Low-Cost Bio-Based Fibers for High-Temperature Processing

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GrafTech International Holdings Inc./ Oak Ridge National Laboratory

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Project Objective

Goal:

- Develop and demonstrate lignin-based carbon fibers in high-temperature applications. Focus on melt processing of fibers.
    - Rigid insulation, felt insulation, graphite electrodes, filtration

Problem:

- No one has yet developed or commercialized scalable and cost-effective lignin precursor technology and demonstrated that fibers are drop-in high-value replacement for fibers in an existing application

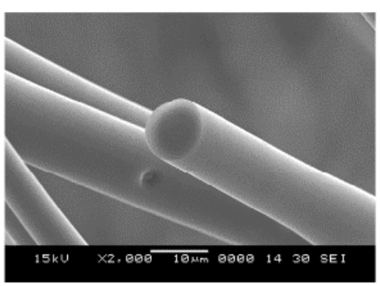
Challenge:

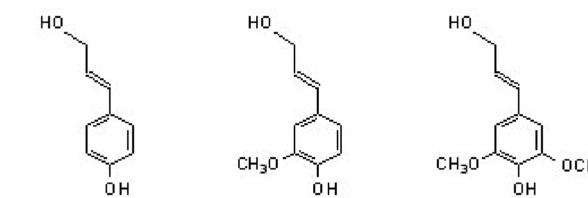
- Lignin has a complicated chemical structure and is difficult precursor
- Developed technology must work with multiple lignin types
- Technology must be scalable, cost-effective, and be better than current
- Insulation is cost driven product. Innovation needed to compete

Technical Innovation

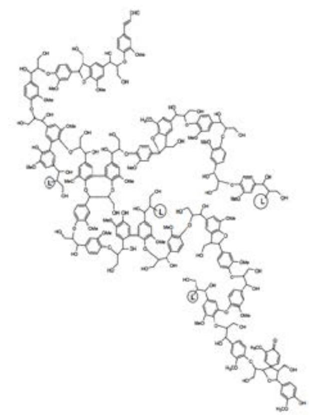
- Today there are no commercial lignin-based carbon fibers
- Only academic studies reported with focus on improving strength
- Lignin is being sold into primarily chemical and agricultural applications. Off-take agreements needed to advance lignin supply

- Our technical approach works with and not against the lignin structure
  - **Application-driven approach**: focus on “functional” properties not structural properties
  - **Cost-driven approach**: focus on meeting cost metrics
  - **Process-driven approach**: focus on scalable technology





p-Coumaryl alcohol Coniferyl alcohol Sinapyl alcohol



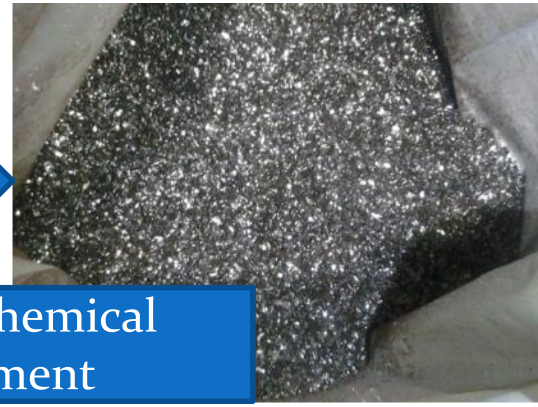
Technical Approach

- Focus low-cost chemical modification of lignin structure
  - BP1 – develop and demonstrate lab scale precursors and fibers
  - BP2 – produce 500 lbs and demonstrate in product prototypes
  - BP3 – produce 5000 lbs and demonstrate in end use environment
- GTI
  - Over 130 years of carbon and graphite product experience
  - USA manufacturing base for insulation but global customer base
- ORNL
  - Over 20 years experience with lignin precursor R&D and scale-up
  - World class precursor labs and fiber spinning capabilities
  - ORNL CFTF key component of scale-up assessment
- Risks
  - Technology does not work – pursue and downselect from parallel paths
  - Technology does not meet cost – focus on low cost chemical treatment
  - Technology does not scale-up – focus on scalable chemical treatment

Technical Approach

Lignin powder

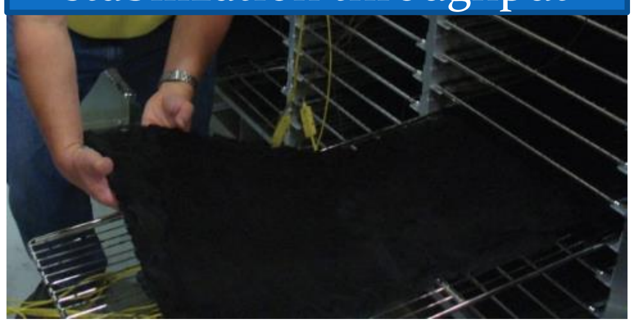
Lignin chemical treatment



Lignin pellets

Lignin fiber mat by melt processing

Treatment enhances stabilization throughput



Lignin mat after stabilization

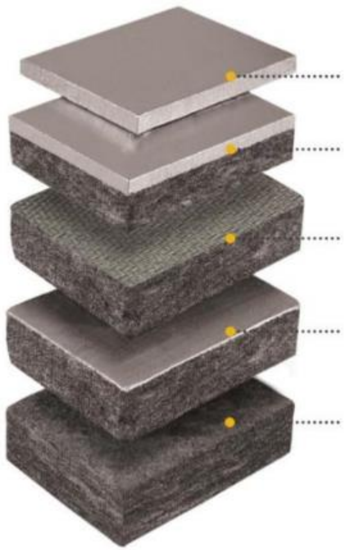
Lignin fiber after carbonization and milling

Insulation
Graphite electrodes
Filtration

Transition and Deployment

- Carbon fiber is expensive. Many applications are cost driven.
- Selected applications need functional not structural properties
- Lignin carbon fibers could be low cost and high performance
- Applications
  - High-temp insulation (rigid and felt)
  - Graphite electrodes
  - Filtration (activated fibers)
- Commercialization approach
  - Develop scalable lignin precursor technology
  - Demonstrate high throughput melt processing of lignin
  - Demonstrate lignin carbon fibers in prototype products
  - Validate products in customer environment
- Sustainment
  - Development of scalable lignin fiber technology platform
  - Qualification of multiple lignin types (hardwood, softwood)

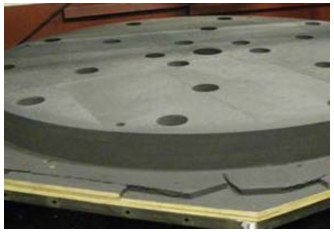
Transition and Deployment



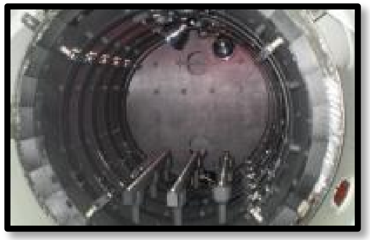
GRI™ Insulation
- Rigid, high-temperature insulator
- 100% bonded carbon fiber
- Standard and purified grades
- Optional anti-dust coating
- Panels and machined parts

Applications
- Vacuum furnaces
- Brazing furnaces
- Sintering furnaces
- Crystal growth furnaces

Various GRI™ products machined into shapes.

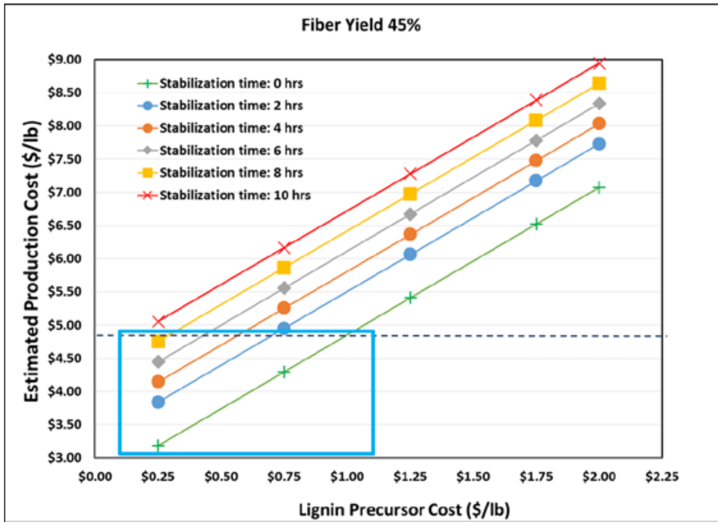


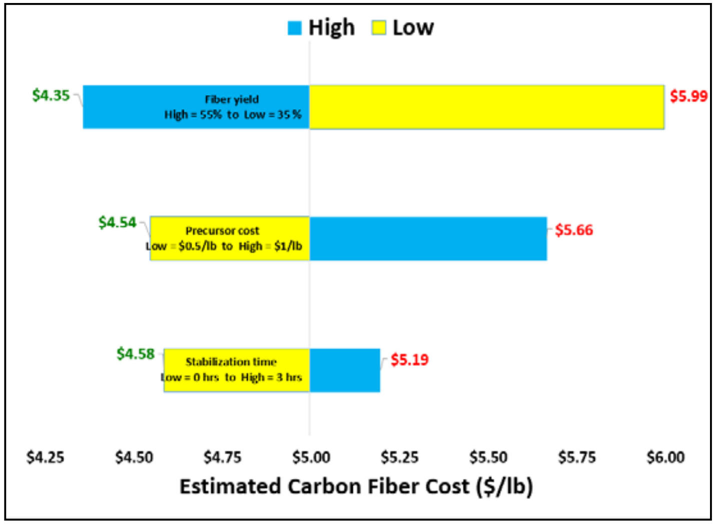
FiberForm®



Preliminary Lignin-based Carbon Fiber Cost Modelling (Production Cost)

Main Effects:
-Carbon yield
-Precursor cost
-Stabilization time





Measure of Success

- Insulation is price driven market
  - Target for est. lignin carbon fiber production cost is $5.00/pound
  - Customers expect low cost and high thermal efficiency
  - Customers expect high purity (ash in product less than 500 ppm)

- Existing insulation product lines targeted for immediate impact
  - Drop-in replacement for current rayon and pitch foreign-sourced fibers

- Lower-cost lignin fibers could have several impacts:
  - Displacing rayon and pitch fibers with USA-based fibers
  - Lower cost product, increasing market competitiveness
  - Better insulation product, increasing energy efficiency
  - Lower CO_2 vs PAN: lignin is already polymerized and can be melt processed
  - Jump starting lignin fibers for other applications

- Benefits: cost, higher performance, energy efficiency, CO_2 avoidance

Project Management & Budget

- Project began 12/22/2014. End date 6/21/2018.

- BP1 has 4 main tasks and milestones. Ending 6/21/2016
  - Identify and screen at least 10 different lignin samples
  - Show at least 4 that meet process and cost criteria ($5.00/pound)
  - Also must meet purity level (ash < 500 ppm in final fiber product)
- BP 2 has 4 mains tasks and milestones
- BP3 has 4 main tasks and milestones

Total Project Budget	
DOE Investment - GTI	$1,250,000
DOE Investment - ORNL	$3,250,000
Cost Share	$1,900,443
Project Total	$6.400,000

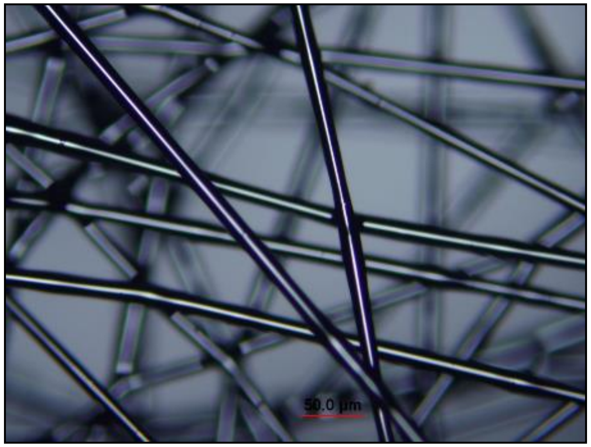
Results and Accomplishments

- BP 1 will finish 6/21/2016
- Significant accomplishments on precursor and process at lab scale
  - Developed Low-cost scalable chemical treatment to accelerate stabilization
  - Works on hardwood, softwood, and grasses, but to different degrees
  - Accelerated thermal stabilization of less than 3 hours achieved for all
  - Stabilized fibers can be carbonized with acceptable yield
  - Carbonized fibers can be milled to suitable fibers for insulation
- Currently evaluating scale up from gram scale to kilogram scale

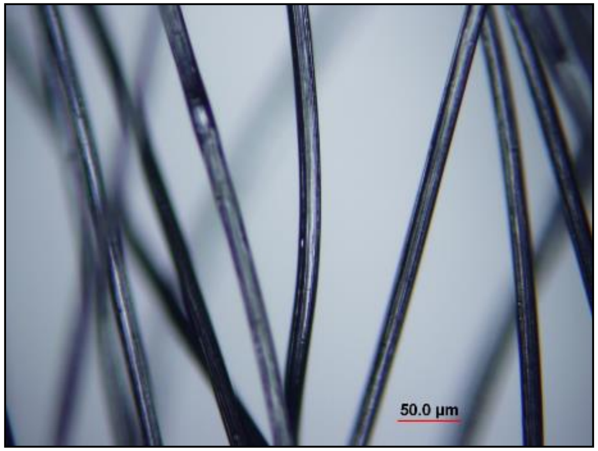
ID	Sample	Chemical Treatment	Melt Spin Temp	Best Stabilization Time To Date	Carbon Yield	Est. Production Cost
1	HW orgnsolv	YY + ZZ	155 - 170 °C	3 hours	44%	$4.99/lb
4	SW kraft purified	WW + YY + ZZ	130 °C	1.5 hours	41%	$5.00/lb
5	Grass kraft purified	WW+ZZ + YY	127-133 °C	2 hours	42%	$5.03/lb
DD	DD	YY + ZZ	136 °C	2.5 hours	44%	$4.94/lb

Results and Accomplishments

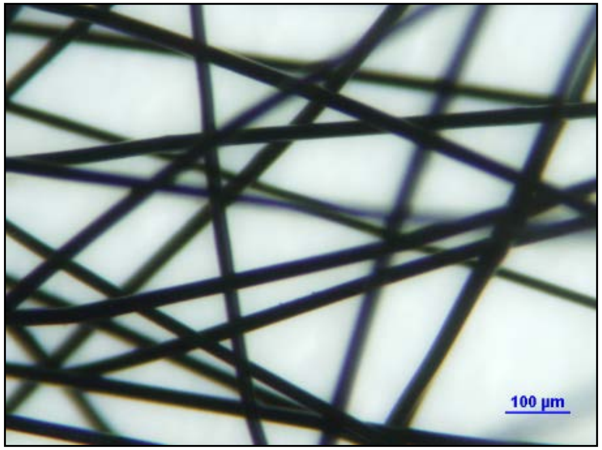
Microscopy of Stabilized Fibers at Lab Scale



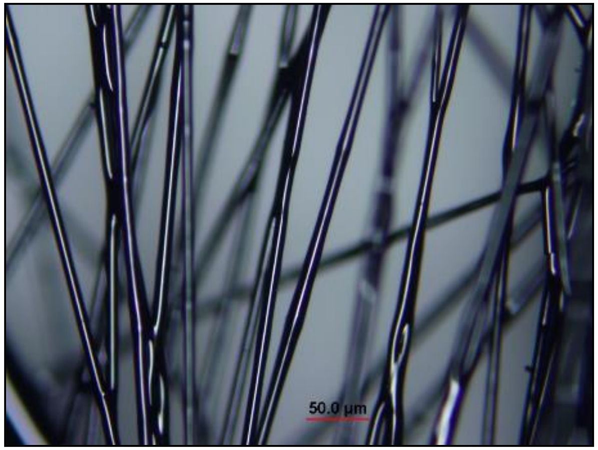
HW organosolv – 3 hours



Softwood kraft purified – 1.5 hours



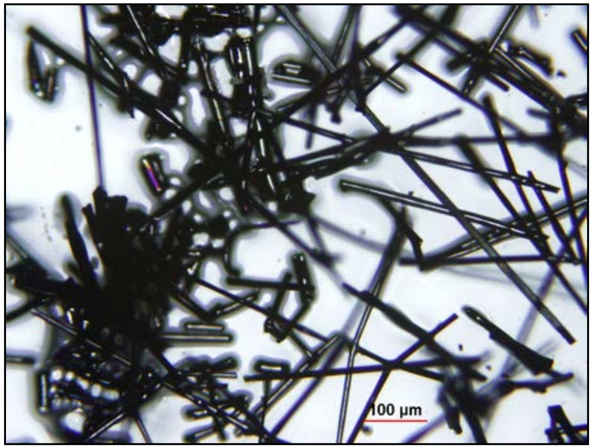
Grass kraft purified – 2 hours



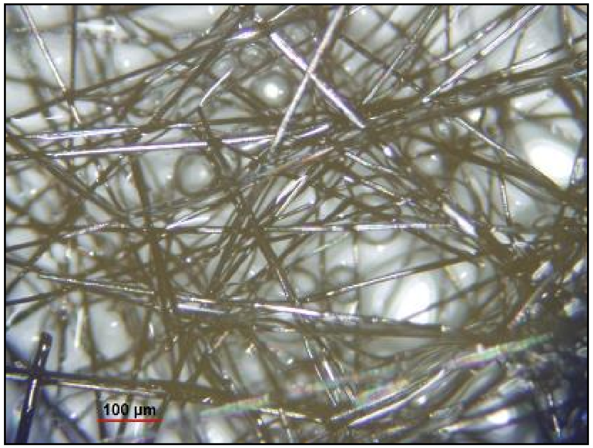
Lignin #DD – 2.5 hours

Results and Accomplishments

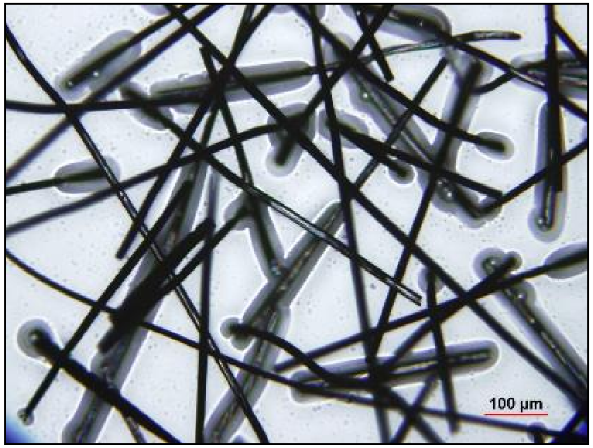
Microscopy of Carbonized and Milled Fibers at Lab Scale



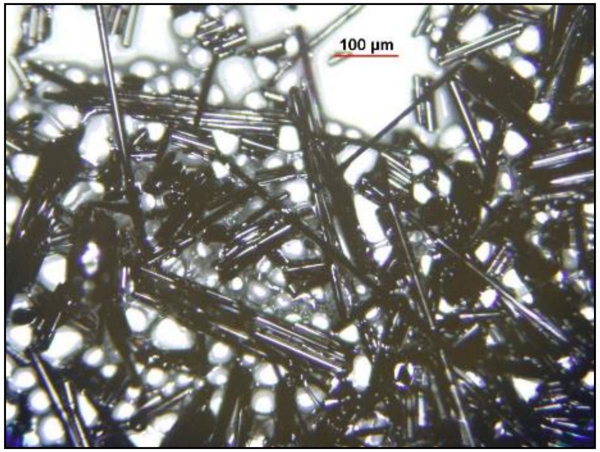
Hardwood organosolv



Softwood kraft purified



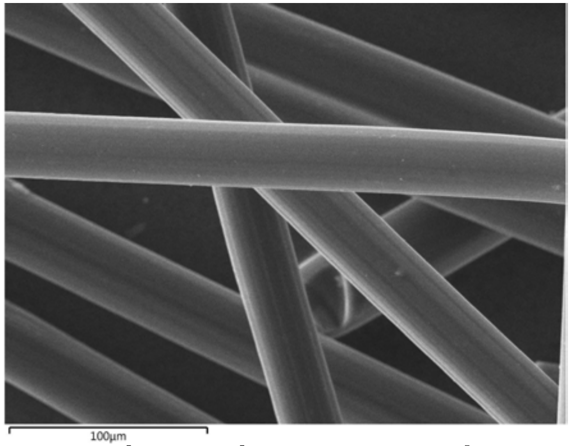
Grass kraft purified



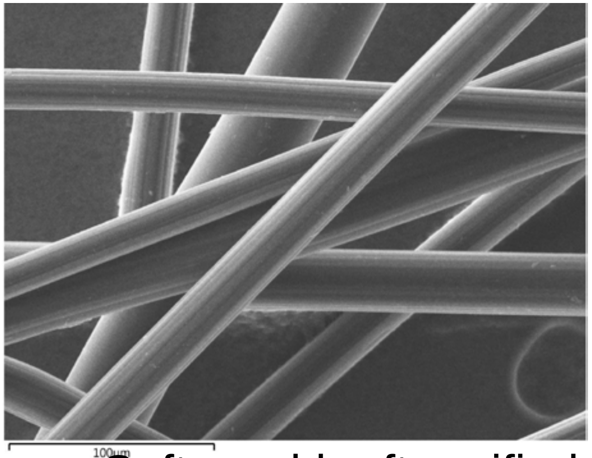
Lignin #DD

Results and Accomplishments

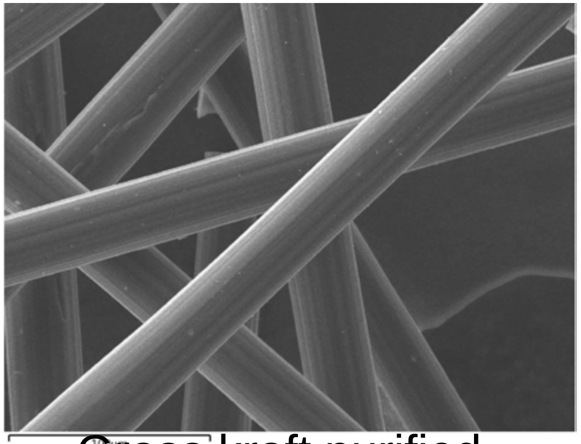
Microscopy of Carbonized and Milled Fibers at Lab Scale

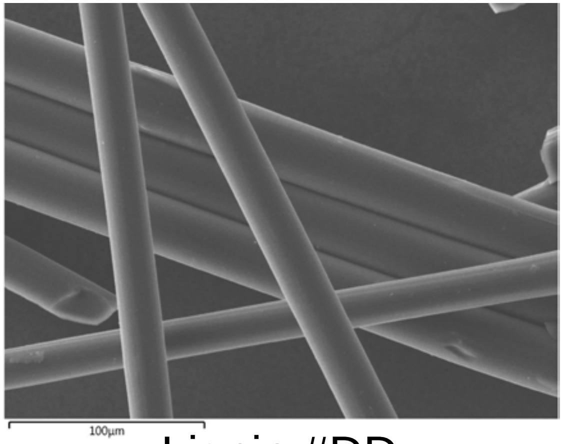


Hardwood organosolv



Softwood kraft purified


Grass kraft purified



Lignin #DD