Developments in CNC Commercialization

PRESENTED BY:

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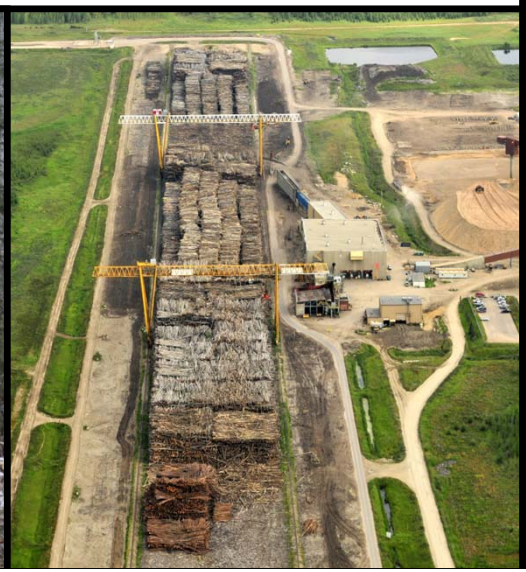
ALBERTA PACIFIC FOREST INDUSTRIES INC

HOKUETSU GROUP

TAPPI

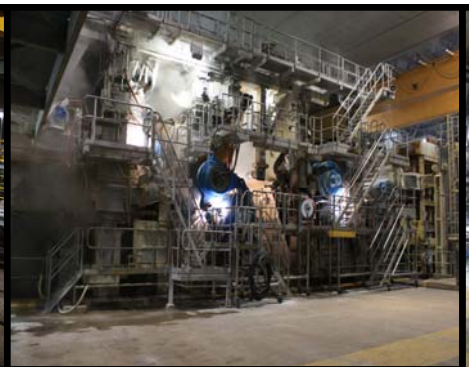
Outline

- About Alberta-Pacific Forest Industries Inc.
- Technical and Market Update
- Health & Safety, Regulatory Approval Update
- Nano Celluloses: CNC and CNF
- ASPEN-323



About Alberta-Pacific

- **Modern Pulp and Power Mill**
  - **640,000 ADMT/yr of Kraft Pulp**
  - **860,000 MW/yr green energy**
  - **Bio-Methanol**
- **64,000 km^2 boreal mixed wood forest**
- **FSC certified since 2005**
- **Striving to be a preferred global supplier of high quality Cellulose NanoCrystals (CNC)**



Technical Update

- Completed upgrades (equipment and process) to CNC Pilot Plant
  - Debottlenecking, increase capacity, improve reliability, lower costs
- Sample Production: Constantly producing CNC material to keep ahead of sample demand
- Video tutorial available online to assist with redispersion of spray-dried material (available in both English and Japanese)
- Free technical support available to assist with:
  - redispersion of dried material
  - mixing CNC with a variety of preferred polymers
  - tailoring / modifying / functionalizing CNC for various application

Technical Update: Continuous CNC Reactor

- Continuous reactor → smaller/cheaper
- Plug flow → consistent reaction
- Reduce acid & cost by ~3x
- Improved economics when acid re-used in mill
- Increased production capacity
- Acid recovery reduces costs:
  - Overall plant opex savings, ~10%
  - Overall plant capex savings, ~25%

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Continuous CNC Reactor

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- Use kraft pulp/DP as feedstock
- No mixing required after initial contact with acid
- ~2.5 : 1 acid ratio (pure acid basis)
- Ran 5 continuous hours: filled storage capacity
- Product characterization indicates production of high quality CNC

Installation and Integration with InnoTech Alberta CNC Pilot Plant

Demonstration Facility: 2021

- Product: Cellulose NanoCrystals, "Aspen – 323"
- Process: Sulphuric Acid Hydrolysis using Kraft or Dissolving Pulps
- Production Capacity: 0.5 MT/day - Spray-dried & never-dried formats (range of concentrated liquid products)
- Targeting 2 grades based on market requests:
  - Premium grade: (food contact/ medical / personal care & cosmetics)
  - Performance grade: (functionality, but lower purity)
- Unmodified: sulphate half esters – self-dispersing, well suited for surface and chemical modification

Technical Update: Demonstration Facility

- First of kind continuous CNC demonstration production facility
- Located at our Pulp Mill facility in Central Alberta
- Built to de-risk but also scalable to grow with market demand
- Preliminary design and Class 3 +20/-15% engineering is underway for 0.5TPD front-end & 0.1TPD back-end, expandable to respond to market demand
- Interim: increasing production capacity at Alberta Innovates

Market Update

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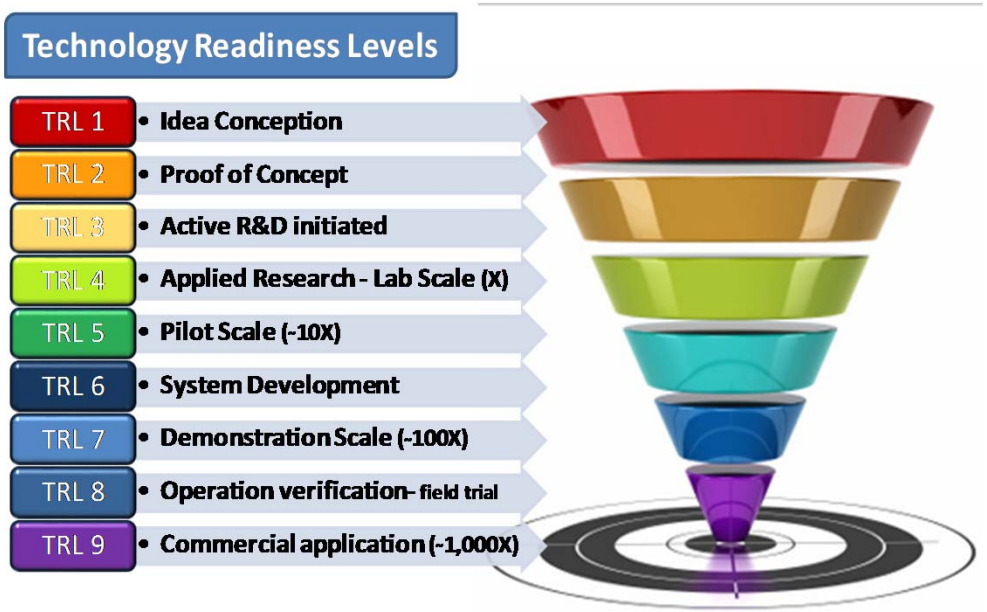
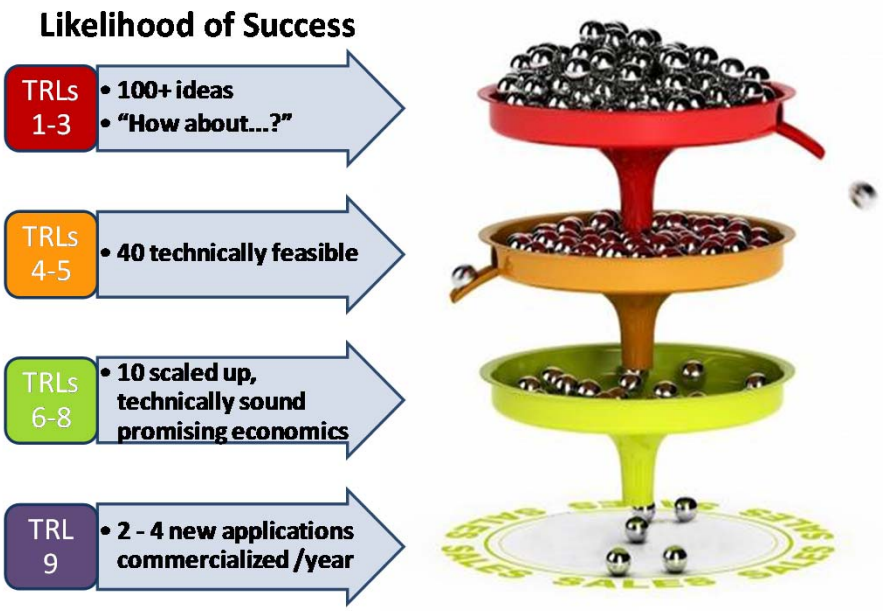
- CNC Challenge 3
  - TRLs 1-3: 12 projects @ 1kg of CNC and $25k
- BioProduct (CNC) Pursuit
  - TRLs 4-6: 9 projects @ up to 10kgs of CNC and $300k





CNC Health & Safety and Regulatory Approvals

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- Inventory Expert Service (IES) Confirmation:
  - **C**hemical **A**bstracts **S**ervice (CAS) Registry Number: **9005-22-5**
  - CAS Index name: Cellulose, hydrogen sulfate, sodium salt
- In Canada, CNC is on the Domestic Substance List
- In the United States, CNC is listed on the non-confidential portion of the U.S. Toxic Substances Control Act (TSCA) Inventory
  - Pursuing Generally Recognized as Safe (GRAS) status through the U.S. Food and Drug Administration (FDA)
- In Japan, CNC is listed on listed on the:
  - Existing and New Chemical Substances (ENCS) Inventory under the Chemical Substances Control Law (CSCL) and;
  - Industrial Safety and Health Law (ISHL) list
- CNC assigned an International Nomenclature Cosmetic Ingredient (INCI) name in the International Cosmetic Ingredient Dictionary and Handbook (ICID)
- Have initiated market specific regulatory approval processes for key geographies

Food Use Safety Study

Partners: P3Nano, Alberta Innovates, U.S. Govt. Granting Agencies & Other Industry Partners

Led By: Vireo Advisors, LLC

Completed Testing

- Physical/Chemical Characterization
  - Microscopy; size distribution;…
  - Certificate of Analysis meeting Food Chemical Codex Specifications for Impurities and Microbiological Tests
- Toxicology Studies
  - 90-day feeding study, 2%, 3%, 4% in diet
  - no effects seen at highest dose
  - No Observable Adverse Effects Level (NOAEL) >4%
- Alternative Testing Strategy
  - To allow "read across" and meet EU guidance
  - *In progress*

Study Details in Session 19:
Methods in Safety Evaluation of CNM
Wednesday June 5; 15:30

90 Day Dietary Study Update
Dr. Kimberly Ong, Vireo Advisors 16:38

Alternative Testing Strategies Updates
Dr. Jo Anne Shatkin, Vireo Advisors 15:54
Dr. Christie Sayes, Baylor University 16:16

CNC Meets Purity Specifications for Food

- Certificate of Analysis - ensures products meet specifications
- If use biocide, then have to make sure it is approved
- Food Chemicals Codex (FCC) specifications

Alberta-Pacific's CNC Certificate of Analysis

Chemical Test	Al-Pac CNC Test Result	FCC Specification
Ash (total), %	0.02	≤ 0.3
Chloride, %	< 0.01	≤ 0.05
Heavy Metals, ppm as Pb	0.07	≤ 3.0
Loss on Drying, %	0.10	≤ 0.7
pH (10% suspension)	6.59	5.0 - 7.5
Water Soluble Substance, %	0.01	≤ 1.5

Microbiological test (25g sample)	Test Result	Specification
Standard Plate Count, cfu/g	0.00	≤ 1,000
Yeast and Mold, cfu/g	0.00	≤ 100
Salmonella	Negative	Negative

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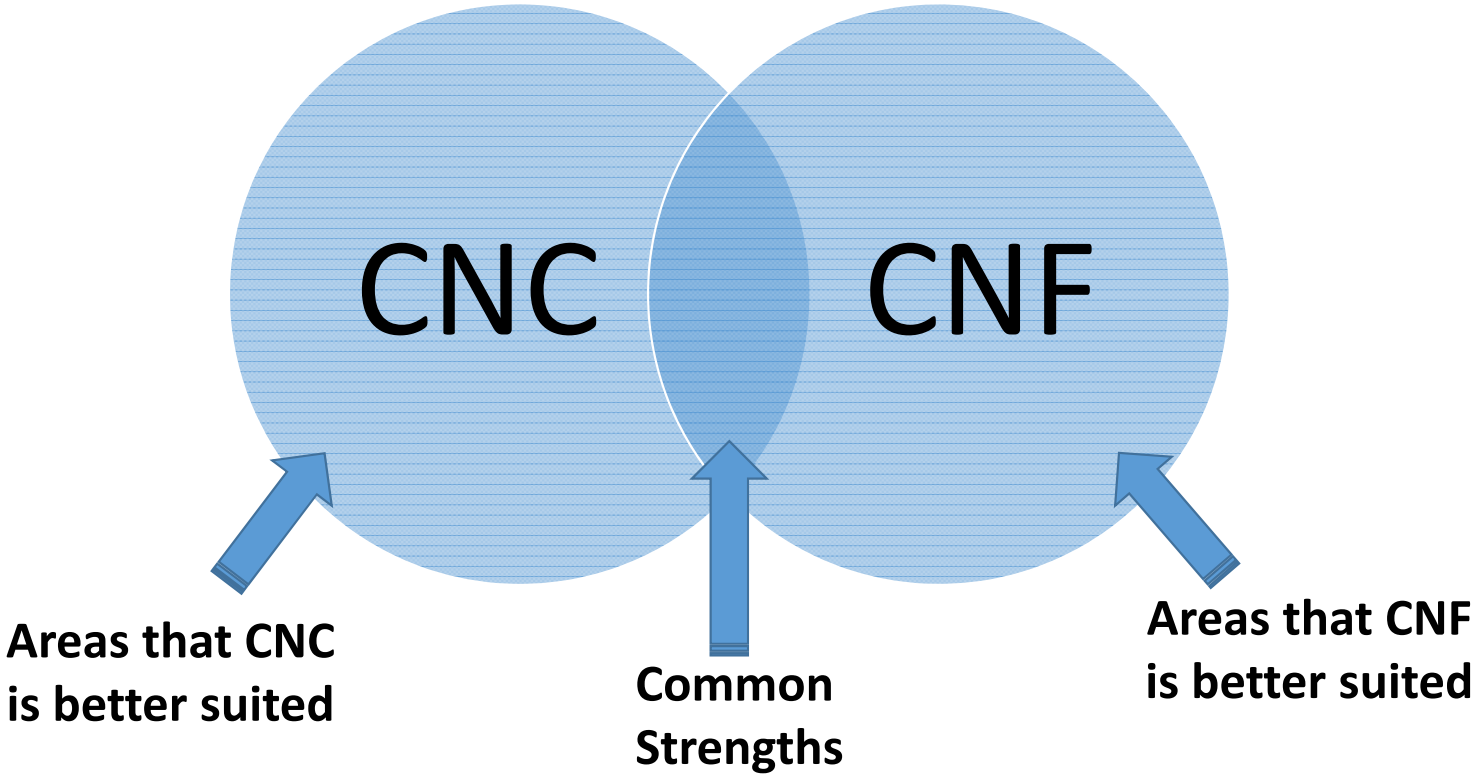
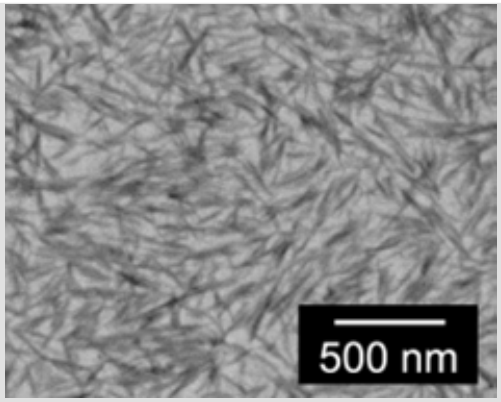
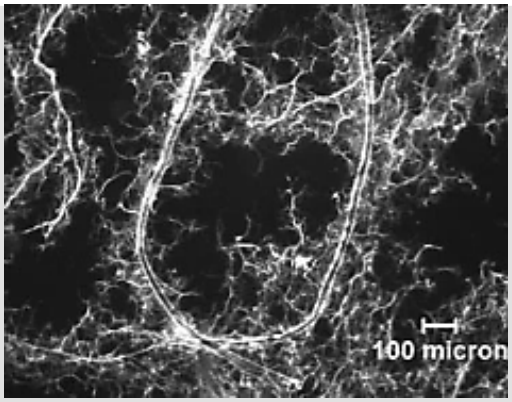
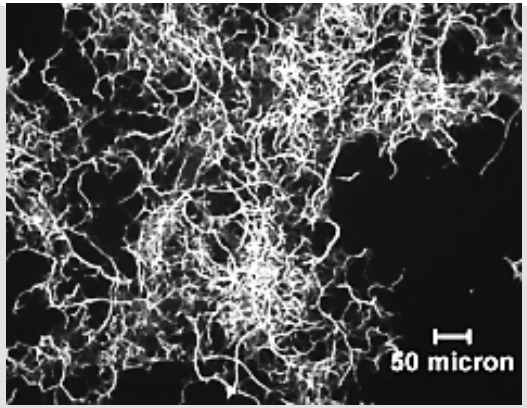
NanoCellulose Materials Fibres and Crystals

Very common question in Japan: Which is better; CNC or CNF?

Answer: Both have very strong potential; depending on use/need



Material Characteristics

CNC	CF	CNF
		
Rigid spindles	Fine fibrils attached on/to fragments	Highly entangled fine fibrils
Interactions		
H-bonding & van der Waals dispersion forces	Physical entanglement	Physical entanglement

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NanoCellulose Materials Fibres and Crystals

Properties	Cellulose NanoCrystals (CNC)	Cellulose NanoFibers (CNF)
Process	H_2SO_4 hydrolysis process is most widely accepted method	Widely varies, most accepted is TEMPO-mediated oxidation, mechanical defibrillation, lignin-CNF, and bacterial CNF
Surface functionalities	Sulfate groups	Carboxyl groups
Morphology	Width: 3-10 nm Length: 100 - 200 nm	Width: 5-20 nm Length: 400 - 4,000 nm
Uniformity	Uniform	Variable
Process	Chemical intensive	Energy intensive
Re-Dispersion of dry particles into nanosized particles in aqueous systems	Straightforward	More difficult

Note: This summary is prepared based on our best knowledge of information. However, results can vary by surface chemistry and morphology of each material.

Material Characteristics

Properties	*CNC*	*MCC*	*CNF*	*Kraft Softwood Pulp*
Morphology	*Spindle*	*Particulate*	*Network*	*Rod*
Length, nm	*~ 100-250**	*N/A*	*≥ 5,000***	*~ 1,500,000*
Diameter, nm	*~ 5-20*	*~ 50,000*	*~ 5-30*	*~ 30,000*
Specific Surface Area, m^2/g	*~ 400*	*~1-10*	*~ 20-100*	*~ 4*
Degree of Polymerization (DP)	*~ 100*	*~ 200-300*	*400 < DP < 1000****	*> 1000*
Crystallinity Index, %	*> 85*	*~ 50-60*	*~ 50-60*	*~ 50-60*
Tensile Strength, MPa	*~ 10,000*	*~ 11 (compressive strength)*	*~ 200†*	*~ 700*
Elastic Modulus, GPa	*~ 145-150*	*-*	*~ 10-20†*	*~ 20*

* Values are for wood-derived CNCs. CNCs from tunicate can reach 1000 nm, and are whisker-like. But, they are of limited practical value.
** CNF is a fibrous network, hence it is inaccurate to speak of discrete "nanofibril length." We here refer to the possible length within the entangled web.
*** DP for CNF depends on type and extent of treatment, e.g., TEMPO oxidation. More treatment leads to lower DP.
† Tensile strength and elastic modulus value are measured for films of CNF and not individual nanofibrils.

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Material Characteristics

	Density (g/cm^3)	*Tensile strength (MPa)*	*Young's modulus (GPa)*	*Elongation at break (%)*
CNC	**1.56**	**10,000**	**145-150**	**6.7**
SWCNT*	**1.2**	**30,000**	**1054**	**6**
MWCNT**	**2.6**	**30,000**	**1000-1280**	**12.5**
Carbon	**1.7**	**4,000**	**230-240**	**1.4-1.8**
Kevlar 29	**1.44**	**2,800**	**183**	**4**
Aramid	**1.4**	**3,000-3,150**	**63-67**	**3.3-3.7**
E-glass	**2.5**	**2,000-3,500**	**70**	**2.5**
S-glass	**2.5**	**4,570**	**86**	**2.8**
302 Stainless steel	**7.75-8.05**	**1,280**	**210**	
Kraft softwood	**~1.5**	**~700**	**~20**	**~2-4**
Cotton	**1.5-1.6**	**287-800**	**5.5-12.6**	**~7-8**
Jute	**1.3-1.45**	**393-773**	**13-26.5**	**1.16-1.5**
Flax	**1.50**	**345-1,100**	**27.6**	**2.7-3.2**

*SWCNT = Single-walled carbon nanotubes
**MWCNT = multi-walled carbon nanotubes

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CNC vs CNF Functionalities

CNC

- ❑ High strength
- ❑ High surface area
- ❑ Active surface
- ❑ Rheology modifier
- ❑ Self assembly
- ❑ Chiral
- ❑ Optical and photonic properties
- ❑ Electromagnetic properties
- ❑ Electrochemical properties
- ❑ Electromechanical properties

CNF

- ❑ Moderate strength
- ❑ Moderate surface area
- ❑ Active surface
- ❑ Rheology modifier

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CNC vs CNF: Application Opportunities

A concrete example – both have a role

CNC

1) Rheology mixing & pouring
2) Cement precasting applications
3) Porosity and permeability control
4) Flexural strength

CNF

1) Moisture Retention
2) Shrinkage Prevention
3) Crack Prevention

Al-Pac's CNC Flagship Product

Introducing Al-Pac's premium grade of Cellulosic NanoCrystals

ASPEN-323

An unmodified CNC targeted towards performance and appearance grade products with stringent technical expectations.

Premium Grade CNC – pure, bright white
Meets specs as per Canadian Domestic Substance List

Physical Properties

- Appearance: white powder or flakes
- 5% moisture content
- Particle length: 100-200 nm
- Particle diameter: 5-15 nm
- Specific surface area: 300-400 m^2/g
- Density: 1.6 g/cm^3
- Crystallinity index: > 80%
- Zeta potential in deionized water: <-50mV
- Onset of degradation temperature: 260ºC
- Total sulfur content wt% 0.5-1 (EA^3)

Acknowledgements

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Thank you

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