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NATIONAL LABORATORY

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FOR THE DEPARTMENT OF ENERGY

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**CRADA NO. ORNL02-0644**

**CRADA FINAL REPORT  
FOR CRADA NO. ORNL02-0644**

**MATERIALS DEVELOPMENT FOR PULP AND PAPER MILLS,  
TASK 9 PROOF OF COMMERCIAL CONCEPT: COMMODITY  
CARBON FIBERS FROM WEYERHAEUSER LIGNIN BASED  
FIBERS**

**Between**

**UT-Battelle, LLC**

**And**

**Weyerhaeuser Corporation**

**From:** Bissett, Paul [paul.bissett@weyerhaeuser.com]  
**Sent:** Friday, August 13, 2010 2:35 PM  
**To:** Eberle, Cliff  
**Cc:** Keiser, James R.; Phillips, Jeanne R.  
**Subject:** RE: Weyerhaeuser CRADA Title  
**Attachments:** Bissett, Paul.vcf

Cliff,

We have reviewed the document and approve the content for public release. No modifications are necessary.

Thanks,

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## Background

Tasks were assigned to Oak Ridge National Laboratory (ORNL) researchers for the development of lignin-based carbon fiber from a specific precursor that was produced by the Participant (Weyerhaeuser Corporation). These tasks included characterization of precursor polymers and fibers; and the development of conversion parameters for the fibers. ORNL researchers provided recommendations for in-house characterization of the precursor at the participant's laboratory.

During the early stage of the precursor fiber production trials of various spools of fibers with varied compositions were produced. Some of those samples were sent to ORNL (by the Participant) for the development of conversion protocol. The trial tow samples were oxidized at ORNL's precursor evaluation system (PES), a bench-scale facility consisting of an oven, filament winder, tension controller, and a let off creel. The PES is a modular tool useful for the development of precursor conversion protocol. It can handle a single filament to a large single tow (50k filaments). It can also offer precise tensioning for few-filament tows. In the PES, after oxidation, fibers are typically carbonized first at low temperature,  $\leq 600^{\circ}\text{C}$ , and subsequently at a higher temperature,  $\leq 1200^{\circ}\text{C}$  with controlled residence time. ORNL has recently installed a new carbonization furnace with  $1700^{\circ}\text{C}$  limit and a furnace with  $2500^{\circ}\text{C}$  capacity is under installation.

## Summary

A protocol for the oxidation and carbonization of the trial precursor fibers was developed. Oxidized fiber with a density of  $1.46\text{ g/cc}$  (oxidation time: 90 min) shows qualitative flame retardancy via simple flame test (fibers do not catch fire or shrink when exposed to flame). Oxidized and carbonized filaments of the Weyerhaeuser precursor fibers show moderate mechanical properties and 47-51 % carbon yield (based on oxidized fiber mass) after carbonization between  $1000^{\circ} - 1400^{\circ}\text{C}$ . The properties of fibers from non-optimized composition and processing parameters indicate the potential of low-cost, low-end carbon fibers based on renewable resource materials. Further work is necessary to produce high quality precursor and the corresponding carbonized filaments of superior properties.