## SUSTAINABLE NANOMATERIALS FROM FOREST PRODUCTS: UMAINE PERSPECTIVE

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## Stone Age ...... Bronze Age ...... Iron Age ...... Nano Age?

## Ligno-Cellulose: Maine's Niche to Compete in Nanotech



"From the Sawmill to the Nanomill?"



## **Overarching Research Focus**

"Establish world-class R&D capacity in the field of ligno-cellulose derived nanomaterials leading to transformative technologies and industries that can change the face of Maine's natural-resource based economy."





## Nanocellulose is a renewable resource abundant in Maine





#### A Wealth of Cellulose Nanofibril Architectures are Possible









bacterial cellulose nanofibrillated cellulose electrospun cellulose cellulose nanocrystals

#### **Applications Range from Nano to Macro Components**













#### U.S. Forest Products Nanotechnology Research Roadmaps - Needs

#### 2005



#### 2006



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2010

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www.nanotechforest.org www.agenda2020.org

http://www.nano.gov/html/research/NNISigInitSustainableMfrFINALJuly2010.pdf

#### Nanomaterials From Forest Products UMaine Research Projects

- Scalable Production of Cellulose Nanofibrils: A joint venture with USDA Forest Products Laboratory
  - Pilot-Scale Nanofibrillated Cellulose Production
  - Pilot-Scale Spray Drying Capacity
- In situ surface modification of CNF during drying to promote improved polymer compatibility and thermal stability
- Ice-segregation-induced self-assembly (ISISA) to template lignin and cellulose nanofibers into a 3-D non woven mat that can be freeze-dried to liberate the nanofiber mat
- Carrier systems for CNF in hydrophobic polymer composites
- Renewable nanocomposites made from lignocellulosic fillers and transparent polymer matrices



- Utilization of CNF in paper coatings
- Utilization of CNF in packaging applications
- Production of CNF Aerogels for structural insulating foams
- High CNF content materials including films, filters, etc.
- NFC-clay matrix composites
- Carbonized lignin nanofibers as an additive to create conductive polymers





## Scalable Production of CNF

## Objectives

- Demonstrate pilot scale capability to produce CNF using a scalable manufacturing process.
- Determine the effects of species (i.e. hardwood and softwood) and pretreatment parameters on CNF properties

## Deliverables

- Develop capacity to provide pilot quantities of slurry and dry CNFs (NFC and CNC) to Consortium members
- Further scientific understanding of feedstock, pretreatment and processing conditions on CNF properties



## Scalable Production of CNF

## Timeline

- Ultrafine Grinder
  - Bench unit is operational
  - Pilot-scale unit is ordered July installation
- Spray Dryer
  - Performance trials completed
  - Pilot-scale unit is ordered with an expected August shipping date
- Pilot-scale production of NFC
  - Aqueous suspensions expected Q3 2012
  - Spray-dried NFC, CNC and TEMPO (from FPL) expected Q4 2012



## **Broader Impacts**

## Scientific

 Ability to source NFC and CNC samples for fundamental research and application development

## Industrial Relevancy

 Demonstrate the value of NFC/CNC in applications at a commercially relevant scale and a viable pathway to commercial production



## Scientific Approach

- Production of aqueous suspensions of NFC via mild pretreatment and mechanical treatment of lignocellulosics
- Characterization of aqueous suspensions
- Production of dried NFC/CNC via spray drying of aqueous suspensions
- Characterization of spray-dried NFC/CNC
  - Particle size
  - Morphology
  - Surface area/surface energy



## **Hydrodynamic diameters**

CNC: 21 ~ 51 nm & 79 ~ 342 nm NFC: 712 ~ 1484 nm





## Key Findings – Aqueous Suspensions TEM





















## Key Findings – Spray Dried CNF

#### CNC





![](_page_14_Picture_5.jpeg)

![](_page_14_Picture_6.jpeg)

![](_page_14_Picture_7.jpeg)

![](_page_15_Picture_0.jpeg)

## Key Findings – Spray Dried CNF

## Morphologi G3S

Particle characterization based on image analysis of dry powder

![](_page_15_Picture_4.jpeg)

CE diameter (µm)

![](_page_15_Figure_5.jpeg)

Sampla							
Sample	D(n,0.1	D (n, 0.5)	D (n, 0.9)				
CNC	1.31	3.06	6.76				
NFC	1.59	2.96	7.48				

![](_page_16_Picture_0.jpeg)

#### Scale up from bench to pilot scale

![](_page_16_Figure_2.jpeg)

Particle size distribution of NFC analyzed based on image analysis

Sample	Distribution pressure (bar)	d(0.1) (µm)	d(0.5) (µm)	d(0.9) (µm)	D[4,3] (µm)	D[3,2] (µm)
NFC (Dried by Buchi B-290)	5	1.79	3.30	6.97	15.72	9.614
NFC (Dried by GEA)	5	0.28	5.04	10.93	18.93	13.66
NFC with surfactant (Dried by GEA)	5	0.27	5.11	11.33	20.33	14.47

![](_page_17_Picture_0.jpeg)

## In situ surface modification of CNF

## **Scientific Approach**

![](_page_17_Figure_3.jpeg)

![](_page_18_Figure_0.jpeg)

## Cost estimates for CNF samples

## **Consortium Members**

- Aqueous suspension of NFC
  - \$10.39 per dry lb of NFC FOB Orono, ME, ~2% solids
- Dried NFC or CNC
  - Drying charge \$95.91 per dry lb of NFC or CNC
  - Does not include material costs or shipping

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![](_page_19_Picture_0.jpeg)

## Challenges

## Control of CNF Morphology

- Dispersion of aqueous suspensions
- During drying processes

## Reduction of production costs

- Energy consumption
- Pretreatment costs
- Drying productivity
- Economies of scale

## \*Processing of CNF nanocomposites by classical methods (extrusion, injection molding)

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## Benefits to Maine, the U.S. and Society

#### **Benefits for Maine:**

- Transform its Forest Products Industry
- Become an Active Player in the Nanotech Revolution

#### **Benefits for U.S. and Society:**

Renewable recyclable, sustainable technology

![](_page_20_Picture_7.jpeg)

#### 20% lighter

- Intelligent products with nanosensors for measuring forces, loads, moisture levels, temperature.
- Building blocks of nanoproducts with substantially enhanced properties.
- Coatings for improving surface qualities to make existing products more effective.
- Basis for making lighter-weight products from less material and with fewer energy requirements.

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# Thank You!

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