filtration / Nonwoven Applications

- 100 % Nanofibrillated Fiber Wet-Laid Papers / Microporous Structure
  (Mean Pore Size < 1 Micron)
- High Efficiency Binder Fiber for Other Fibers / Active Particulates (Without Blinding Absorptive Particles/Fibers)
- Coatings

Nanofiber Papers with Microbial Barrier Properties (4)



Nanofiber Papers with Microbial Barrier Properties (4)

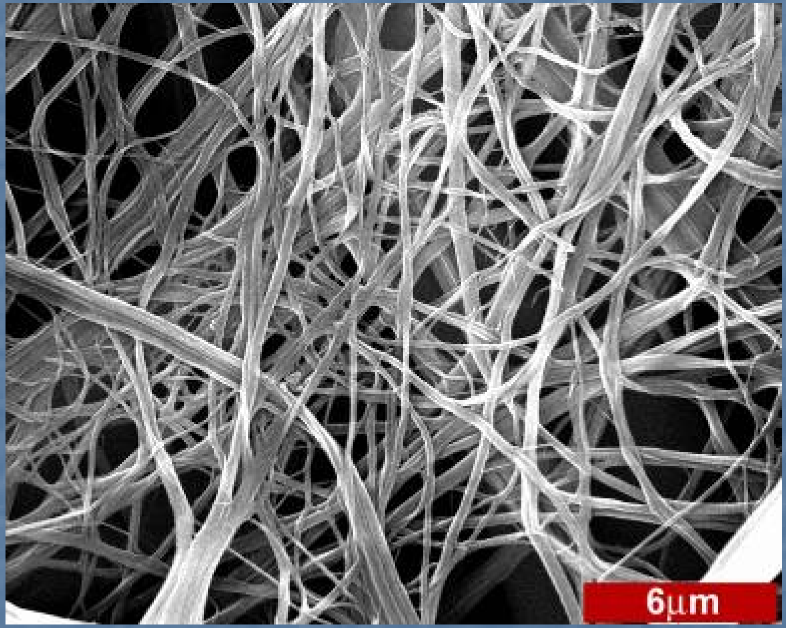


Comparison of Nanofibrillated Fiber and Other Filtration Media (5)

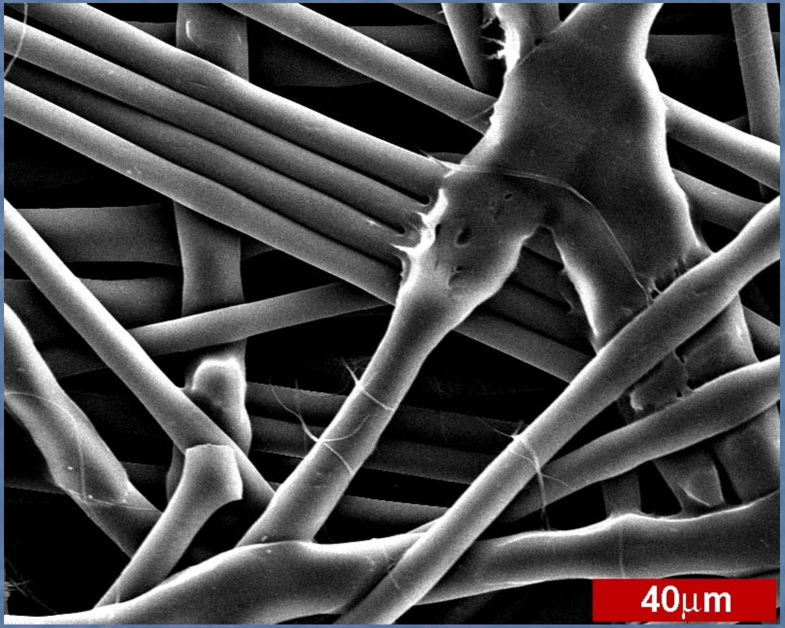
Paper Construction	Mean Pore Diameter (microns)	Air Permeability@ 1 psi (L/psi/cm^2/min)	Thickness (mm)
Nanofibrillated Fiber	0.35	2.3	0.85
Microglass Fiber	3.25	30	0.85
Meltspun Web	10	105	0.90

Nanofibrillated Fiber Coating On Nonwoven Filter Media

Coated Side

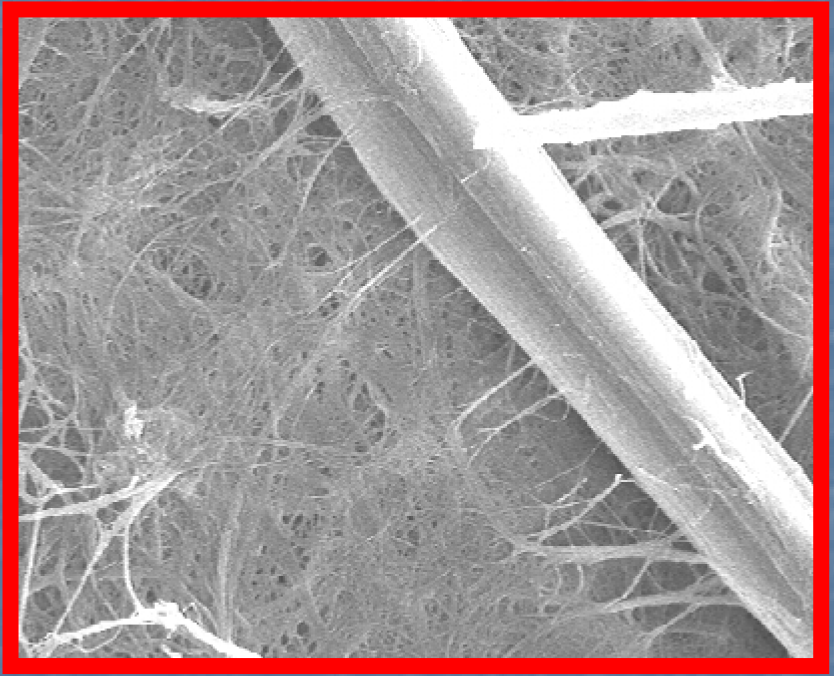


Uncoated Side



Nanofibrillated Fiber Coated Webs

- Coating Weights of 0.5 – 5 g/m^2 Typically Applied
- Slurry Coated
- Dual Headbox
- Other

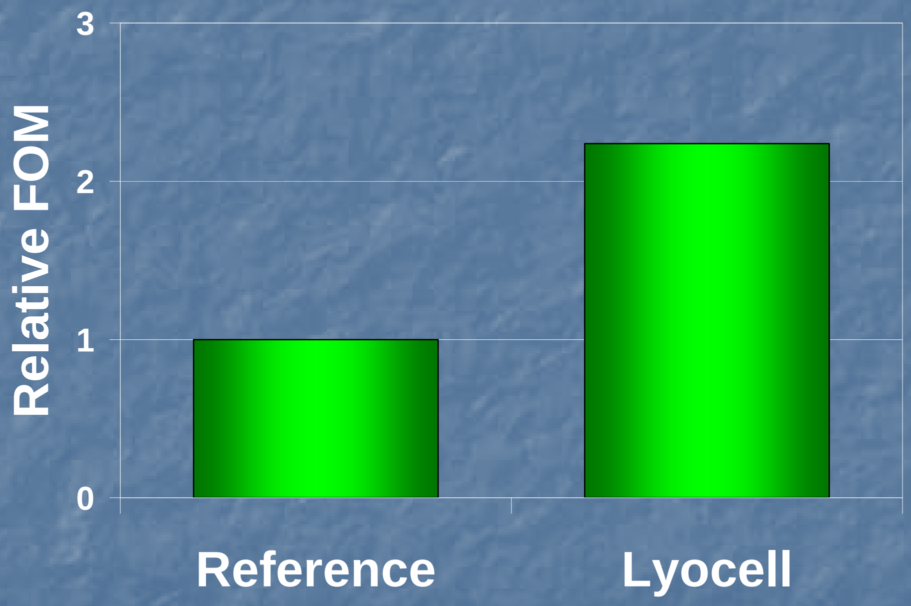


Effect of Nanofibrillated Coatings on Filtration Efficiency of Automotive Air Filter (6)

- 1.6 g/m^2 Lyocell Nanofiber Coating Level on Resin Bonded Cellulose Filter
- Relative performance can be compared using Figure of Merit (FOM)

$$FOM = \frac{-\log(1 - efficiency)}{\Delta P}$$

- Can be thought of as benefit to cost ratio
- Tested using 0.18 μm DOP aerosol at 32 l/m airflow



Nanofibrillated Fibers for Paper Coating Applications

Advantages of Nanocellulose Coatings:

- Extremely light weight additions (down to 0.005g/sq. meter)
- Increase smoothness of paper surface
- Do not have rheology issues with other additives
- Enhance pigment application in printing processes /brighter colors /greater clarity of print
- Can be charged to enhance pigment attraction to print surface.

Potential Benefits of Nanofibrillated Fibers / Wet-laid Papers

- Better Filtration Efficiency
- High Level of Particle / Fiber Retention
- Higher Wet / Dry Strength
- Lower Basis Weight
- Higher Absorbency
- Better Surface Smoothness
- Increased Barrier Properties
- Better Printing Quality
- FDA Approval / Biodegradable

Other Potential Applications- EFTec™ Nanofibrillated Fibers

- Structural Reinforcements
- Surface Modifiers
- Print Clarity Coatings
- Wet /Dry Strength Enhancers
- Processing Aids
- Particle Binders

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Summary

- EFTec Nanofibrillated Cellulosic Fibers Offer New Products for New Applications
- Production Capabilities in Place for EFTec Nanofibrillated Fibers
- Various Product Forms Can Be Supplied to Meet Specific Requirements of Papermakers
- Customer Specific EFT Cellulosic Nanofibrillated Fibers Can be Produced

Acknowledgements

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