Demonstration of CNC Production at Dramatically lower Acid Ratios

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New Technologies for the Pulp Industry

LignoForce™
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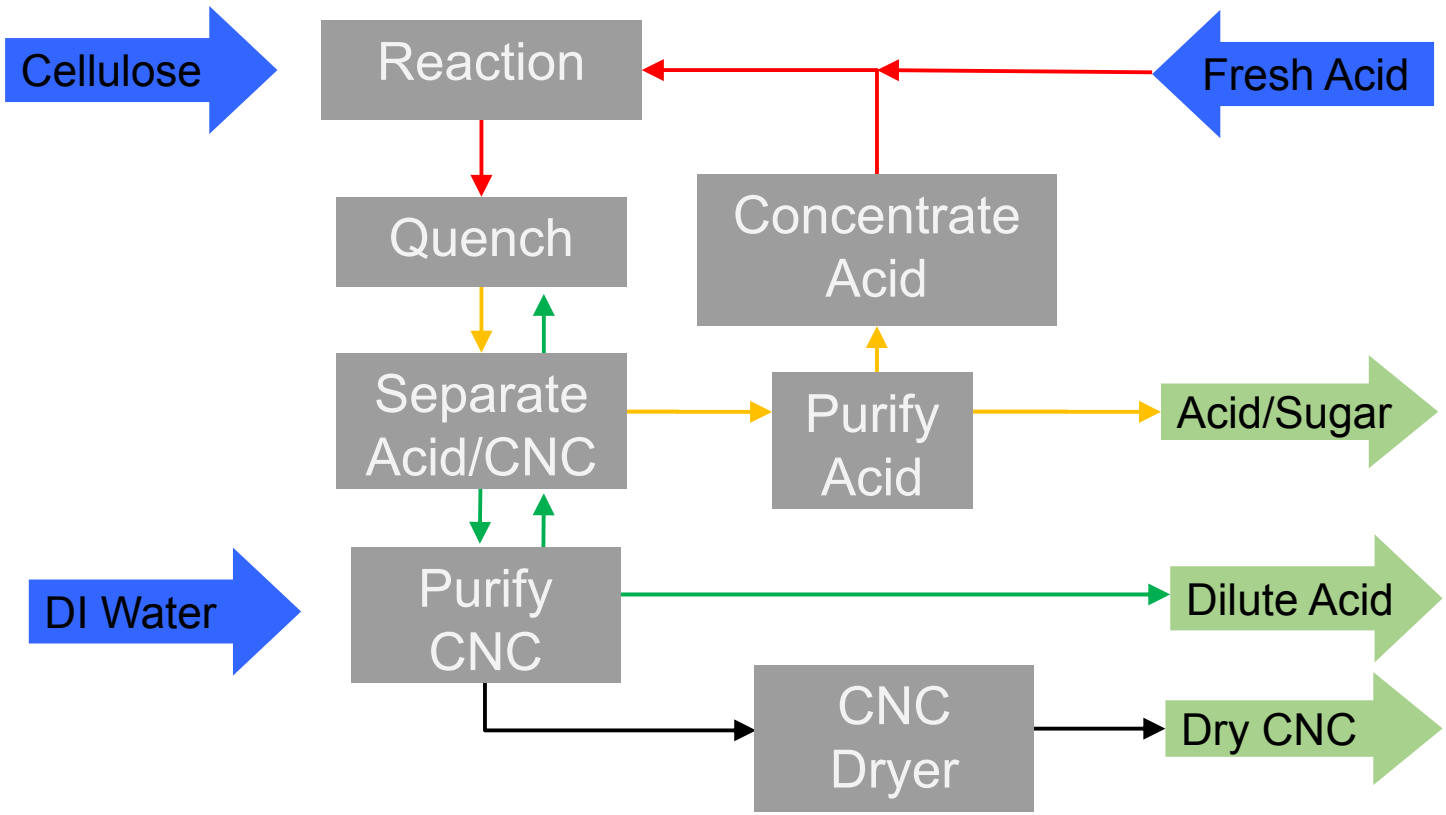
Design and Technology
CNC, Cellulose Nanocrystals

PDP™
Recovery Boiler
Precipitator Dust Purification

Simplified Process for CNC Production

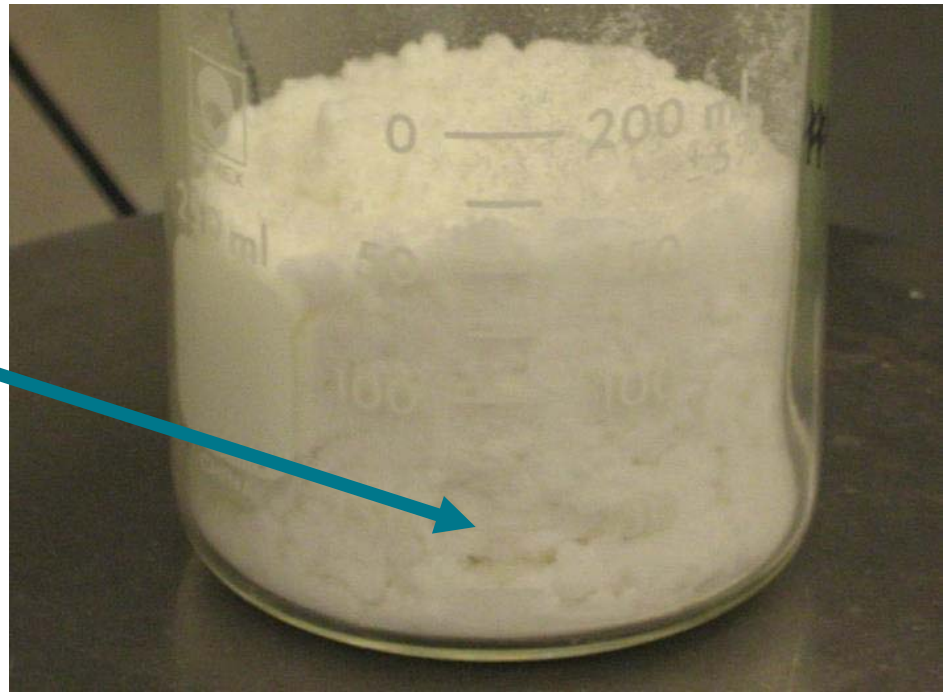


Need for a new approach for the reactor design

- Acid/Pulp reactors only ~5% of capex **but…**
  - Design drives acid use and recovery costs
    → which are **>25%** of CAPEX
- Also impacts:
  - Residence time distribution
    →Product yield & quality

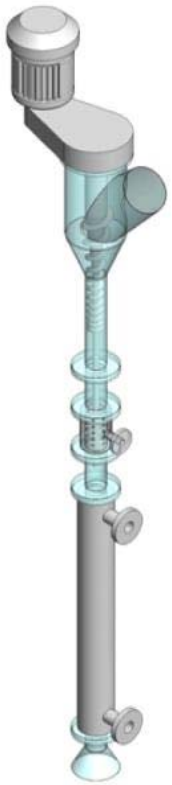
The crux of the problem

At 10x acid/pulp (wt/wt),
acid is ~1/3 of pulp volume



Very difficult to mix and contact well with acid

Continuous Compression CNC Reactor



Conceptual design & techno-economic study:

- Continuous reactor → smaller/cheaper
- Plug flow → improved residence time distribution
- Reduce acid & cost by ~3x
- Greater CNC potential when acid re-used in mills
- Increased capacity
- Reduced acid recovery costs:
  - Overall plant opex savings, ~10%
  - Overall plant capex savings, ~25%

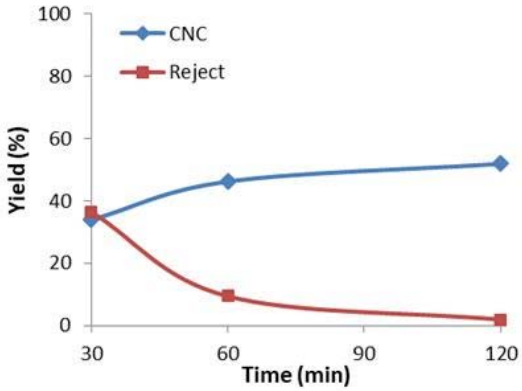
PCT Patent Application PCT/CA2012/050658, 2012.

Potential improvement

for 1 kg of pulp containing ~6 wt% moisture

	Traditional approach	Compressed pulp
pulp density (kg/L)	0.05	0.50
pulp volume (L)	**20.0**	**2.0**
Solids (L)	0.6	0.6
Void (L)	19.4	1.4
60 wt% acid (kg)	10.0	3.3
100 % H_2SO_4/dry pulp (kg/kg)	6.4	2.1
100 % H_2SO_4/AGU (mole/mole)	10.6	3.5
acid volume (L)	**6.7**	**2.2**
effective acid strength (wt%)	59.6	58.9
mixture solids content (wt%)	8.5	21.7

Continuous Compression CNC Reactor



Yield versus time for 58wt% acid at 56°C

Lab scale testing for proof of concept:

- ~3x lower acid use possible
- Rapid wicking of acid by compressed pulp facilitates initial contacting
- No mixing required after initial contacting
- Tested acid conc. (58-64 wt%), reaction time (30-120 min.) & temp (45-65°C)
- Characterization showed CNC properties and yield matching results for high acid ratios
- CNC yield: ~35-70% depending on reactor conditions

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Continuous Compression CNC Reactor

Design of Continuous Demonstration Scale Reactor:

- 12” ID x 9ft height: ~200L
- ~60min at ~1 TPD
- Variable acid conc. & temperature
- Up-flow design due to layout constraints (required pump)
- Low cost / manual system for testing

Continuous Compression CNC Reactor Development Process

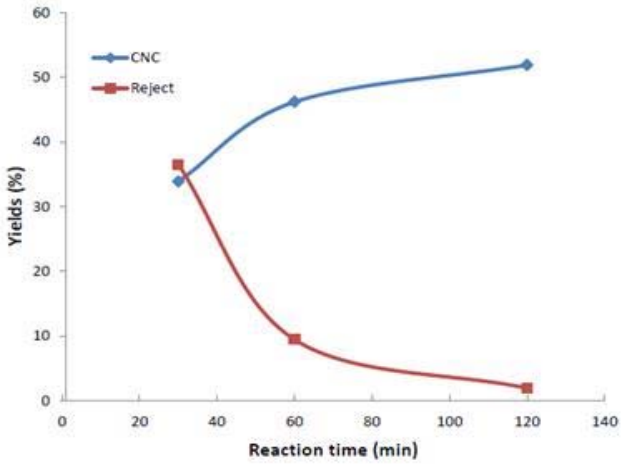
Construction & initial testing of compression and wetting at BC Research:

- Good acid distribution/wetting

Continuous Compression CNC Reactor Development Process

Demo unit product characterization:

- Sampled at pump discharge
- Slurry is a thick, homogeneous paste
- Held samples prepared with 61 wt% acid for different times at 65°C without mixing, then quenched
- Trend in CNC properties (Zeta potential, DLS), yield and rejects matched lab-scale results



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Continuous Compression CNC Reactor Development Process

Installation and Integration with InnoTech Alberta CNC Pilot Plant

Continuous Compression CNC Reactor Development Process (2018)

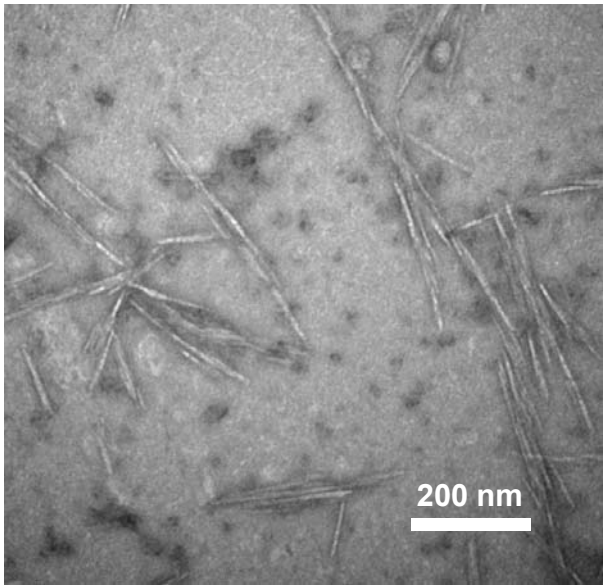
System installed & tested at InnoTech Alberta :

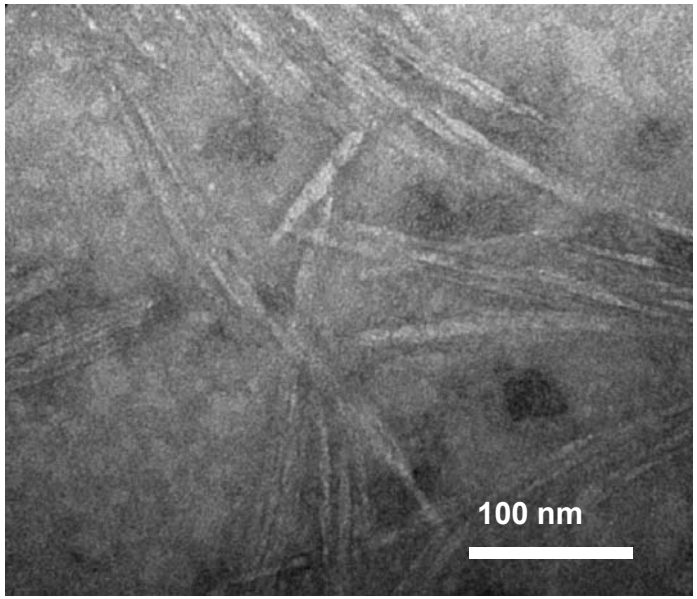
- Complete system with pulp compression, acid mixing, reactor & quenching
- Ran twice for ~1.5 hours
- Pressure trips cut runs shorter than planned

Product characterization from first demo run (2018)

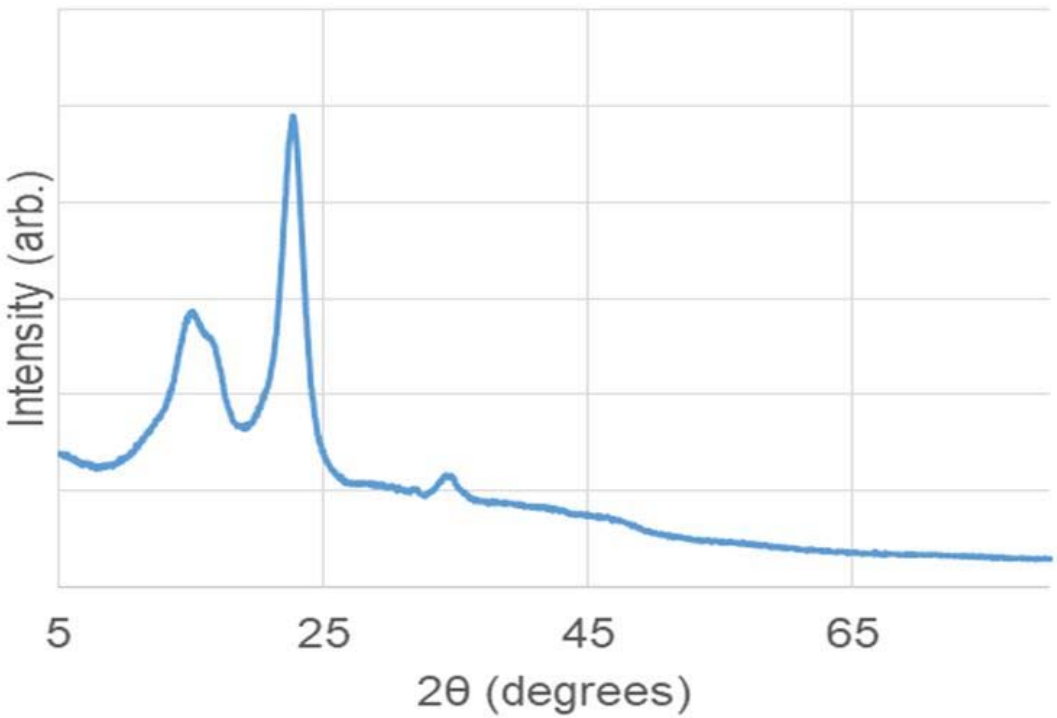
- Zeta potential of –40-50 mV
- Hydrodynamic radius (DLS): 85-110 nm

- TEM





Product characterization from first demo run (2018): XRD



- Estimated crystallinity (Segal method): 85-90%*
- Overall CNC properties appear similar to conventional high acid process

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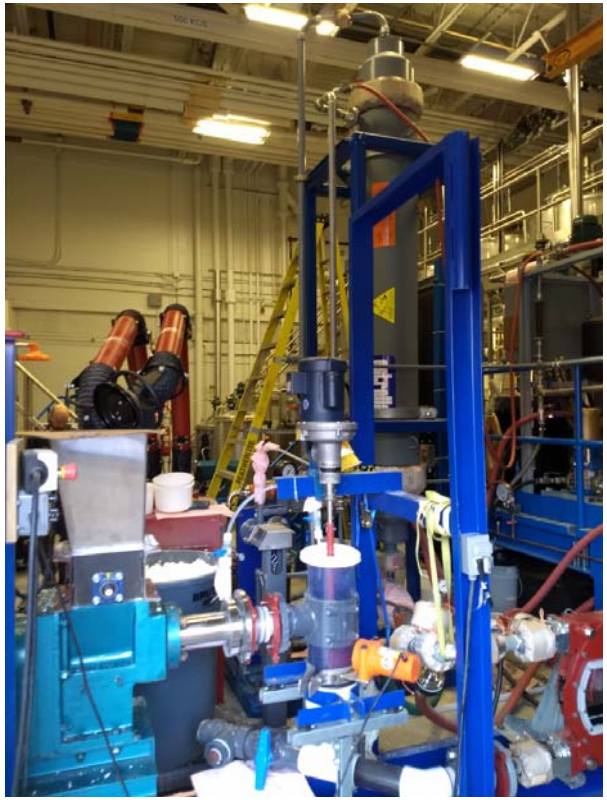
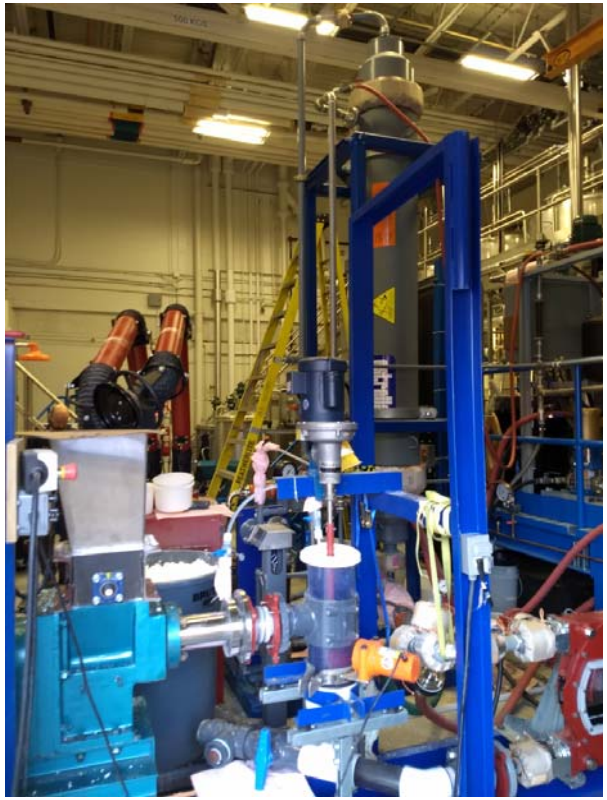
Continuous Compression CNC Reactor Development Process (2019)



- Improvements made to eliminate pressure trips
- Tested different pulp feedstock (dissolving pulp versus previous proprietary pulp)
- Feed system required modification due to much lower density feedstock more prone to bridging
  - Reduced pulp mass flow rate (~0.5 TPD or ~20 kg/h)
  - Increased residence time (~2 hr)

Continuous Compression CNC Reactor Development Process (2019)

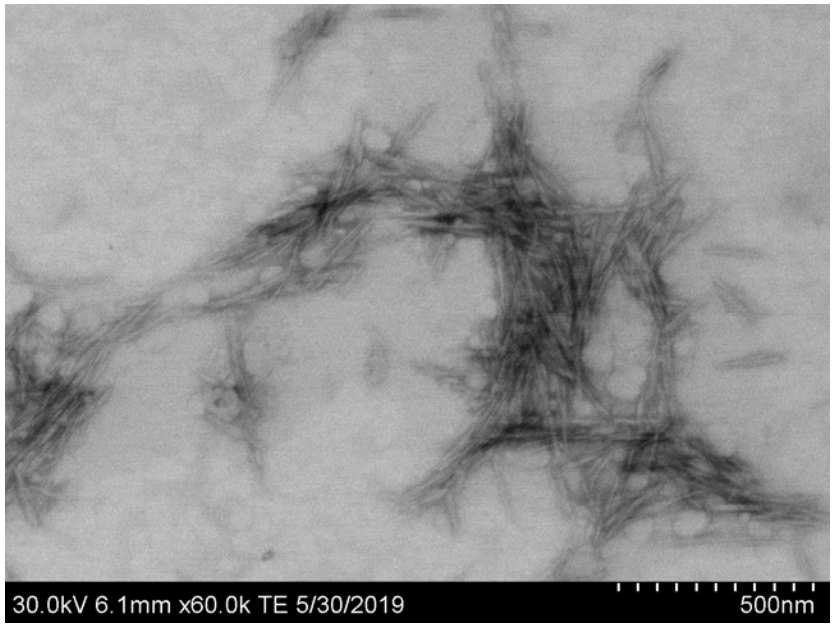


- Feed system maintained good pulp compression and acid dispersion / wicking with new pulp
- Ran two campaigns at 3 & 5 hours
  - 5 hr run stopped after storage tanks filled
- Ran with ~2.7 pure acid weight ratio (61 wt% sulfuric acid/pulp)
- Reactor conditions 55°C and 120 min.

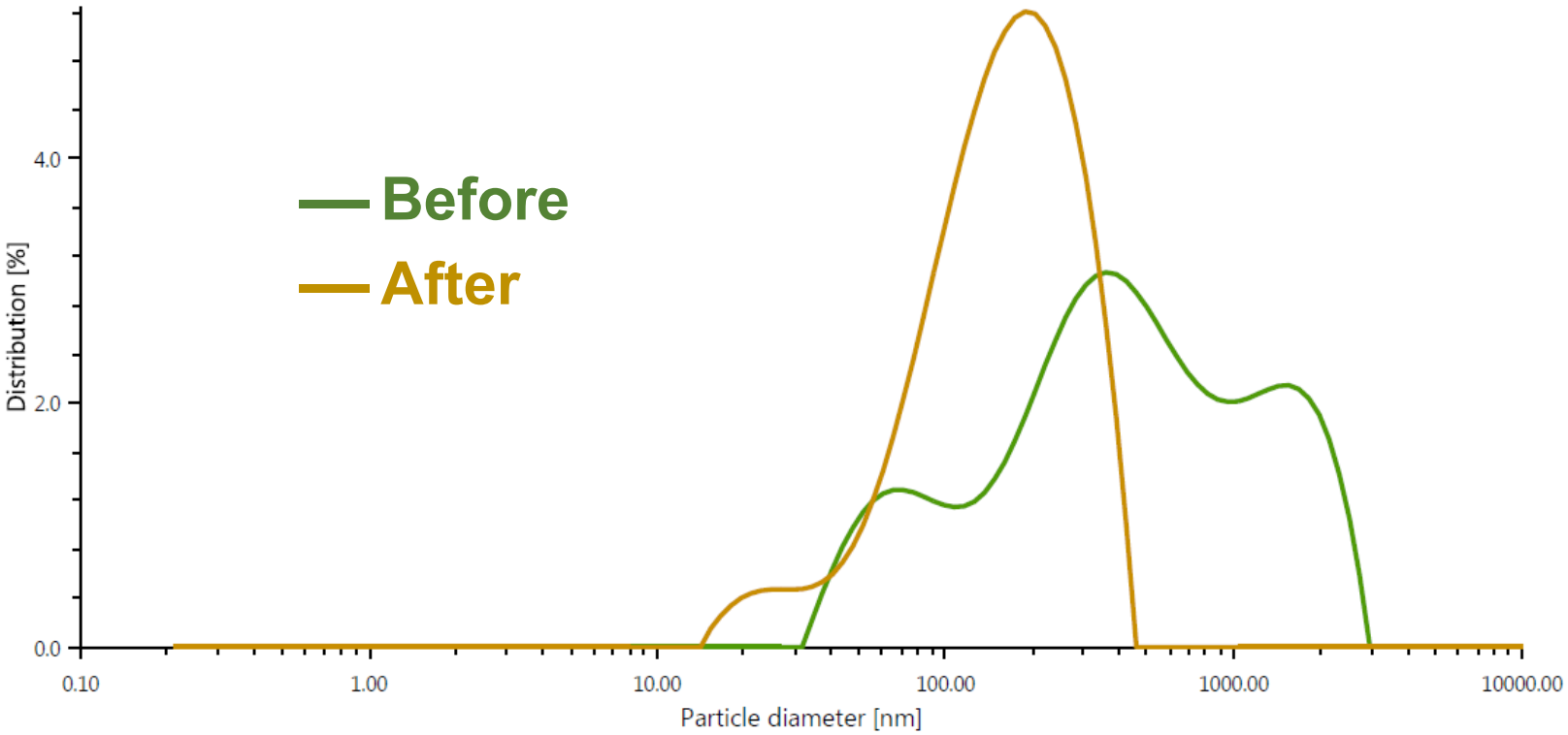
Product characterization

- **Low variability in CNC produced over time (30, 120, 180 min):**
  - **Zeta potential between samples: -42 to -46 mV**
  - **Hydrodynamic radius (DLS): Sonicated: 122 to 140 nm**

- **TEM (180 min. sample)**



Product characterization
PSD before and after sonication (180 min. sample)



Product characterization from first full demonstration scale run:

- Yield ~55% CNC and ~2% oversized material (reject) from starting material characterized as containing 61% crystalline cellulose
- Overall yield and CNC properties appear very similar to conventional high acid batch process

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Summary:

- Design, construction and commissioning of demonstration scale continuous compressed cellulose CNC reactor has been completed at 0.5-1 t/d size
- Demonstrated continuous pulp compression and acid wetting with two different pulps.
- Achieved continuous operation for over 5 hours
- Demonstrated ~2-3x reduction in acid consumption versus traditional batch process
- Product characterization indicates high quality CNC

Acknowledgements:

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Thank you

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