Electrochemical Dewatering of Cellulosic Nanomaterials

Santosh H. Vijapur, Huong Le, Santosh More, Timothy D. Hall, EJ Taylor, Maria Inman, & Stephen Snyder Faraday Technology, Inc.



Kim Nelson GranBio USA



Robert Handler Michigan Tech University





santoshvijapur@faradaytechnology.com



Nanocellulose





Problem/Opportunity

Challenges:

- Cellulosic materials contain significant water content
- Not economical to ship long distances





Opportunity:

- Development of novel electrochemical technology to dewater cellulosic materials
 - $\circ\,$ Maintain the material properties when dried and re-dispersed
 - \circ Economical, scalable, and energy efficient

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Key Results To Date

- 1. ElectroDewatering uses electro-osmotic, electrophoresis, and viscosity moderated process.
- 2. Achieves high solid content
 - a) Up to 70 wt.% solid content
 - b) ~25 wt.% solid content without hornification (batch)
 - c) ~17 wt.% solid content without hornification (alphascale continuous)
- 3. Highly efficient
 - a) 50% and 72% reduced energy requirements for CNF and CNC
 - b) 31% reduction in cost per ton for processing CNC
 - c) 51% reduction of emissions for processing CNC
- 4. Can be batch or continuous process

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ElectroDewatering Apparatus

- Continuous processing
- Demonstrated at 0.6 to 2 tpy
- Scalable to
 - 20 tpy (Current Goal)
 - 200 tpy (Pilot scale)
 - 2000 tpy (Full scale commercial)
- Other potential applications
 - Municipal Wastewater
 - Black liquor
 - Algae
 - Food processing products and wastes
 - Coal fines
 - Refuse slurries
 - Sewage sludge



"Method and Apparatus for Electrochemical Dewatering of Suspensions of Cellulosic Nanomaterials" US Pat. Appl. No. 62/842,037, filed U.S. utility on March 10, 2020; PCT Patent Application No. PCT/US20/30232, filed April 28, 2020



Technical Approach

DeWatering Mechanism	Function	Energy Requirement
Water Consumption:		
Joule Heating	Water vaporization	HIGH
Electrolysis	Water electrolysis	HIGH
Separation/Transport:		
Electroosmotic	Water transport to cathode	LOW
Electrophoretic	CNC-CNF transport to anode	LOW
Viscosity	Rotation rate effects: wall slip, shear banding	LOW

- Utilizes a combination of electrochemically driven H₂O consumption and/or CNF-CNC/H₂O separation mechanisms
- Minimize structural damage of the nanocellulose
- Low-energy process by emphasizing low-energy separation mechanisms





Preliminary Batch-Scale



Sub-scale batch reactor



Sub-alpha scale batch reactor

- 25 mL beaker with paddle wheel (sub-scale batch reactor)
- 50 mL beaker with paddle wheel (sub-scale batch reactor)
- 100 mL screw extruder based batch (sub-alpha scale batch reactor)





Range of Final Solid Content



Characterization

- Dispersionanalysis:Re-dispersion occured at ~ 0.2 wt%in water using a vortex mixer for2-10 minutes
 - Full redispersion required in 10 minutes
- Nanocellulose structure/size: Optical microscopy
 - Hornified/un-redispersed samples were rejected



Un-dispersible CNCs at ~61 wt.% solid content



Re-dispersible CNCs at ~25 wt.% solid content





Process Scale-Up

- Batch beaker with paddle wheel
 - Sub-scale reactor
- Screw extruder based batch to continuous system
 - Sub-alpha scale batch
 - Alpha-scale continuous (up to 2 ton/yr)



Sub-scale batch reactor



Sub-alpha scale batch reactor



Alpha scale continuous reactor





TAPP

Alpha-scale Apparatus

"Method and Apparatus for Electrochemical Dewatering of Suspensions of Cellulosic Nanomaterials" US Pat. Appl. No. 62/842,037, filed U.S. utility on March 10, 2020; PCT Patent Application No.

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Energy Comparison

- Observed 50% and 72% energy saving compared to the latent heat of vaporization calculation for CNF/CNC
- Alpha-scale system can process:
 - 0.6 tpy dried CNF
 - 1.3 to 2 tpy of dried CNC





Energy Comparison

- ElectroDewatering shows similar solid content versus energy regardless of type of operation batch or continuous (2 tpy)
- Dewatering mechanism seems to change with solid content, where
 - o Low energy separations (electroosmotic/ electrophoretic) dominates up to 20 wt.% solids
 - \circ Higher energy separations (electrolysis / joule heating) are required for achieving >25 wt.% solids



Life Cycle Analysis (LCA)

Life cycle analysis will be utilized to improve ElectroDewatering method and apparatus such that these processing steps can be transitioned to an industrial scale



* Client confidentiality, unable to reveal the Methods

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- ElectroDewatering provides significantly less global warming potential than dewatering method 1 for CNC
- 51% reduction in GHG emissions

Techno-Economic Analysis (TEA)

Techno-Economic analysis will be used to improve ElectroDewatering method and apparatus such that these processing steps can be transitioned to an industrial scale

Initial % solids	Dewatering Step (% output solids)		Drying Step (% output solids)		
CNF					
3%	Method 1 (100%)				
3%	Method 2	(30%)	Method 1	(100%)	
3%	Method 3	(23%)	Method 1	(100%)	
3%	ElectroDewatering	(14%)	Method 1	(100%)	
3%	ElectroDewatering	(18%)	Method 1	(100%)	
CNC					
7%	Method 1 (100%)				
7%	ElectroDewatering	(14%)	Method 1	(100%)	

TEA Scenarios

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- ElectroDewatering process works best for CNC
- 31% reduction in cost per ton of dried CNC

Summary

• Demonstrated ElectroDewatering process for dewatering of cellulosic materials

• Achievements:

- Design/build of alpha-scale continuous ElectroDewatering system
 - System throughput of up to 2 tpy of dried cellulosic nanomaterials demonstrated
- > Validated dewatering performance for cellulosic nanofibrils (CNF) and cellulosic nanocrystals (CNC)
 - $\circ~$ Up to 70 wt.% solid content
 - $\circ~$ ~25 wt.% solid content without hornification in batch operation
 - $\circ~\sim\!\!17$ wt.% solid content without hornification in alpha-scale continuous operation
 - CNC/CNF maintained structural integrity after re-dispersion
 - 50% and 72% reduced energy requirements for CNF and CNC
 - TEA suggested a 31% reduction in cost per ton for CNC
 - $\circ~$ LCA suggested a 51% reduction of emissions for CNC



Next Steps

- Investigate ElectroDewatering approach for:
 - Other cellulosic materials
 - Pharmaceuticals

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- Waste treatment, etc.
- Modify ElectroDewatering system design to:
 - Reduce energy consumption
 - Increase solid content without hornification
- Scale process to 20 tpy dry cellulosic materials
 - $\circ~$ Target demonstration for CNC is FY24





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THANK YOU FOR YOUR ATTENTION! QUESTIONS?



Contact Information: Santosh Vijapur, Tim Hall, or Maria Inman Ph: 937-836-7749 Email: <u>santoshvijapur@faradaytechnology.com</u> <u>timhall@faradaytechnology.com</u> <u>mariainman@faradaytechnology.com</u>

