What is happening at **UMaine's** Laboratory of Renewable Nanomaterials

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Nutting Hall, Orono, Maine

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FBRI FOREST BIOPRODUCTS Research Institute

SCHOOL OF FOREST RESOURCES forest.umaine.edu

ADVANCED STRUCTURES & composites center

Current Research @LRN

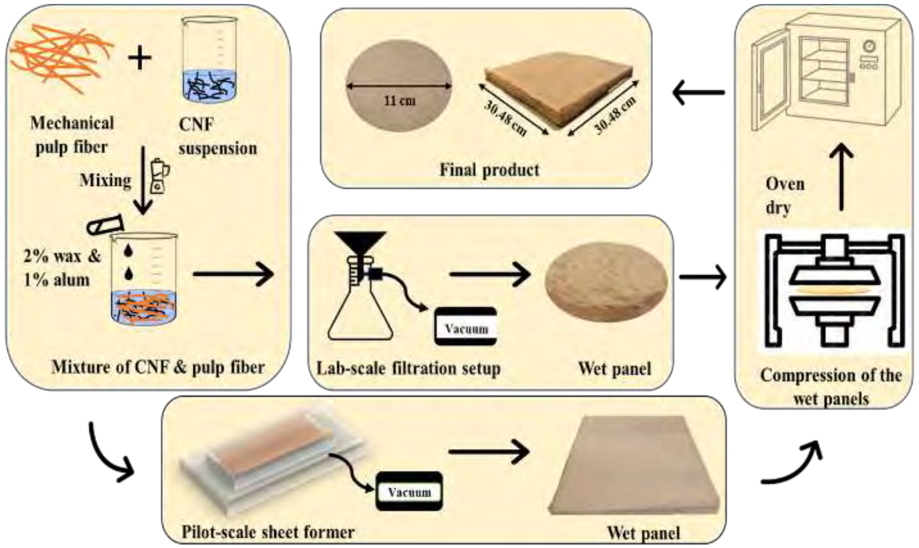
- Building/construction applications
- Packaging (protective/barrier) applications
- Water treatment applications

Building/construction applications

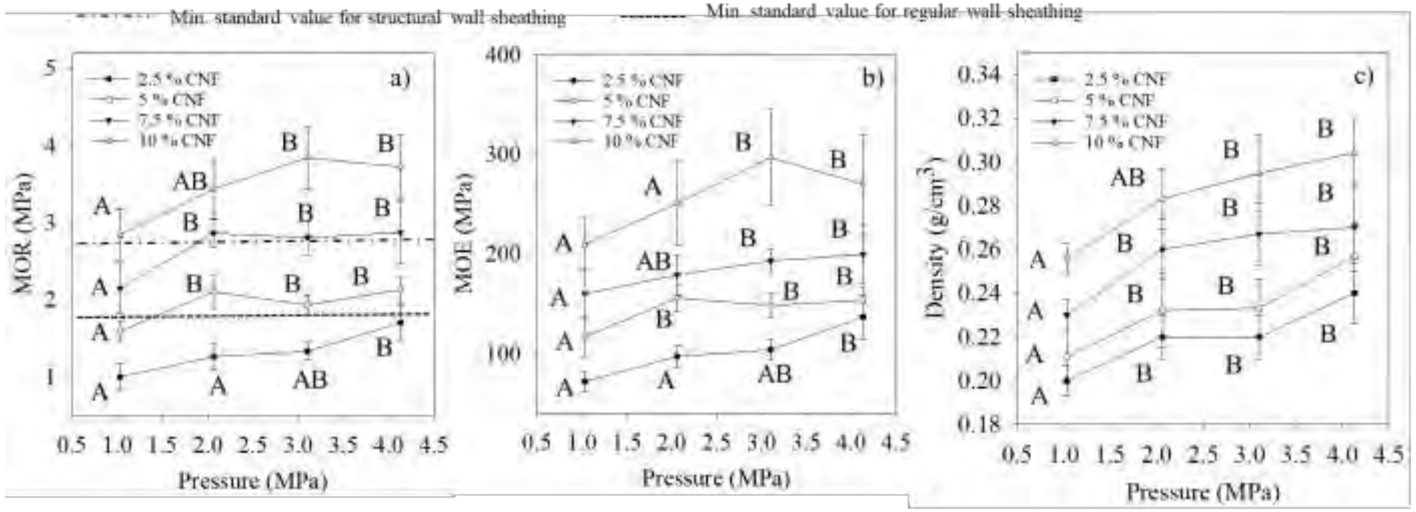
Upgrading regular wood-fiber insulation panels to structural wall sheathing enabled by cellulose nanofibrils

Lead: Rakibul Hossain

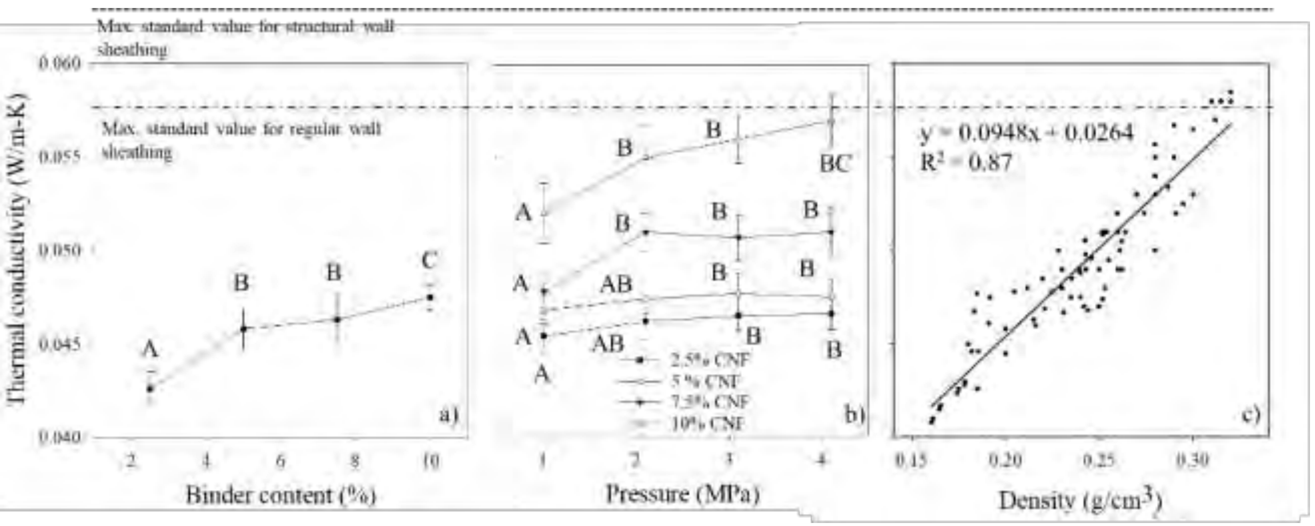
Panel making process (lab and pilot scale)



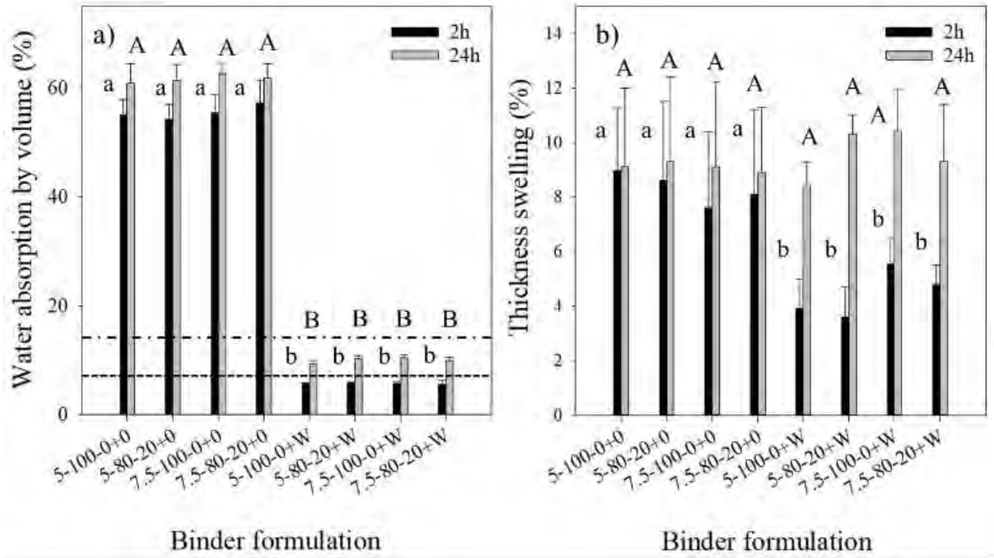
Mechanical properties of the pressure-controlled panels



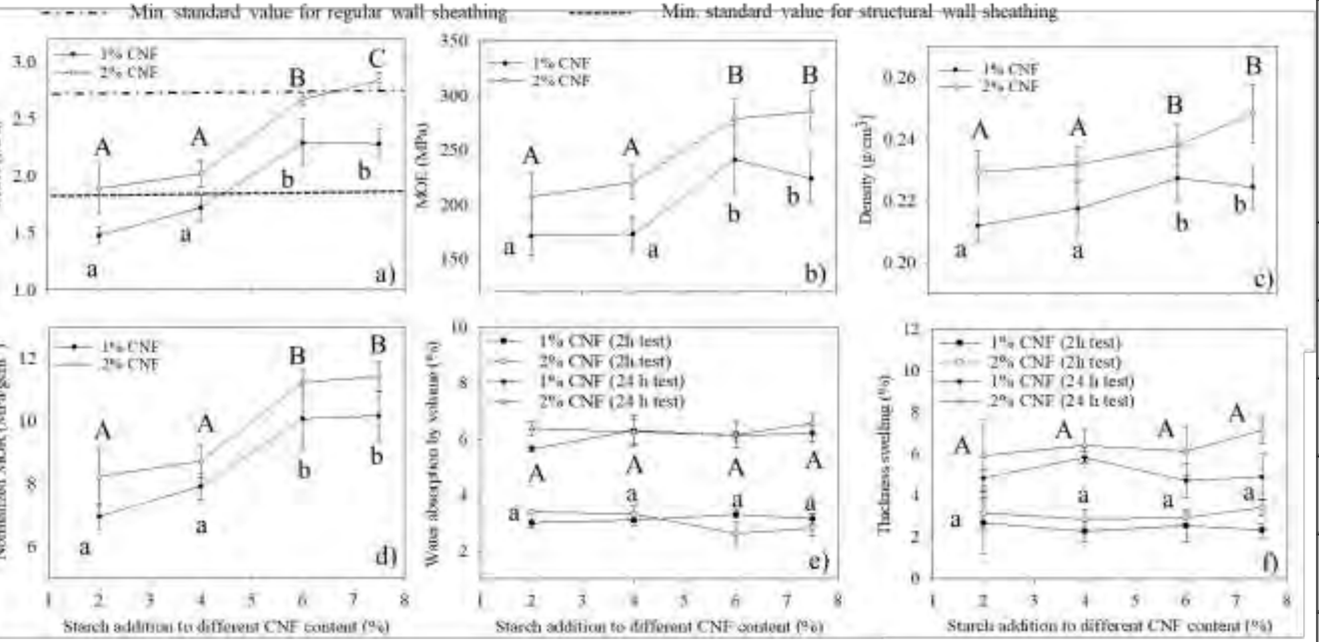
Thermal conductivity



Water absorption & thickness swelling



Properties of the panels made with starch-CNFs as binder



Industrial scale trial

Properties of the panels made in the pilot-scale

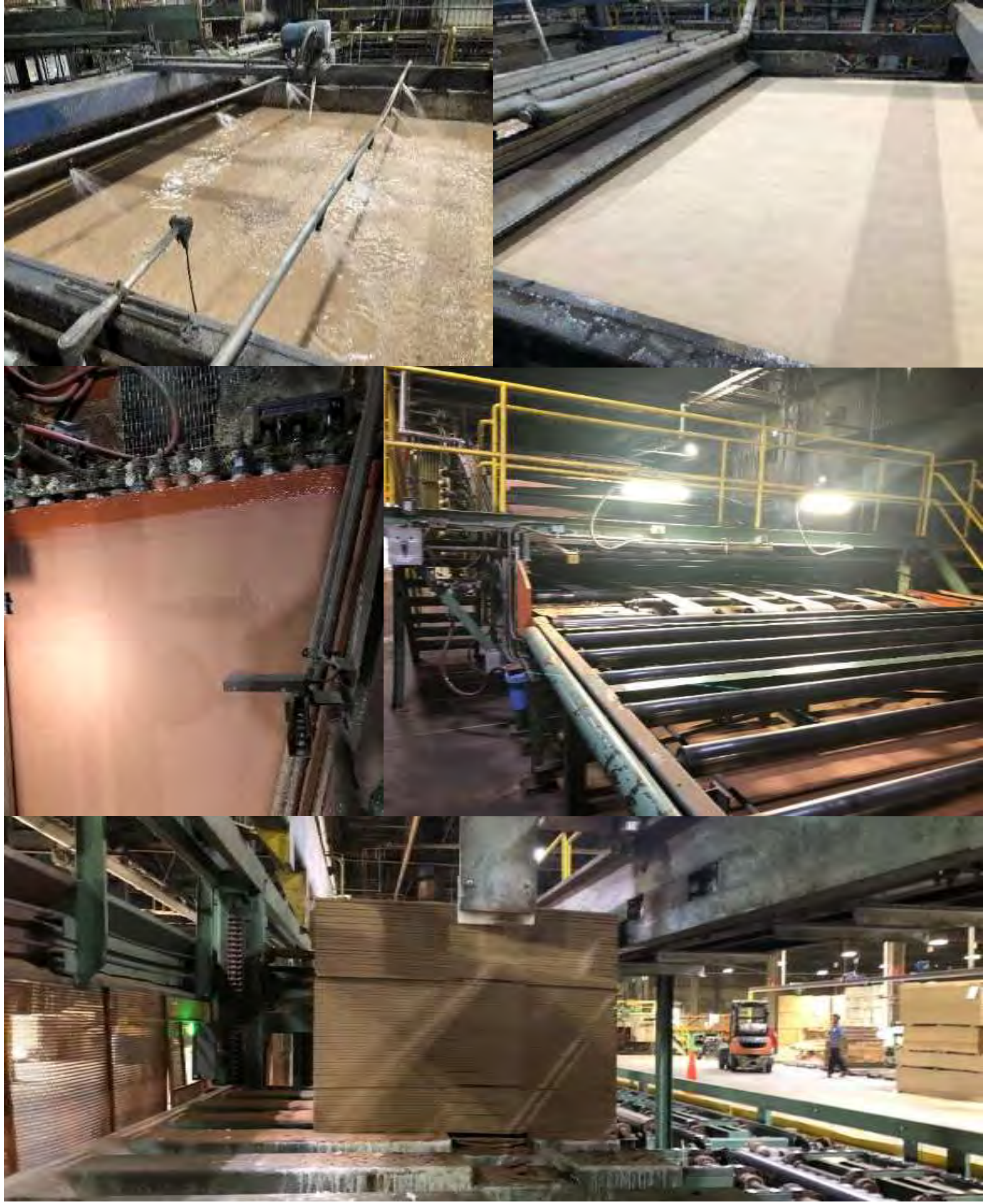
Properties	5 % binder (no wax)	5 % binder (2% wax)	7.5 % binder (no wax)	7.5 % binder (2% wax)	ASTM standard Type IV Grade 1 (regular)	ASTM standard Type IV Grade 2 (structural)
Density (g/cm3)	0.24^{a} (4.2 %)	0.25^{a} (4.0 %)	0.26^{b} (4.6 %)	0.26^{b} (3.8 %)	0.16 – 0.497	
Flexural MOR (MPa)	2.44^{a} (7.4 %)	2.29^{b} (4.8 %)	3.40^{c} (4.7 %)	2.86^{d} (6.6 %)	1.896	2.758
Flexural MOE (MPa)	237^{a} (18 %)	246^{a} (7.2 %)	326^{b} (5.4 %)	301^{b} (8.5 %)	N/A	
Tensile strength (parallel) (MPa)	1.55^{a} (8.4 %)	1.27^{b} (6.1 %)	2.20^{c} (9.1 %)	1.86^{d} (6.7 %)	1.034	1.379
Tensile strength (perpendicular) (kPa)	119.3^{a} (6.9 %)	122.1^{a} (11 %)	190.1^{b} (6.3 %)	185.3^{b} (5.1 %)	28.7	38.3
Water absorption by volume (%) (For 2h test)	62.1^{a} (3.5 %)	3.70^{b} (5.5 %)	63.8^{a} (2.0 %)	3.88^{b} (8.8 %)	7 (max. for 2h)	N/A
Water absorption by volume (%) (For 24 h test)	67.0^{a} (2.9 %)	8.86^{b} (4.5 %)	66.6^{a} (2.6 %)	9.67^{b} (4.6 %)	N/A	15 (max. for 24 h test)
Thickness swelling (2h test) (%)	10.8^{a} (6.1 %)	2.35^{b} (20 %)	11.9^{a} (2.0 %)	2.53^{b} (12 %)	N/A	
Thickness swelling (24h test) (%)	11.8^{a} (1.8 %)	5.34^{b} (7.1 %)	12.0^{a} (3.1 %)	6.20^{b} (2.4 %)	N/A	
Thermal conductivity (W/mK)	0.047^{a} (1.3 %)	0.047^{a} (2.9 %)	0.049^{b} (4.3 %)	0.050^{b} (5.1 %)	0.058 (max)	0.063 (max)
Moisture content by weight (%)	8.0^{a} (2.8%)	7.8^{a} (1.9 %)	7.9^{a} (1.0 %)	7.8^{a} (1.9 %)	10 (max)	

Acknowledgement:

Industrial trial by the numbers

- 2 tons (4,000 lb) (dry basis) of CNFs produced at PDC, that is 66.7 tons @3% solids
- CNF was dewatered to 18% solids (22.2 tons)
- Shipped in 18 super sacs to trial location
- Over 170,000 square feet of panels (5,300 4 by 8 panels) were produced in a continuous process over 6 hours

BLUE RIDGE FIBERBOARD



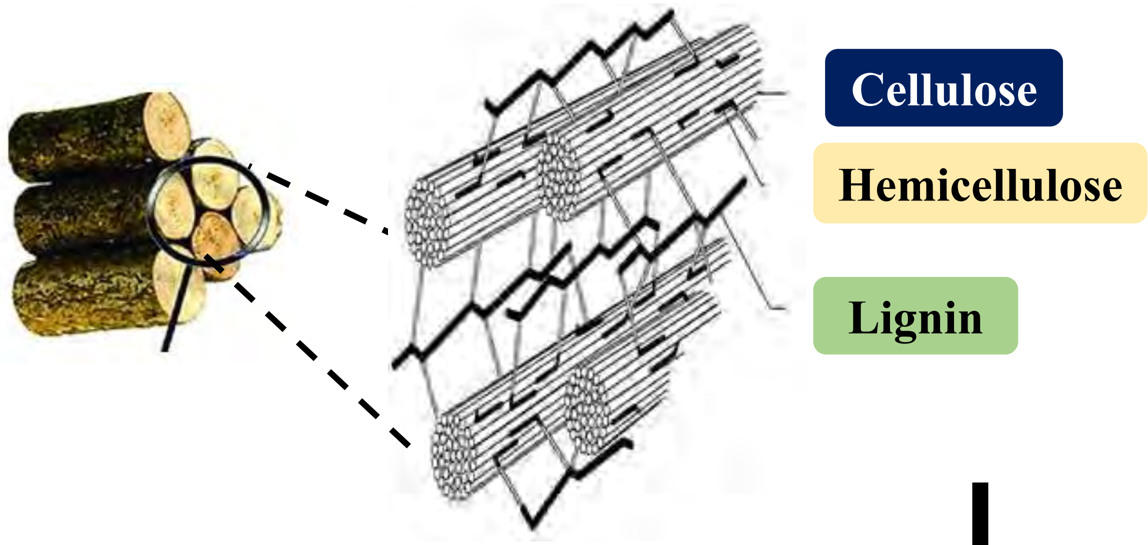
All Biobased (mycelium-based) insulation products for building and packaging applications

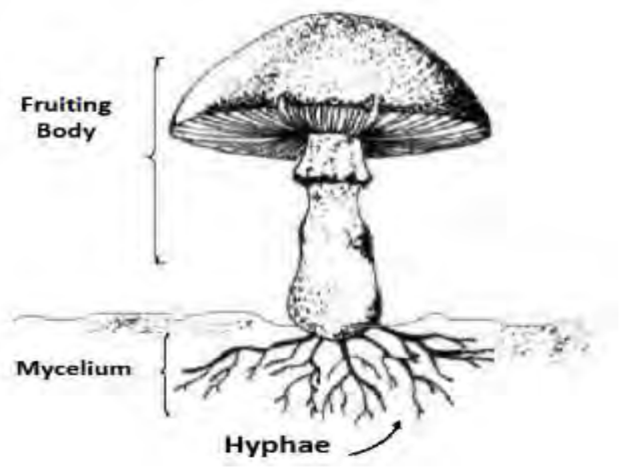
Lead: Maryam El-Hajam

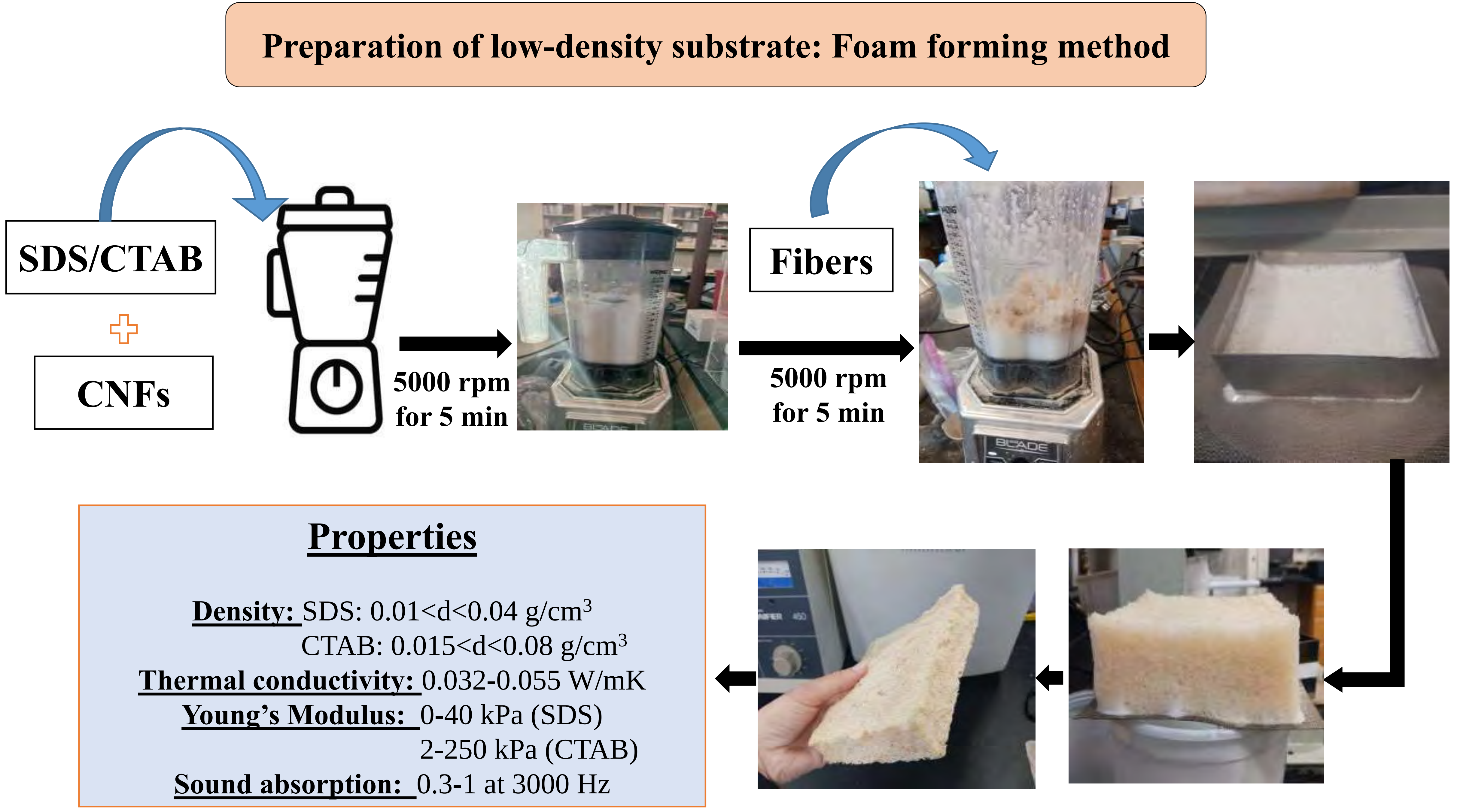
Eco-friendly, Non-toxic, Renewable, and Recyclable

Lignocellulosic materials

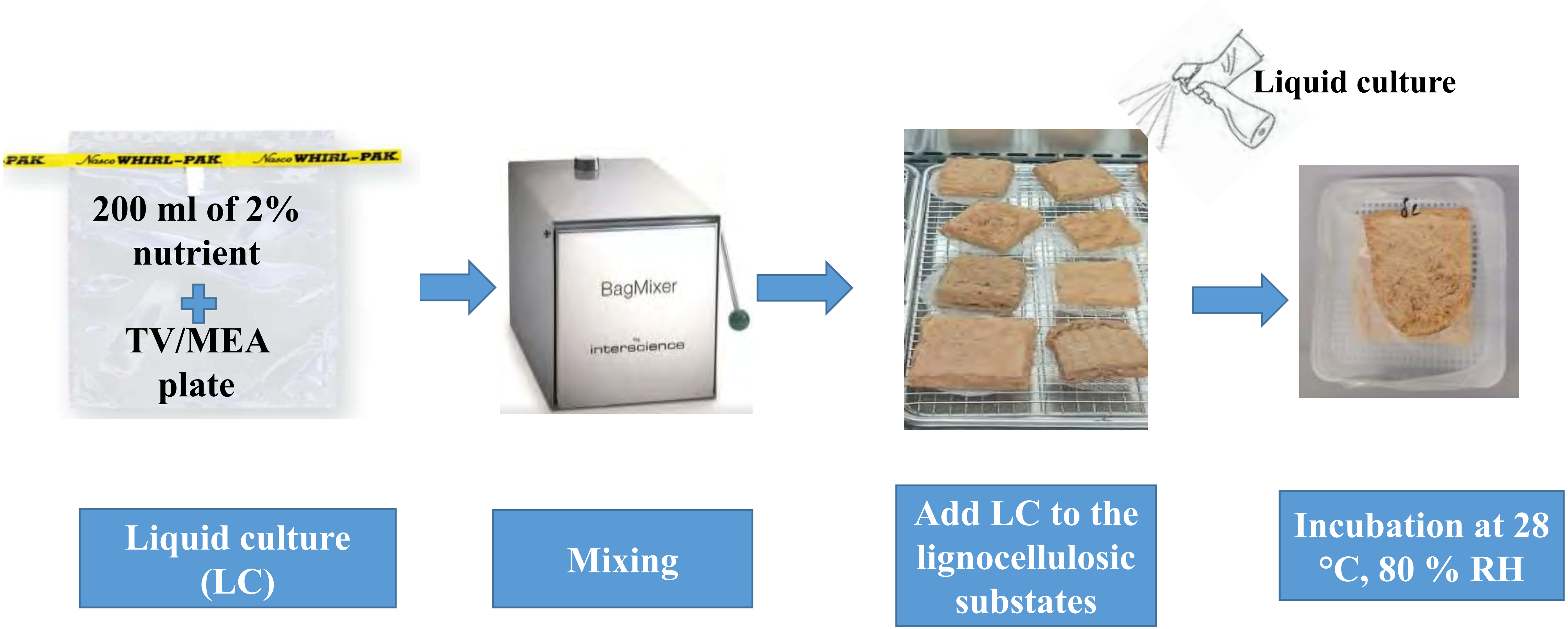
Fungal mycelium






Preparation of low-density substrate: Foam forming method
SDS/CTAB
CNFs
5000 rpm
for 5 min
Fibers
5000 rpm
for 5 min
BLADE
Properties
Density: SDS: 0.01<d<0.04 g/cm3
CTAB: 0.015<d<0.08 g/cm3
Thermal conductivity: 0.032-0.055 W/mK
Young's Modulus: 0-40 kPa (SDS)
2-250 kPa (CTAB)
Sound absorption: 0.3-1 at 3000 Hz

Preparation of lignocellulosic foam reinforced fungal mycelium



Constraints found during the mycelium growth

1. Effect of foaming agent → Washing ← Drying at high T to avoid the collapsing of the foams

2. The presence of borate → New raw TMP fibers without borate → A little growth ❌

Why?
Is it because our TMP is from **Softwood?**

Raw Softwood Pine particles (1-2 mm)

Raw Hardwood Maple particles (1-2 mm)

Packaging (protective/barrier) applications

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Cellulose nanofibril (CNF) enabled non-conventional food serving products

Lead: Mamoona Raheem

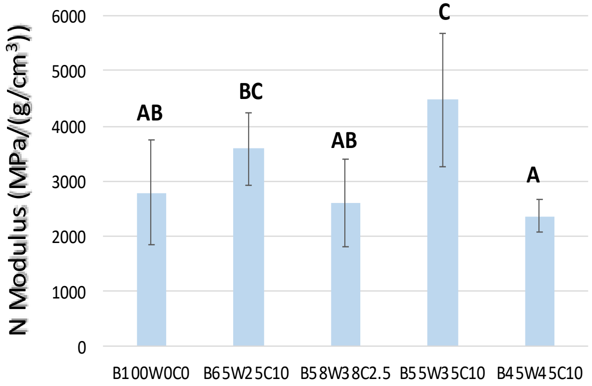
Fabrication of plates

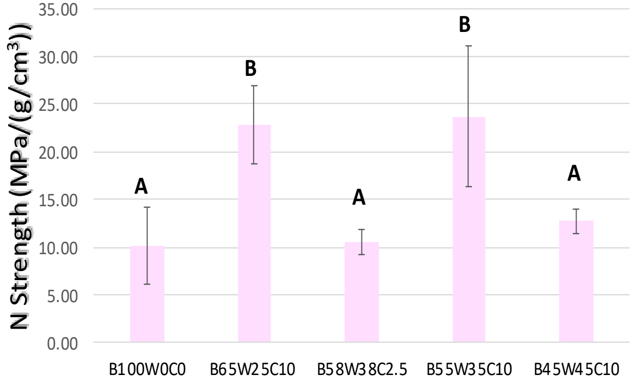
Raw material : Thermomechanical pulp (TMP), Wood Flour (WF), Bleached Kraft Pulp (BKP) and Cellulose Nanofibril (CNF)

Formulations tested

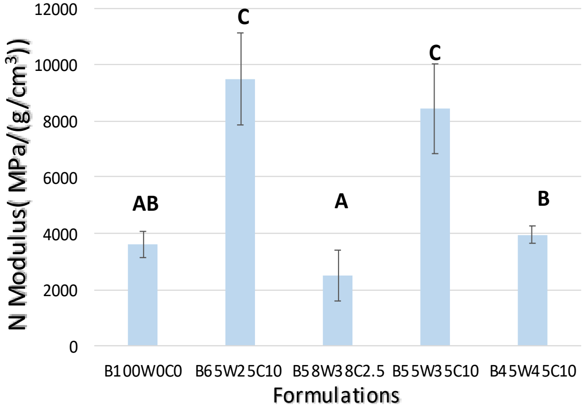
- BKP 100%
- BKP 45% WF 45% CNF 10%
- BKP 55% WF 35% CNF 10%
- BKP 65% WF 25% CNF 10%
- BKP 58.75 % WF 38.75 % CNF 2.5%

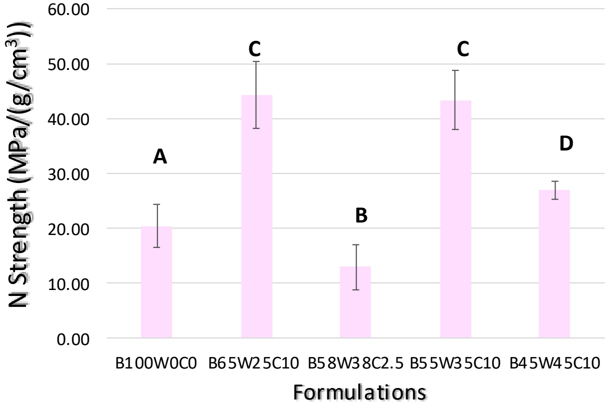
Tensile test





Flexural test





- BKP 55% WF 35% CNF 10% showed optimum results
- Order of addition does not affect the mechanical properties

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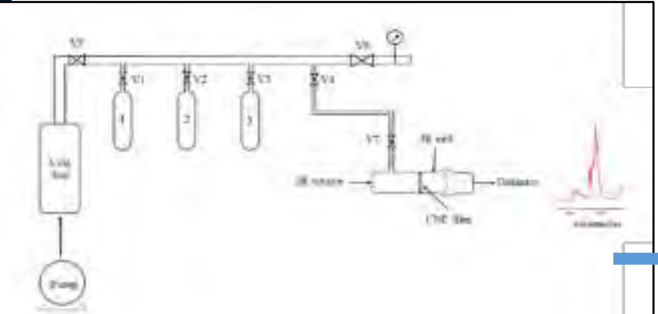
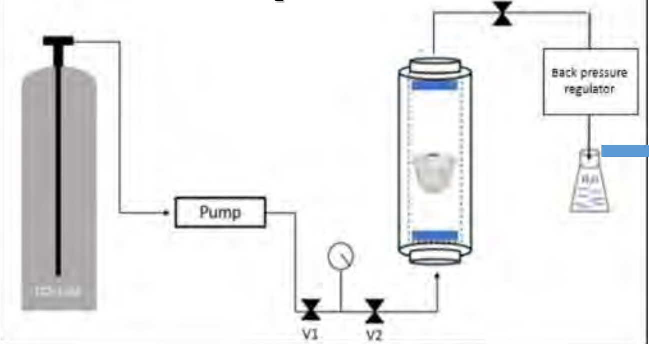
Post-surface treatment on plates

Reactions with hexamethyldisilazane (HMDZ) and hexamethylene diisocyanate (HMDI) with a catalyst



Reactions under Gas phase

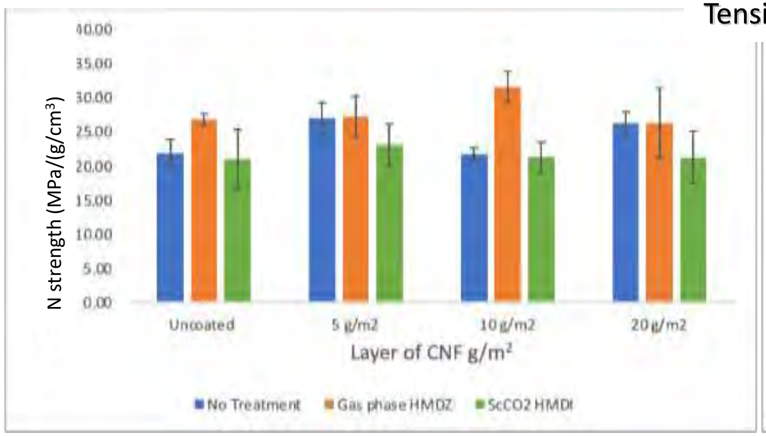
Reactions with Sc-CO_2 as a solvent

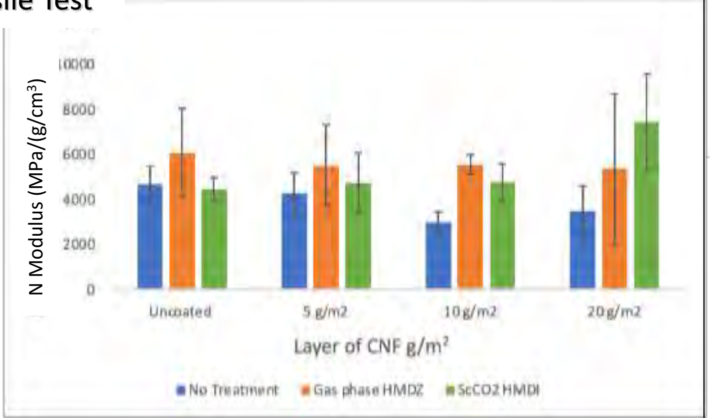


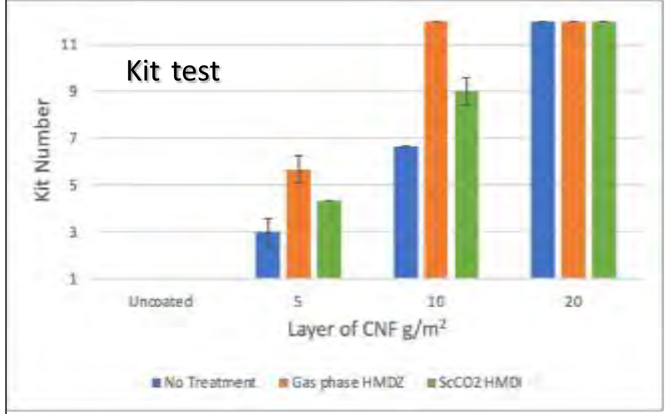
Untreated Treated with HMDZ

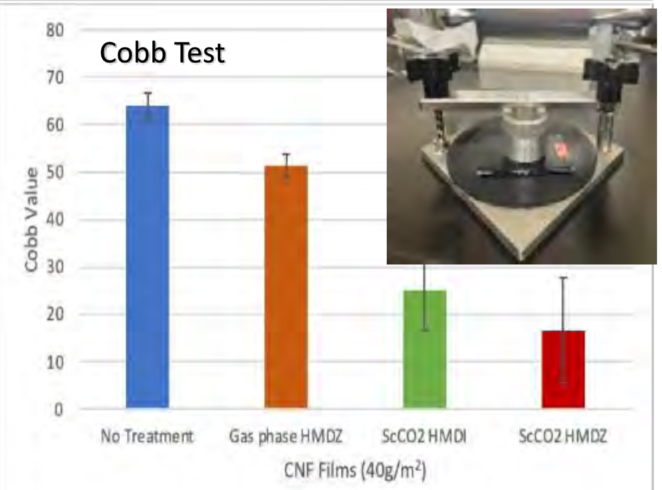
untreated HMDZ HMDI

Tensile Test









- Reactions performed under gas phase and Supercritical CO_2 showed increased Kit and reduced Cobb values as compared to the untreated samples.
- The gas phase (surface treatment with HMDZ) showed increased tensile strength while under supercritical CO_2 (crosslinking with HMDI) showed an increase in modulus.

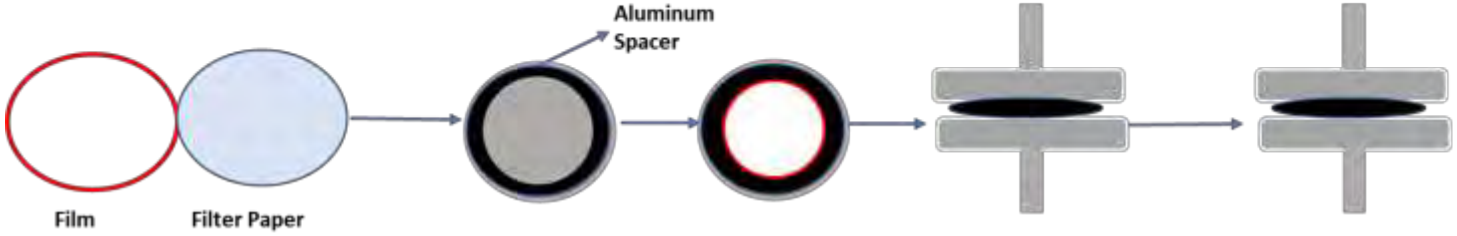
Enhancement of CNF barrier properties by nanofibril alignment

Lead: Nabanita Das

Method:

Auto-Dynamic Sheet Former — Sheet Press — Formed Film

XY – Z shrinkage drying



Filter Paper removed from wet film

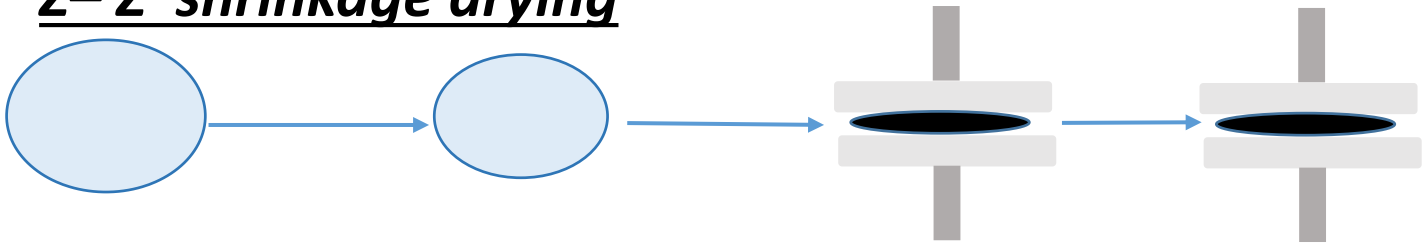
Steel Plate with spacer

The wet film placed between the steel plates

Hot Press the film (**pressure reading zero**) for drying at **150 °C for 8 minutes**

Hot Press Compaction of the film with **no filter paper, no spacer at 150 °C, and 1.1 MPa pressure for 4 minutes.**

Z– Z shrinkage drying



Filter Papers on both surfaces of the film

The stack of wet film and filter papers placed between the steel plates

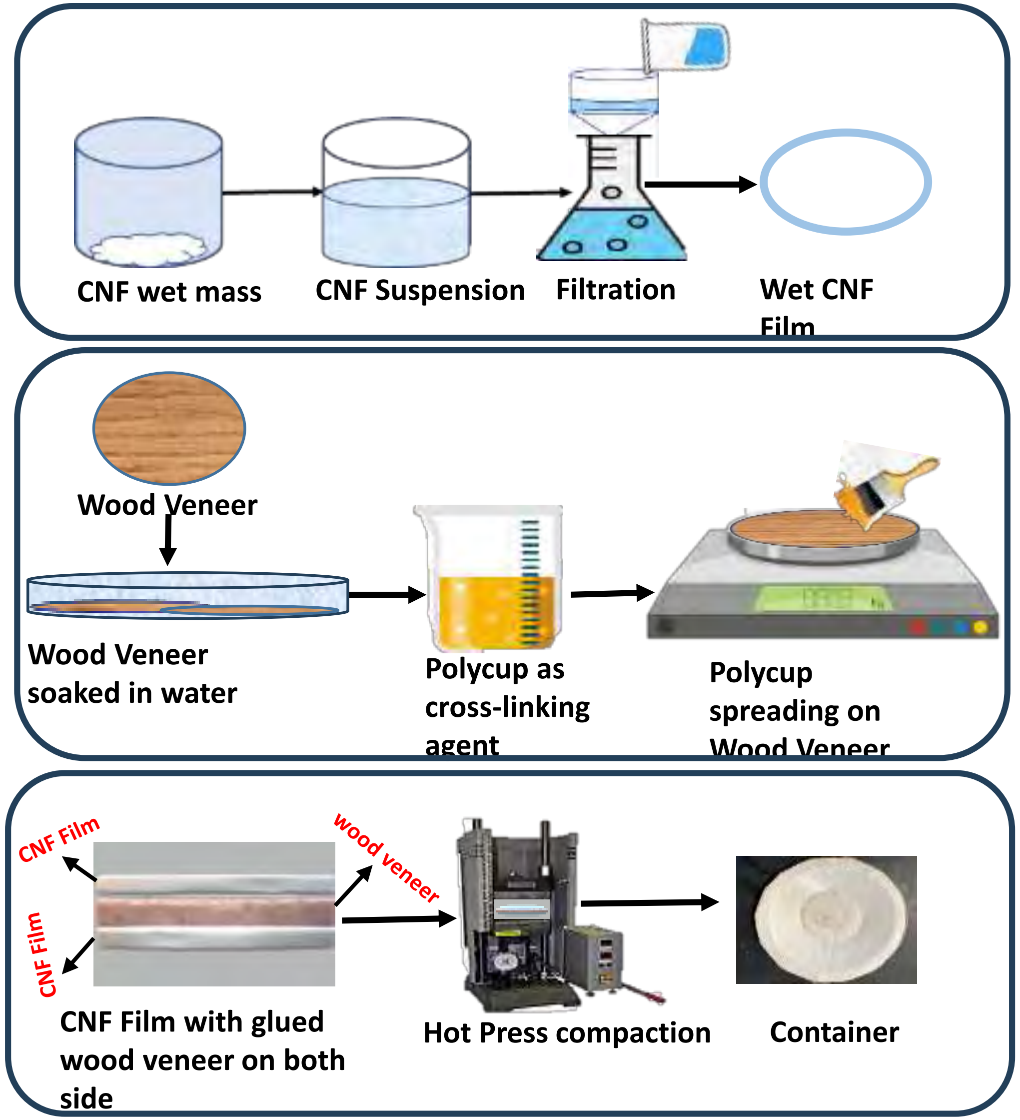
Hot Press the film (**pressure reading zero**) for drying at **150 °C for 8 minutes**

Hot Press Compaction of the film with **no filter papers at 150 °C, and 1.1 MPa pressure for 4 minutes.**

CNFs provided by the University of Maine Product Development Center at **3 wt. % solid content** and **90% fines** were used to make the film. In CNF suspension, three different solid contents, **0.1 wt.%, 0.2wt%, and 0.3 wt.%,** were used. Four levels of wire speeds **900 m/min, 1000 m/min, 1100 m/min, and 1200 m/min** were used to form the films

- Wire-speed – 900 m/min,1200 m/min
- Initial Water Wall – 2mm
- Water wall limit – 10 mm
- Compacting time – 4 min
- Compacting speed – 1400 rpm
- Average flow rate – 2.20 l/min
- Cold press pressure – 30 psi.

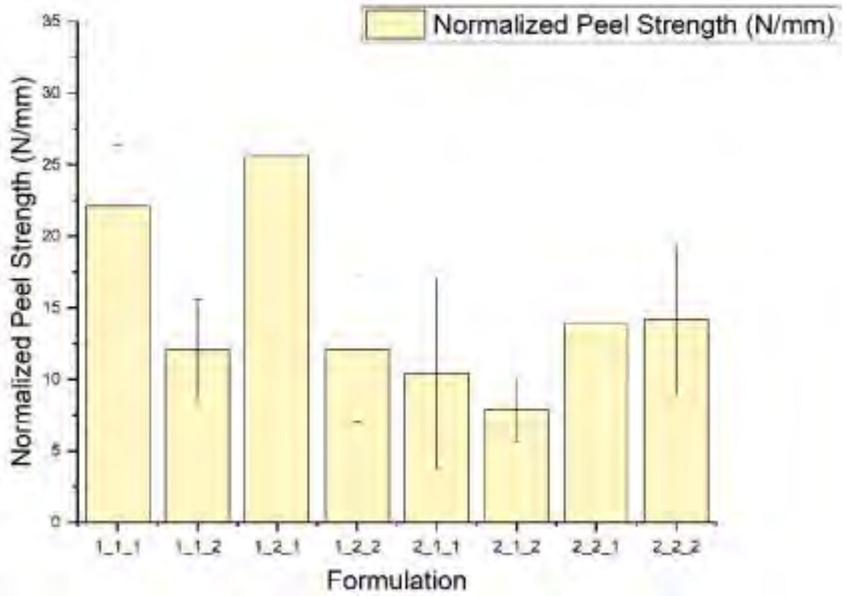
Multi-layer oil and water-resistant food serving container made using CNF laminated wood veneer

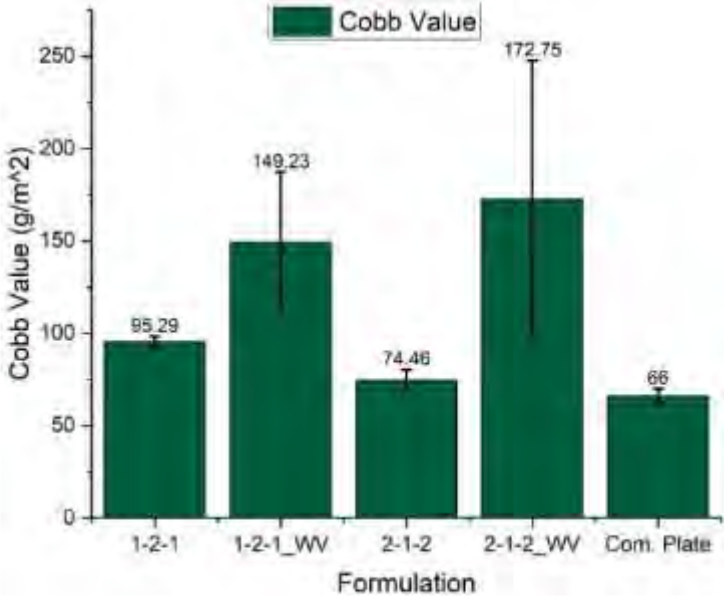


Preparation of wet CNF Films

Preparation of Wood Veneer

Preparation of Food Container

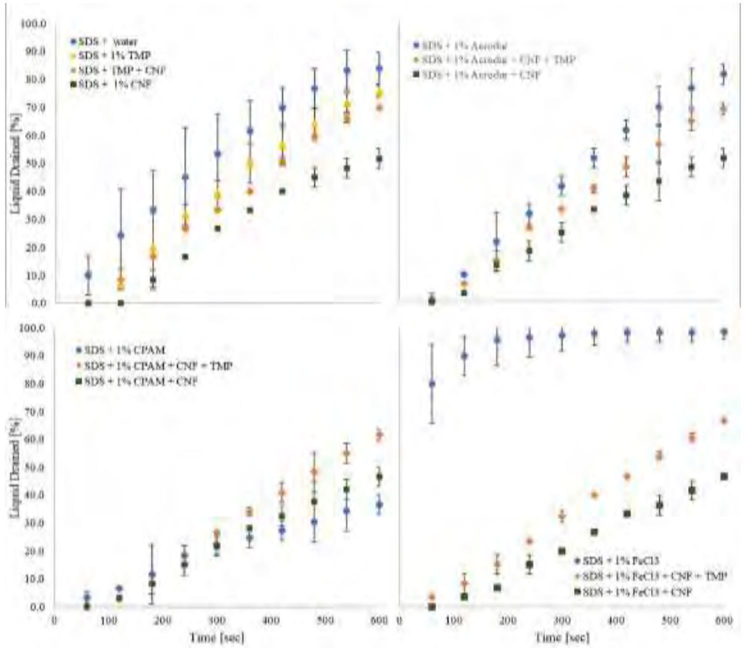




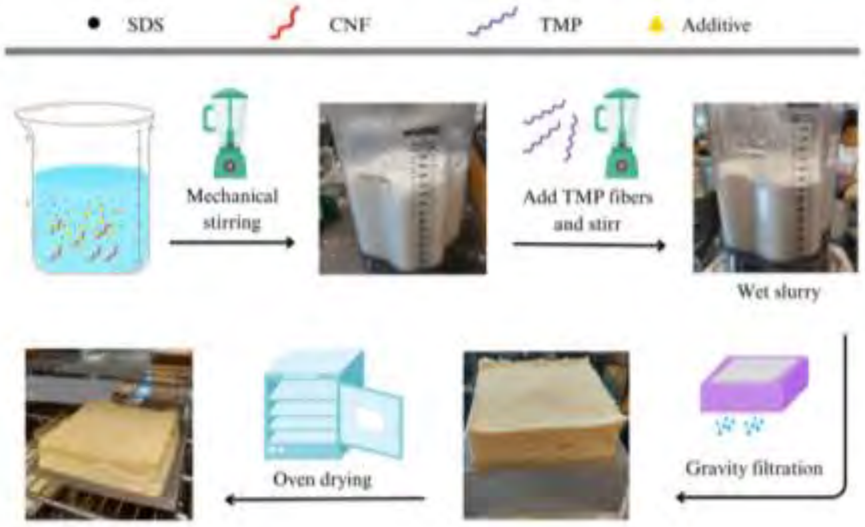
Cellulose nanofibril-reinforced surfactant-assisted lignocellulosic foams for packaging and building applications

Lead: Mara Paulette Alonso, Rakibul Hossain

Stability of the foams by drainage test

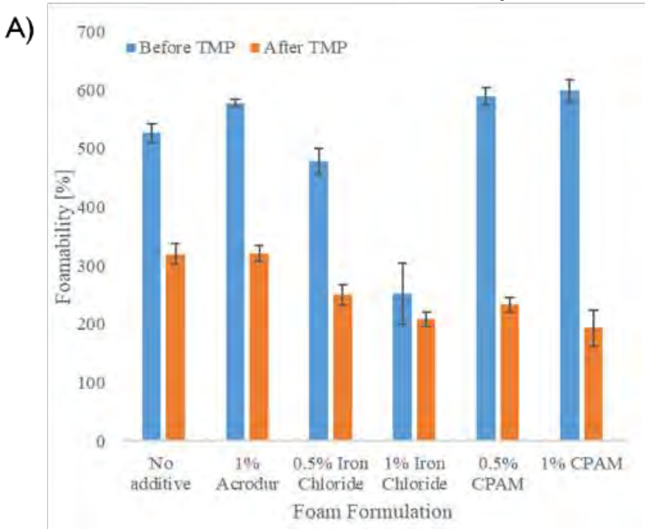


Foam making process



- CNF slows down drainage
- CNF stabilizes the foam
- TMP fibers destroy the bubbles, destabilize the foam
- Different additives affect the bubbles differently
- TMP fibers destroy the bubbles in the foam for both neat foams and with additives

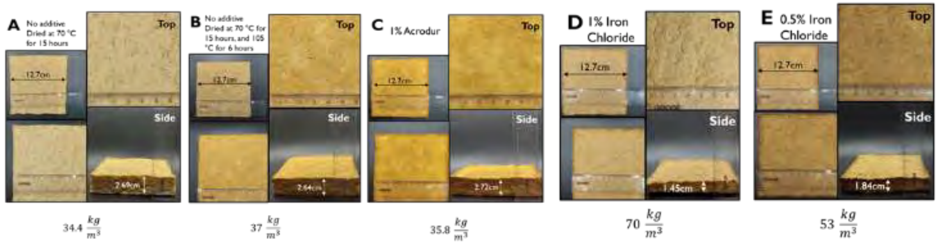
Foamability

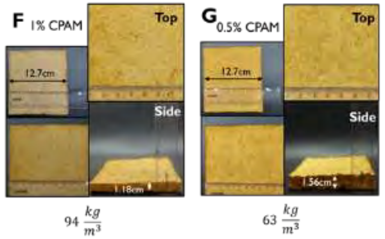


Foam formulation

TMP CONTENT (WT. %)	CNF CONTENT (WT. %)	ADDITIVE TYPES	ADDITIVE CONTENT* (PPH)
95	5	Neat/Non-Additive	0
		Acrodur	1
		Iron Chloride	0.5
		Iron Chloride	1
		CPAM	0.5
		CPAM	1

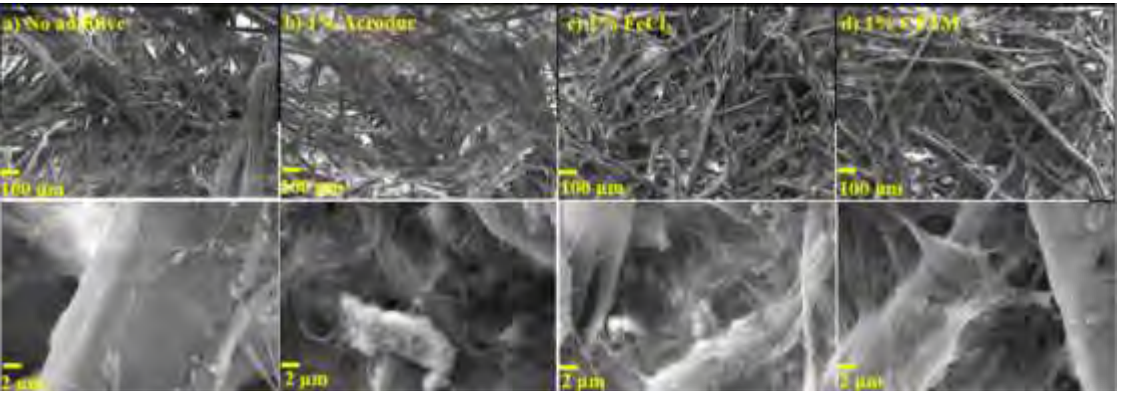
Thickness & density of the foams



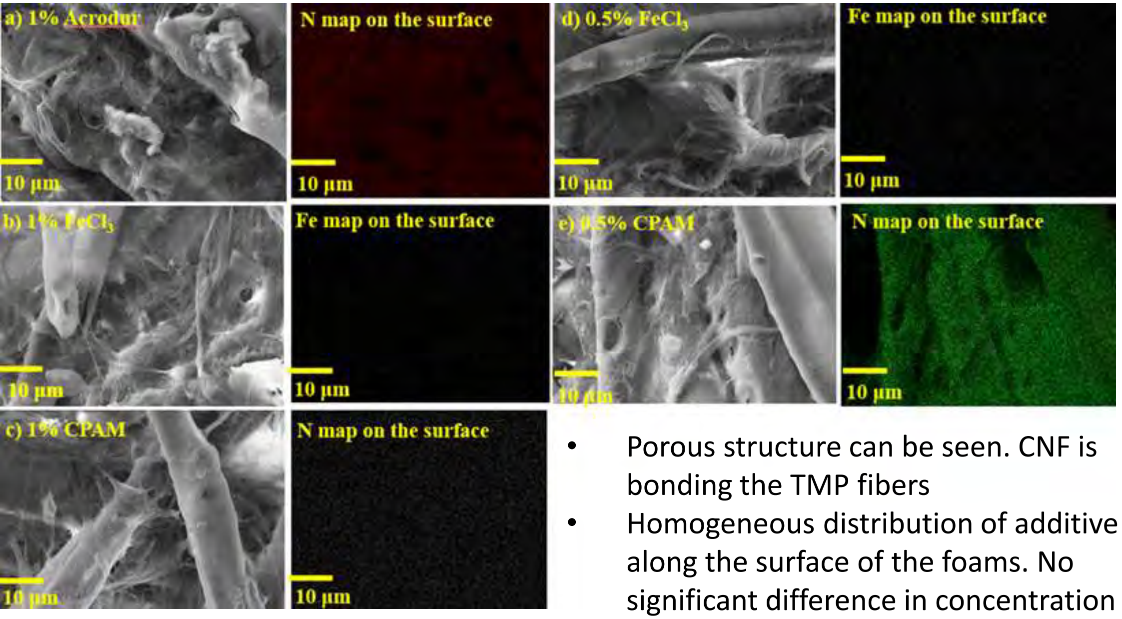


- Decrease in foam volume increases the density
- With the increase in density, the surface of the foams became more rough

SEM analysis of the foams

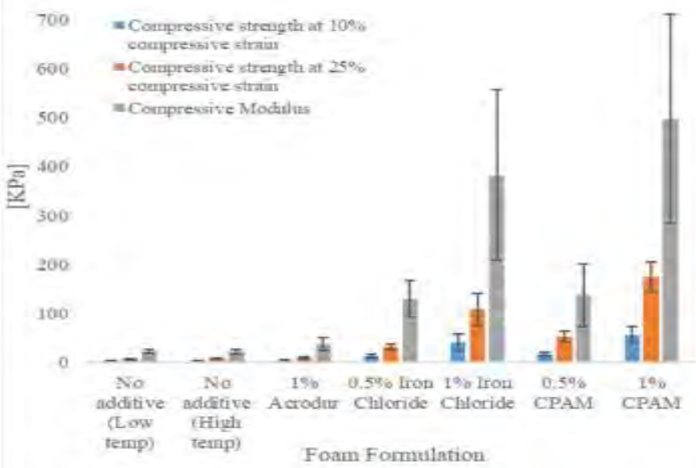


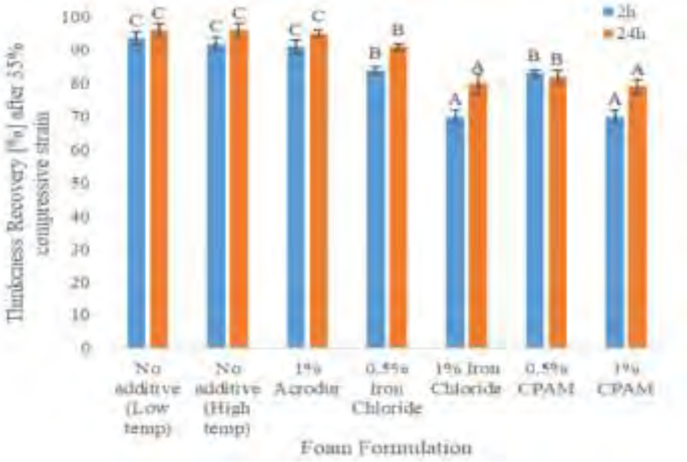
EDS analysis of the foams



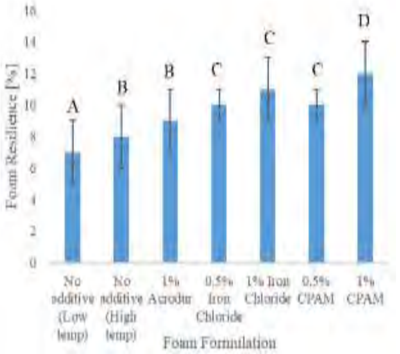
- Porous structure can be seen. CNF is bonding the TMP fibers
- Homogeneous distribution of additive along the surface of the foams. No significant difference in concentration on 0.5% additive compared to 1%

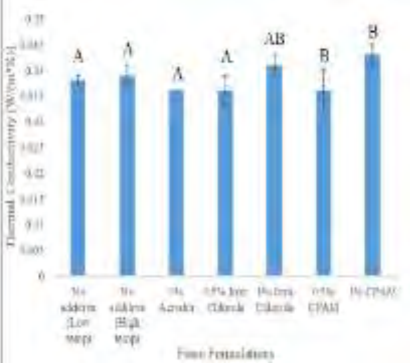
Compressive strength & thickness recovery

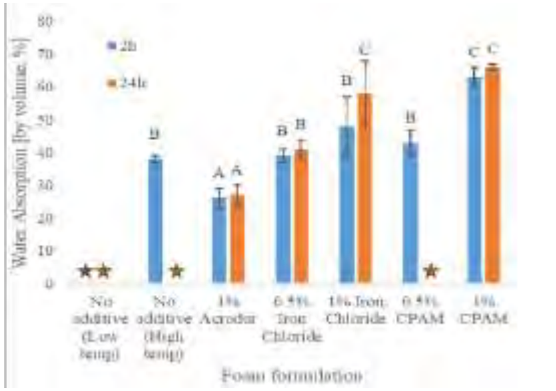




Foam resilience, thermal conductivity & water absorption properties







- Compressive strength & modulus increased to various degrees depending on the additive type and content.
- Foam resilience increased with the increase of density
- All foams exhibited excellent thermal resistivity
- Foams without additives disintegrated during the test, high temperature drying performed better
- Foams with additives maintained their structure except for 0.5% CPAM

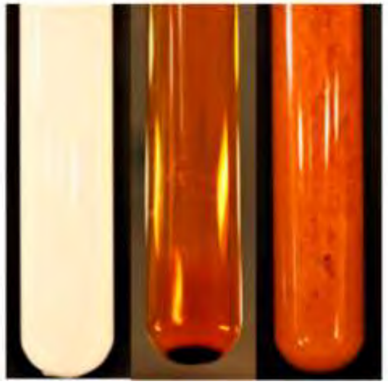
Acknowledgement:

TIMBERHP USDA OAK RIDGE National Laboratory

Water treatment applications

Hybrid Freeze-dried CNF-based Aerogel for Arsenic Removal from Water

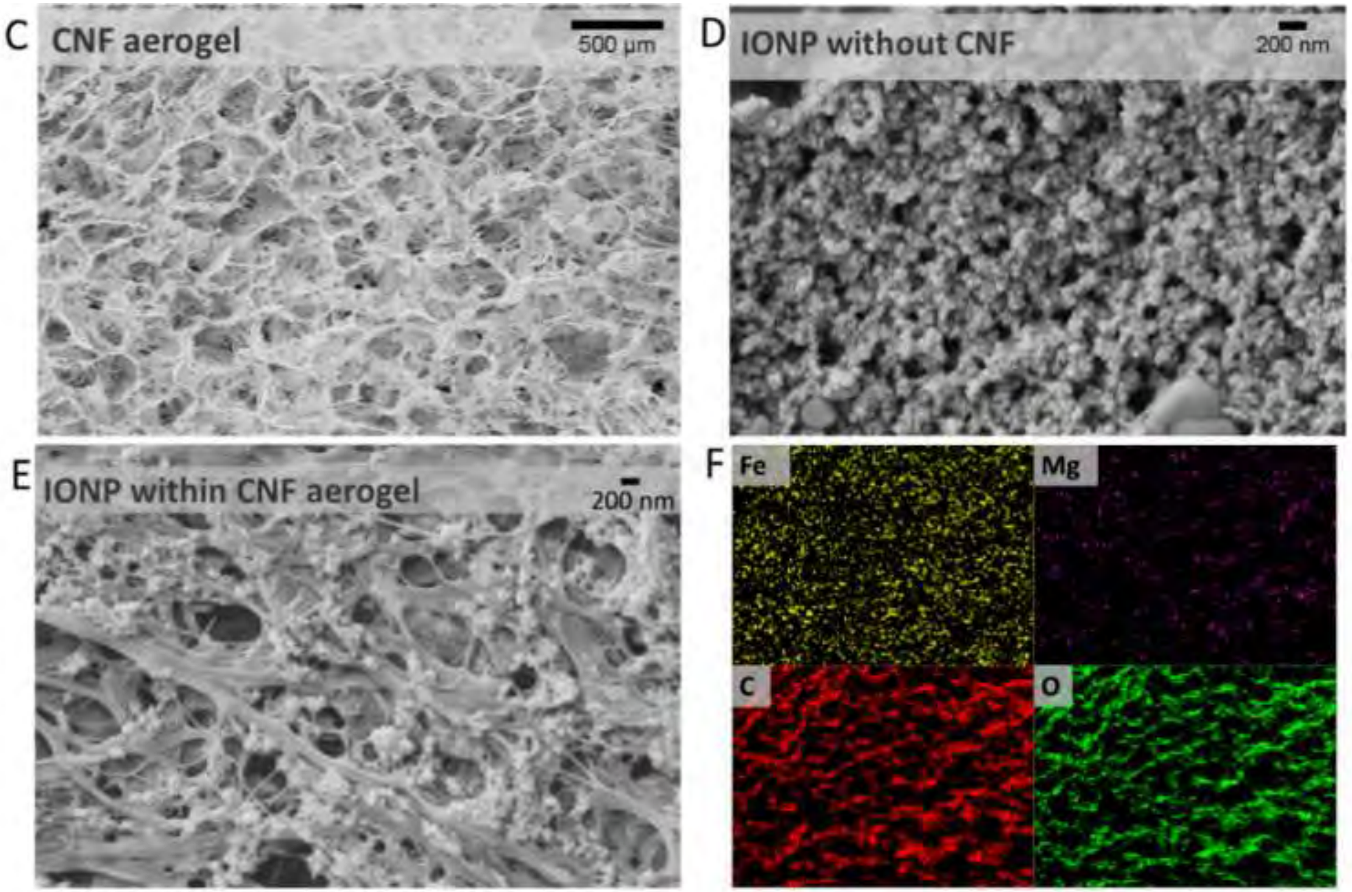
Lead: Md. Musfiqur Rahman

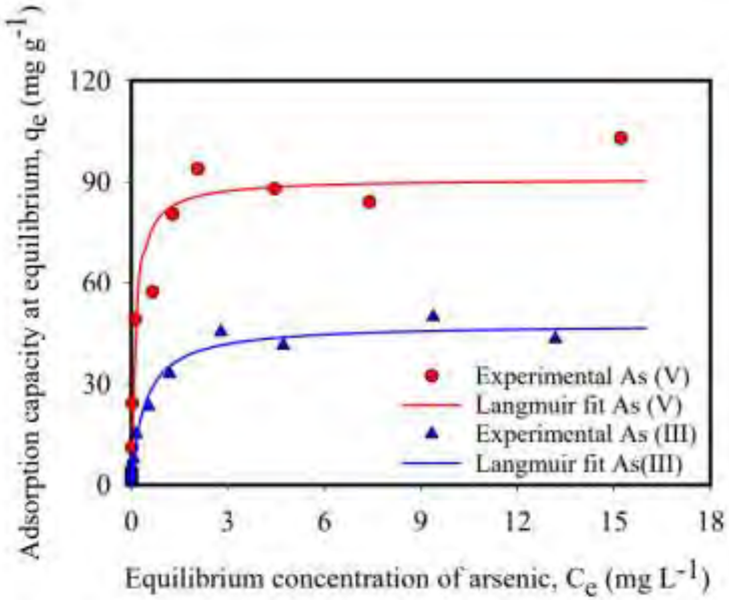


CNF-IONPs suspension

Freeze-drying

CNF-IONPs aerogel
(Density 13 kg/m^3)





Rahman, M.M.; Hafez, I.; Tajvidi, M.; Amirbahman, A. Highly Efficient Iron Oxide Nanoparticles Immobilized on Cellulose Nanofibril Aerogels for Arsenic Removal from Water. *Nanomaterials* **2021**, *11*, 2818.

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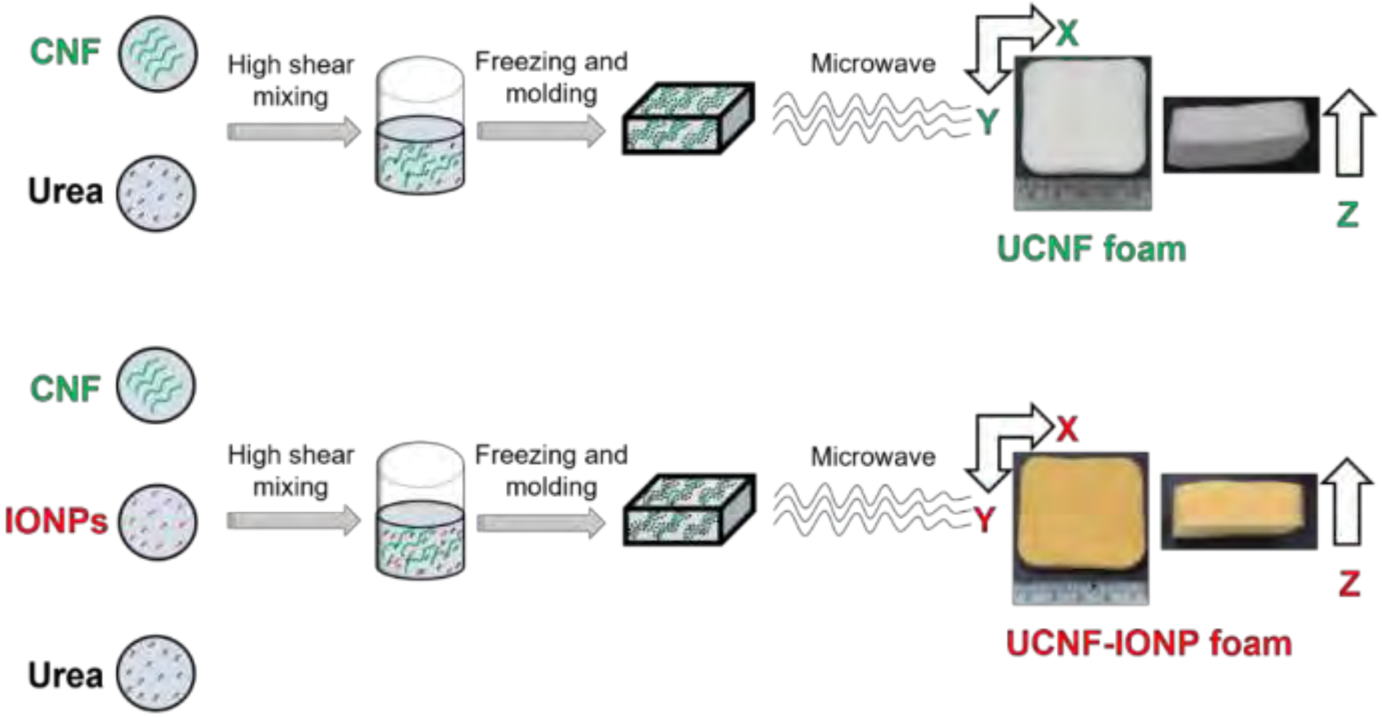
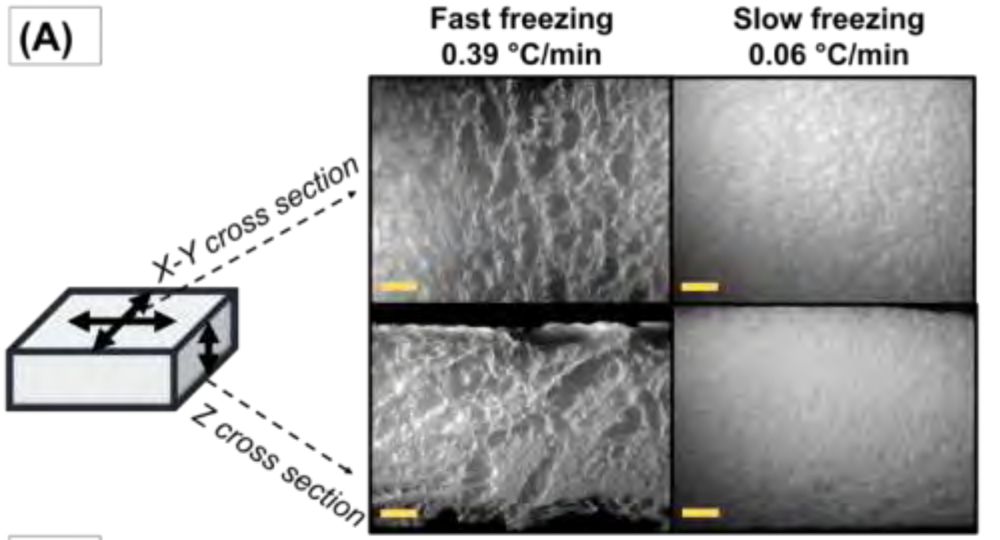
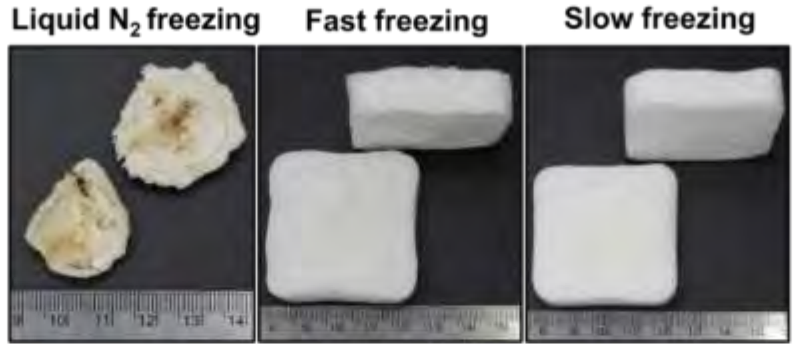
Alternative Approach to Produce Hybrid CNF-based Foams by Microwave Irradiation



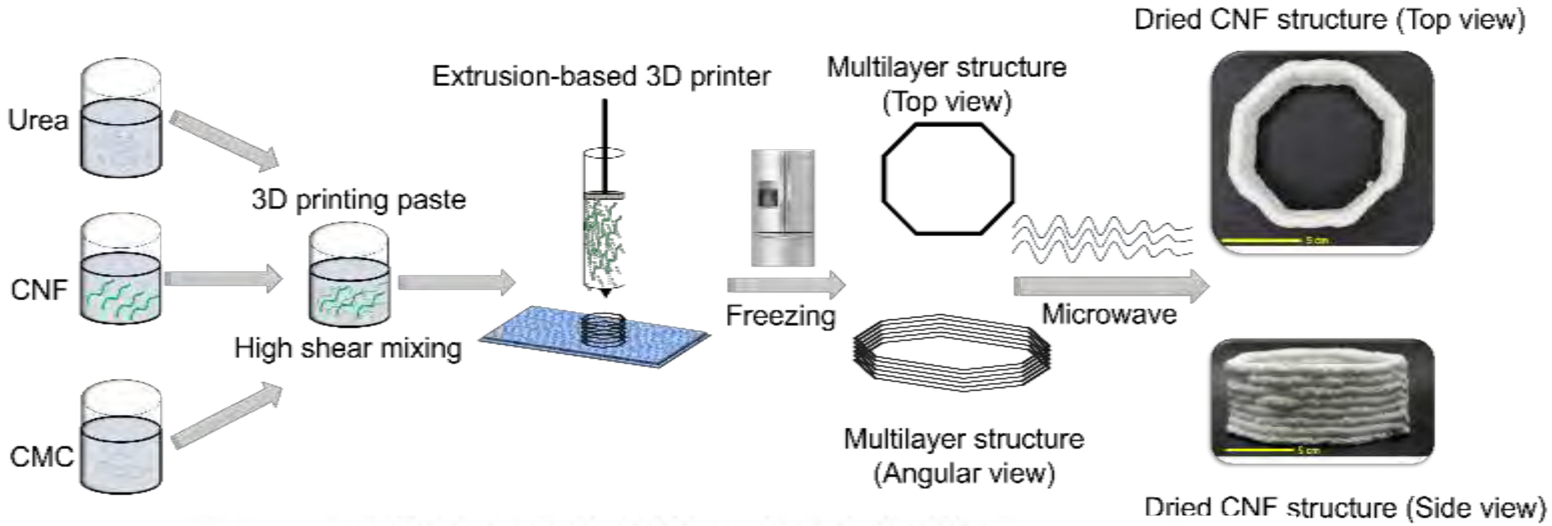




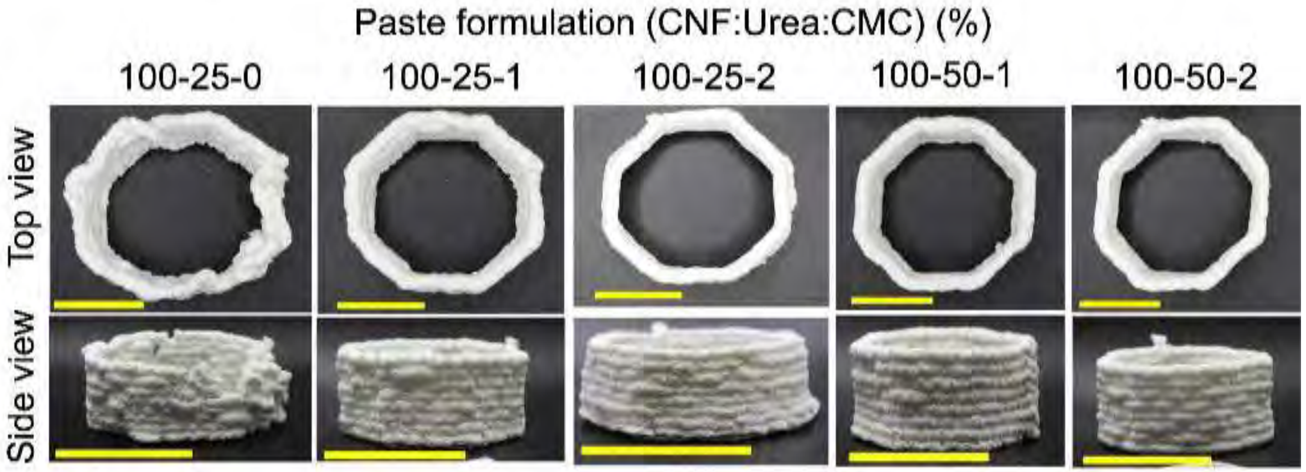
- Density of the foams: 35-37 kg/m^3; porosity 97%
- Freezing rate effects, the pore architecture: slow freezing generates uniform pore architecture
- To date- the fastest & cost-effective method of producing CNF-based foam.

Rahman, M.M.; Hafez, I.; Tajvidi, M.; Amirbahman, A. Fundamentals of Hybrid Cellulose Nanofibrils Foam Production by Microwave-assisted Thawing/Drying Mechanism. *ACS Sustain. Chem. Eng.* (Just accepted)

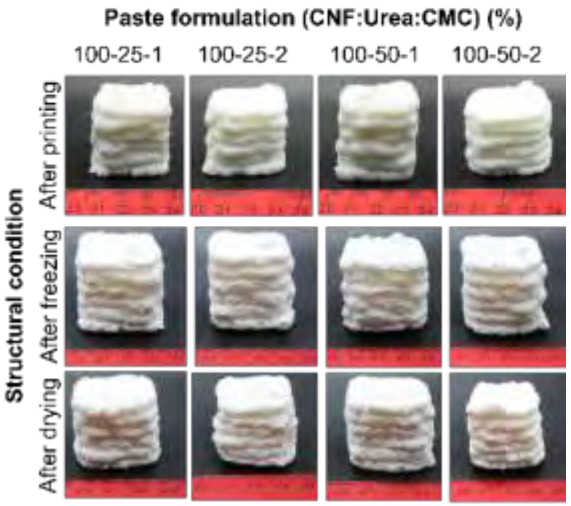
A Novel Cost-effective Approach for 3D Printing with Cellulose Nanofibrils (CNFs)

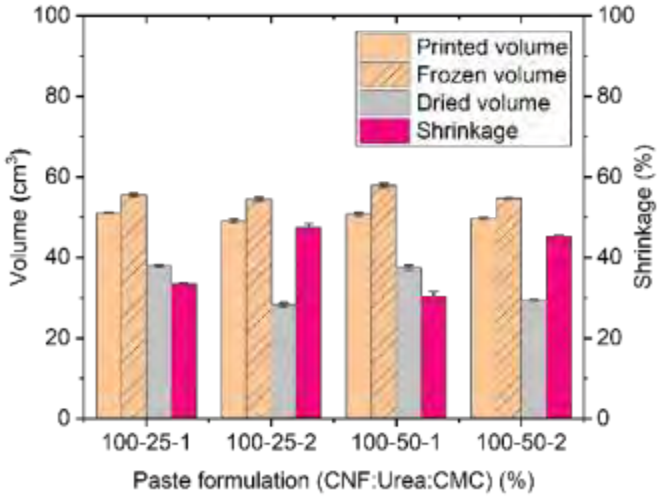


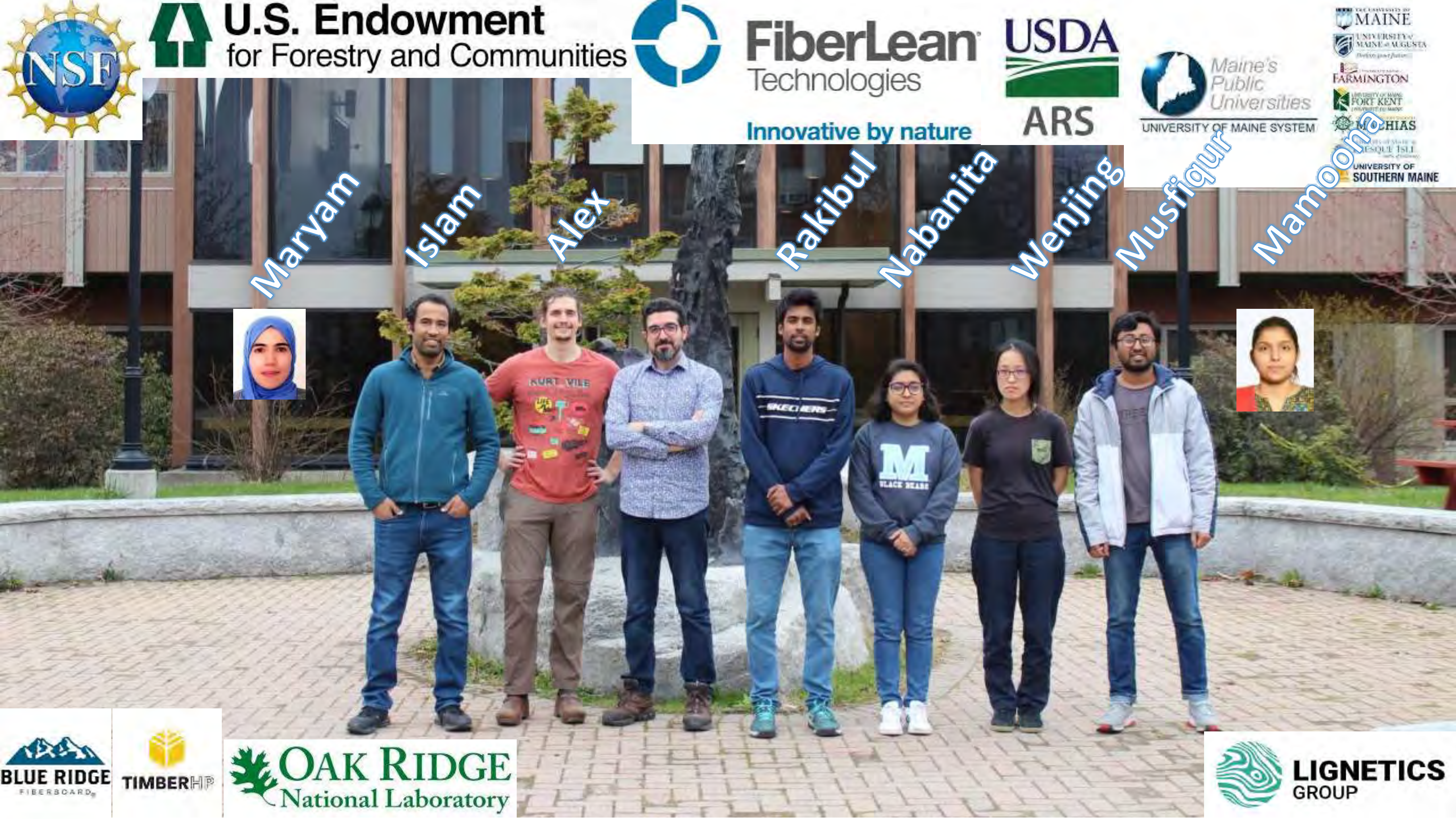
- Assessment of structures in different conditions
- Rheology, compression & tensile testing
- Effects of different components (CNF/Urea/CMC) on internal pore morphology.



*Scale 5 cm



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Maine's Public Universities
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