## Carbon Fiber From Lignin

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Carbon Fiber Composites Consortium Panel Discussion

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#### Lignin Carbon Fiber Historical Context

- Japanese investigations in 1970's achieved ~ 150 ksi strength
- ORNL work began in late 1990's addressing semi-structural automotive applications
  - Initial emphasis on compounding and purification
    - Emphasis shifted in ~ 2005 to demonstrating cost and properties
  - DOE Vehicle Technologies funding discontinued in 2010 due to insufficient advancement in mechanical properties
- Recent ORNL work addresses nonstructural applications
  - Funded by DOE Advanced Manufacturing Office
  - Working on production at scale of 100+ lb scale
- ORNL efforts have focused on processing with limited chemical modification
- Currently, ORNL is investing internal funds in multiple ongoing and future projects
- Appreciable work currently underway at other institutions in US, Canada, Europe and Asia

#### **Keys to Maximizing LCF Value**



**Tendencies:** 

- Hardwood lignin melt spins well and stabilizes slowly
- Softwood lignin stabilizes well but doesn't melt spin
- High purity needed for melt spinning

# Achieving high-rate spinning, fast conversion, and high yield that are scalable has proven to be very elusive



### **Advantages of Melt Spinning**

Parameter	Solution Spun PAN	Melt Spun Lignin
Capital, per CF annual capacity	~\$13/lb	~\$4/lb
Energy per lb of CF	~180 kBtu	~10 kBtu
CO2 emitted per lb of CF	~22 lb	~1 lb
Production line length	~600 ft	~400 ft
Production line speed	~ 200 m/min	~ 2,000 m/min
Precursor concentration at die	~20%	~100%
Environmental toxicity	High	Low

Numbers from independent analysis by Kline and Company, as well as data from Energy Information Administration and industry consultants

Melt spinning is very advantageous but not an absolute requirement



#### **Lignin Specification for Melt Spinning**

- >99% lignin
- <500 ppm residual carbohydrates</p>
- <5 wt% volatiles</li>
- <1000 ppm ash
- <500 ppm non-melting particles larger than 1 micron diameter



#### **Structural Lignin Carbon Fibers**

- Multi-filament tows not despoolable
- We have produced single filaments that are moderately stretchable
- Lignin carbon fiber filaments are graphitic and isotropic; we have not yet produced aligned crystallite morphology
- Mechanical properties
  - ~ 175 ksi / 12 Msi best strength & modulus to date (with softwood)
- ~ 70 ksi / 7 Msi typical strength & modulus

Lignin does not readily produce structural fibers

Filament crystallite orientation



Multi-filament lignin tows



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#### **Costs and Properties of Structural Carbon Fibers**





## We Are Now Taking What Mother Nature Gives Us

- Current focus on functional applications for which lignin is well-suited
  - Thermal management for high temperature processes
  - Reduction of inactive material in chemica batteries
  - Sorbents for filtration
  - Sorbents for gas storage
  - Super / ultra-capacitors

Photos are pitch carbon fiber applications with high potential to use lignin carbon fibers



Monolith for gas storage



# Carbon insulation in 2000C furnace



#### **Recent Accomplishments**

- Successful scale-up of pelletization and fiber spinning
  - Over one ton of pellets produced in ~ 1,000 lb batches
  - ~ 1,300 lb of fibers produced in four batches
- Estimated mill cost ~ \$4-5/lb in web form





Lignin fiber web formation by melt-blowing

9 Managed by UT-Battelle for the U.S. Department of Energy We believe this was the first-ever production of lignin fibers in batch size > 100 lb



#### **Major Challenges**

- Lignin sourcing huge potential with limited current supply
  - High-quality, lab-scale samples
  - Multiple scalable sources meeting cost and quality metrics hardwood and softwood from pulp mills and biorefineries
- High-rate spinning and conversion
- Chemical yield
- Equipment fouling
- Mechanical properties
  - Likely requires major chemistry effort



Residue on an oven exhaust cover after ~ 1,000 hours of lignin heating



# ORNL Strategy-

Plants designed for processing with properties optimized for applications

Chemistry, chemistry, & more chemistry High-rate, scalable process development

Melt spinning preferred but not required

Advanced conversion processes

Apply lessons learned from functional fiber development to achieve structural properties

#### **ORNL Lignin Carbon Fiber Tech Team**



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