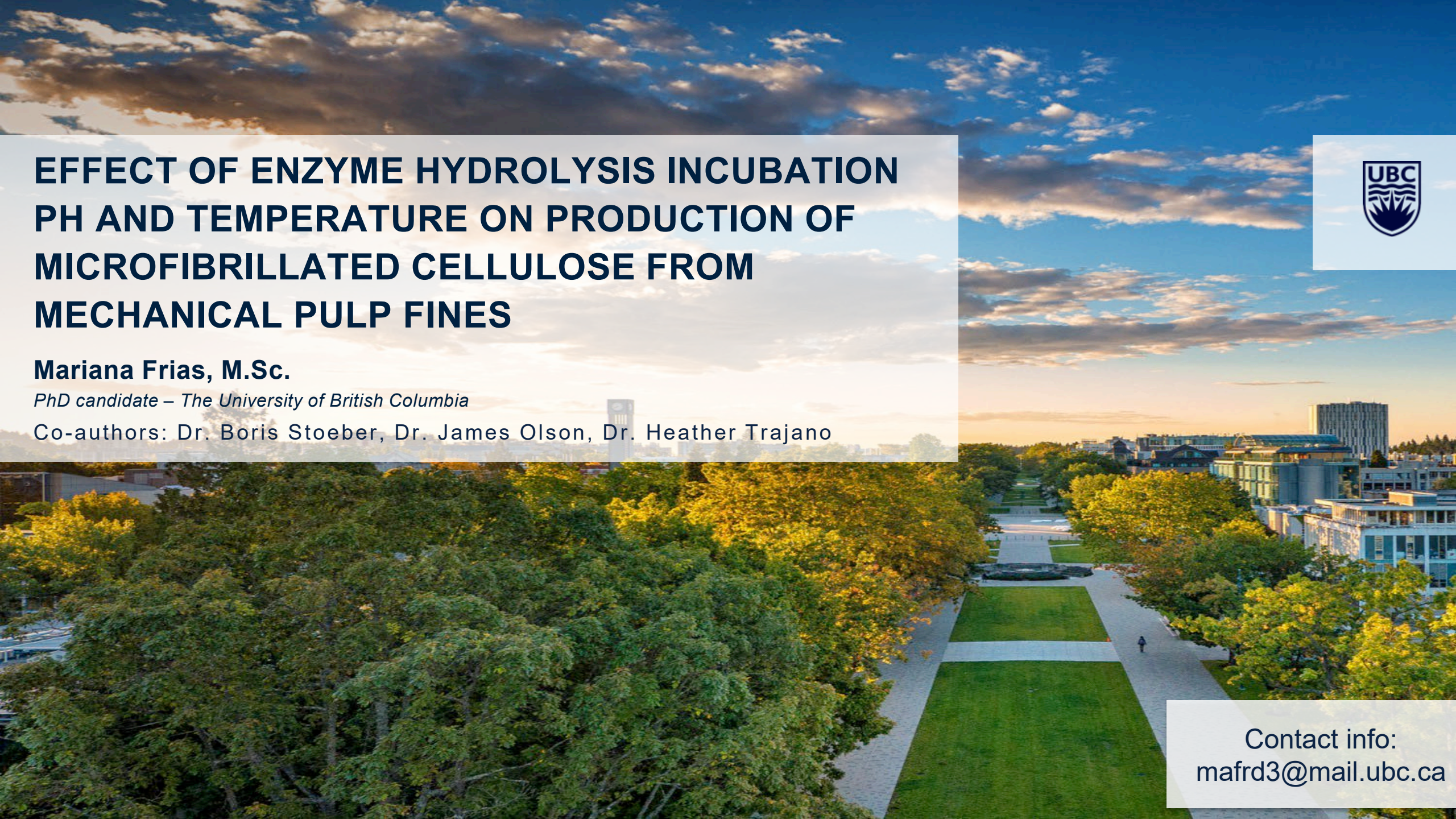

EFFECT OF ENZYME HYDROLYSIS INCUBATION PH AND TEMPERATURE ON PRODUCTION OF MICROFIBRILLATED CELLULOSE FROM MECHANICAL PULP FINES

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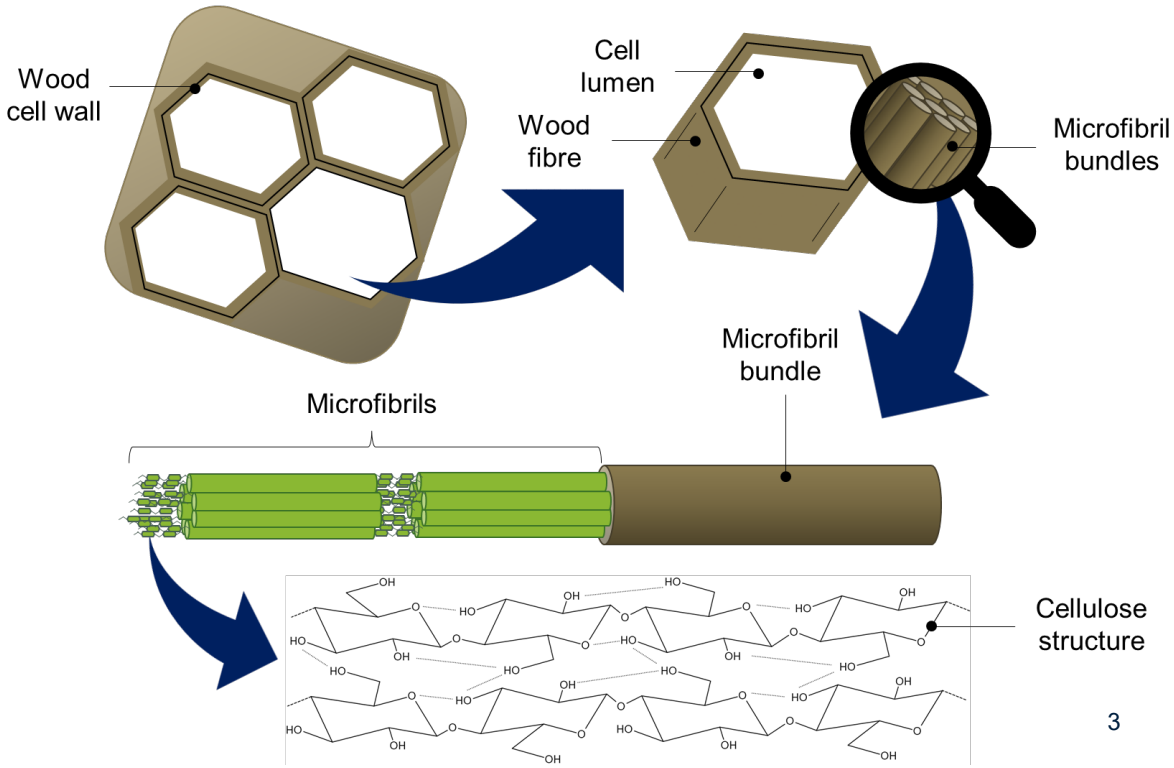
OUTLINE

- Background
- Research objectives
- Methodology
- Results
- Conclusion & Future work

MECHANICAL PULP

- Wood chips → Individual fibres
  - Energy-intensive process
- Fibre chemistry very similar to wood
- Cost-effective fibre source
- **Product diversification**: specialty papers, packaging materials, and textiles
- High-value applications: **lignin-rich MFC**

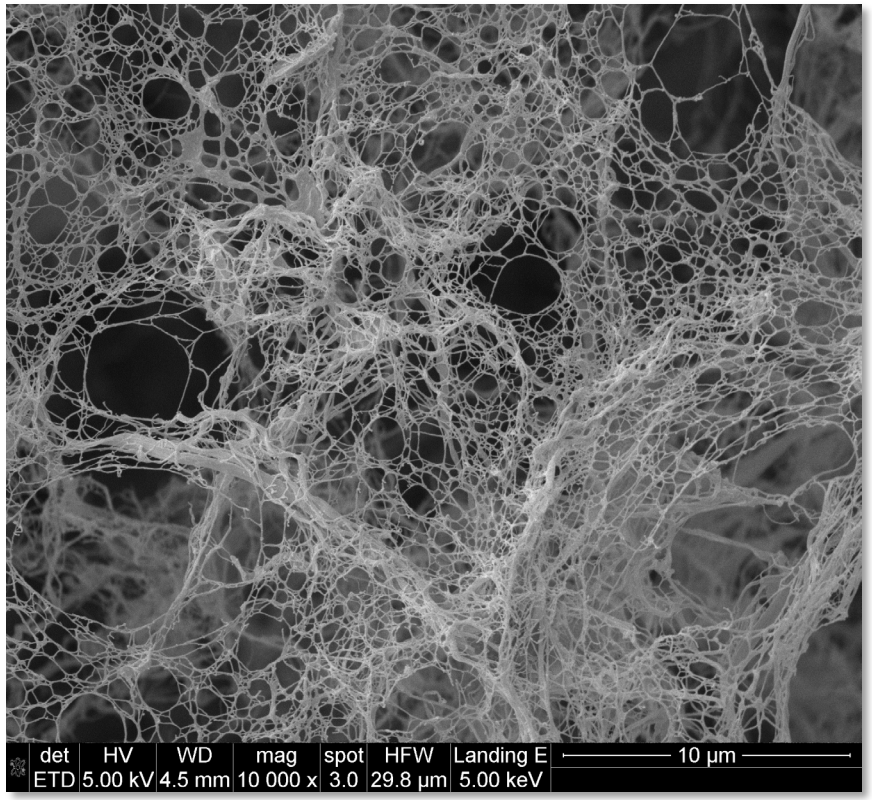
https://www.britannica.com/topic/newspaper



MFC & FINES

- MFC is obtained from fibrillation of the cell wall
  - Usually from *kraft pulp*
  - ~30,000 kWh/ton to produce

- High aspect ratio, stiffness, surface area
- From mechanical pulp short fibres[2]
  - Concentrated fines fraction
- Fines usually partially discarded
- Similar character to coarser MFC

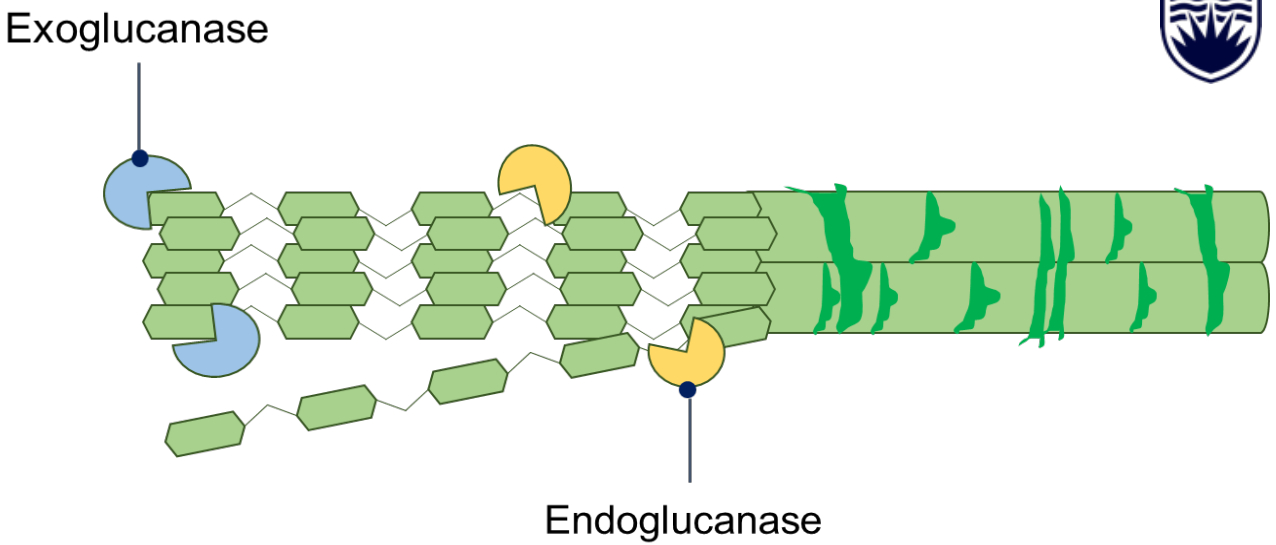


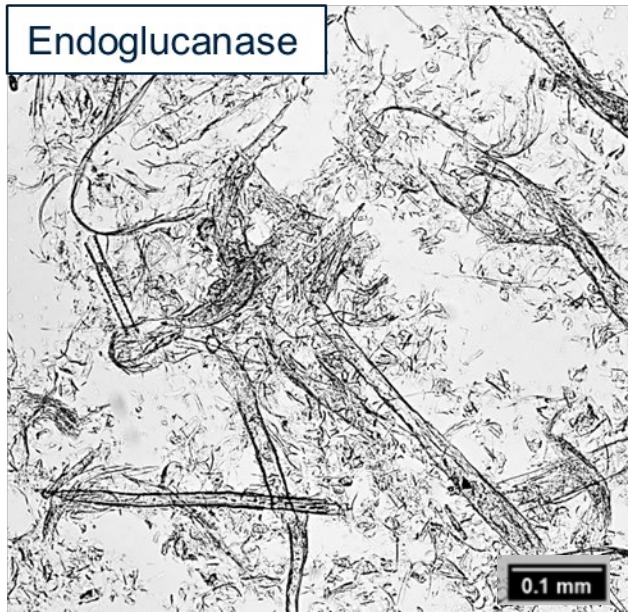
Tingaut et al., 2011[1]

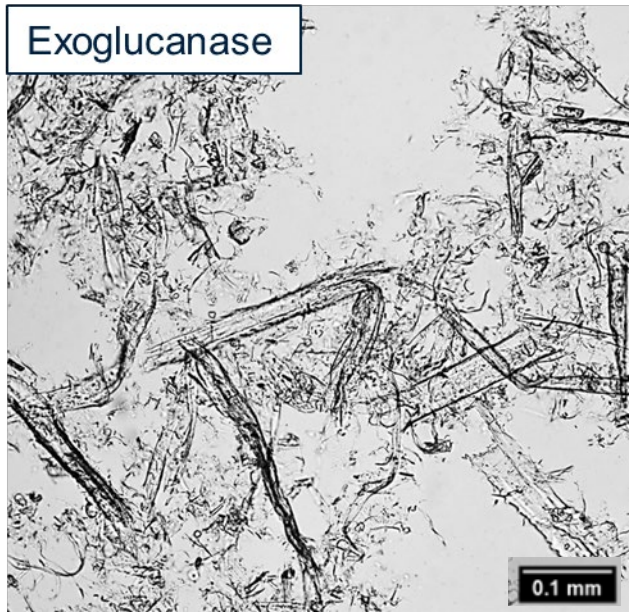
[1]Tingaut, P., Eyholzer, C., & Zimmermann, T. (2011). *Functional polymer nanocomposite materials from microfibrillated cellulose* (pp. 319-334). Rijeka: InTech.
[2]Seifert, R., Gharehkhani, S., Vargas Figueroa, D., Mercuur, J., & Olson, J. (2023). Engineering the paper production by combined fiber fractionation and reinforcement with microfibrillated cellulose. *Cellulose*, *30*(5), 3201-3217.

ENZYME HYDROLYSIS

- Facilitated fibrillation and reduced energy
  - Length reduction
  - Open structure to refining
- **Endoglucanase** hydrolyses internal bonds in cellulose chains
- **Exoglucanase** hydrolysis ends of cellulose chains
- Challenges:
  - Chemical inhibition
  - Reaction settings
  - Complex substrate

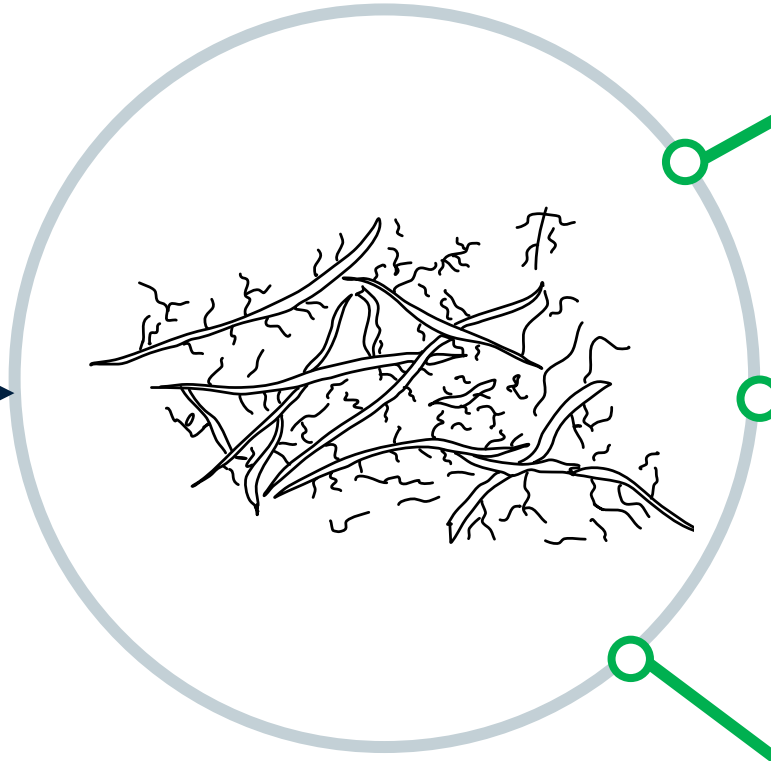






RESEARCH OBJECTIVE

Valorization of mechanical pulp fines



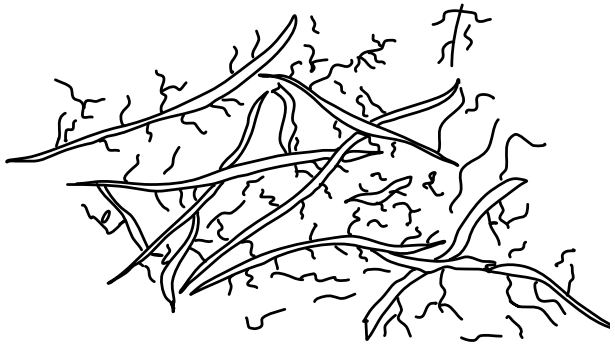
Production of MFC

- Coating
- Reinforcement
- Mill trial

RESEARCH OBJECTIVE

Valorization of mechanical pulp fines

reduced energy



Production of MFC

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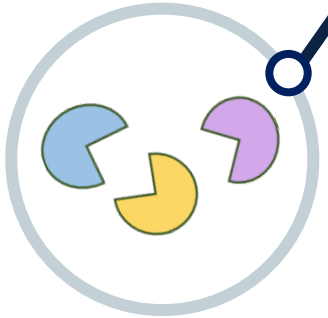
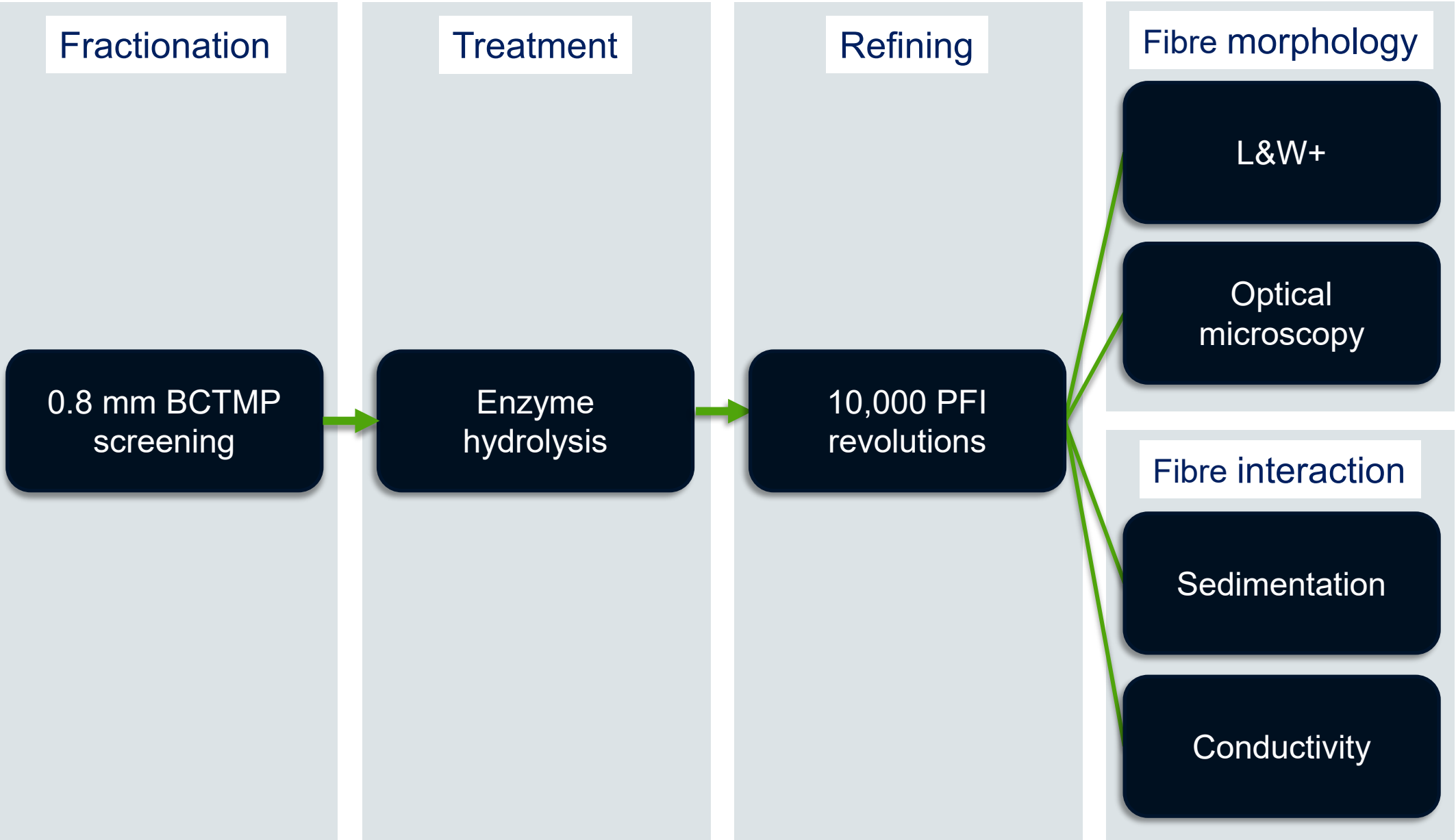


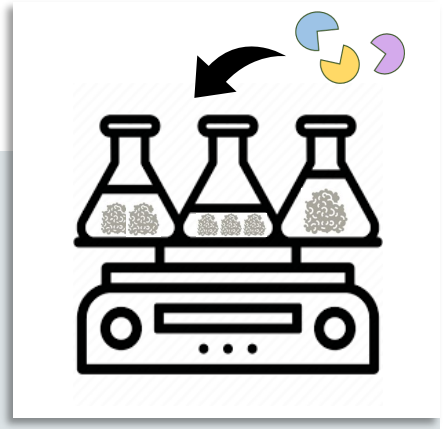
Enzyme hydrolysis

- Dosage
- Type
- **Incubation conditions**

METHODOLOGY



METHODOLOGY



Enzyme hydrolysis

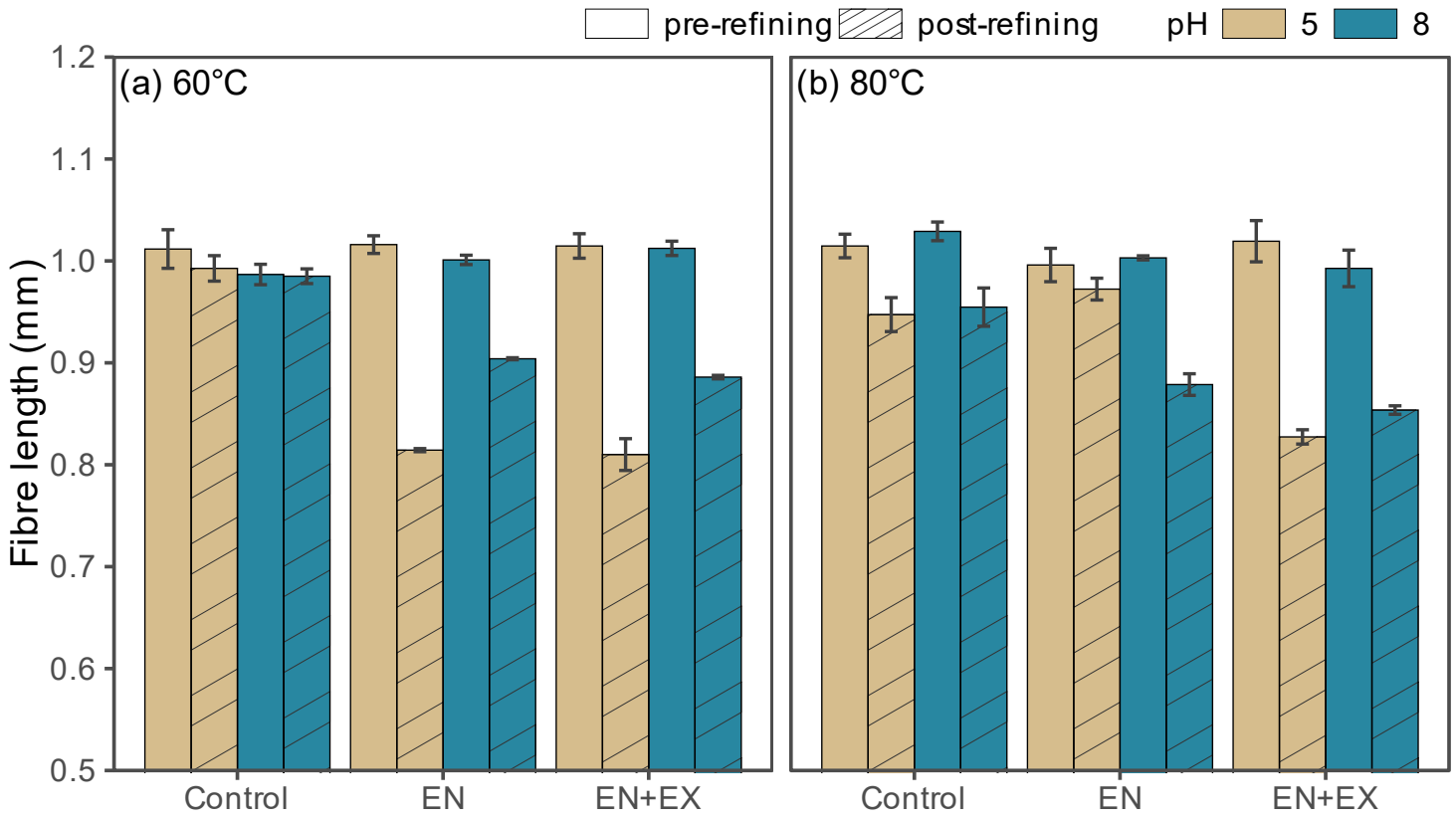
Pulp consistency 3.5%	**Incubation time** 2h	**Enzyme dosage** 1.0 wt%
Solution pH 5, 8	**Incubation temperature** 60°C, 80°C	

Enzyme type		
Refining only (Control)	Endoglucanase (EN)	Endoglucanase + Exoglucanase* (EN+EX)

Auxiliary enzyme added at 10 wt% of Endoglucanase dosage.

FIBRE MORPHOLOGY

- No fibre length Δ pre-refining
- Maximum kinetics: pH 5 and 60°C
- Incubation at 80°C softens fibre structure
- pH 8 and 80°C still promote fibre cutting
- EN+EX treated fibres show constant length



FIBRE MORPHOLOGY

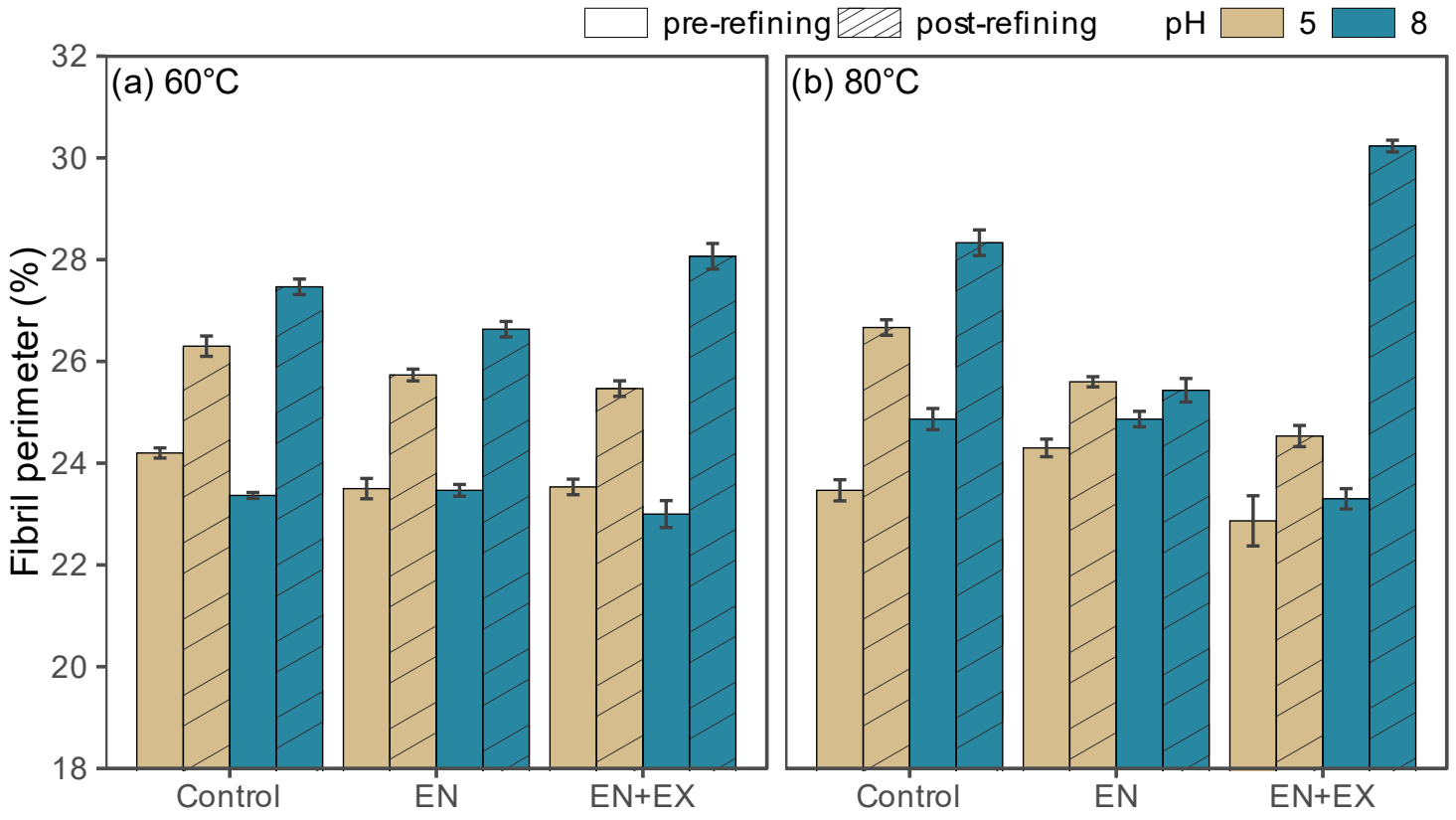
FIBRIL PERIMETER

- Obtained from L&W+
- Macro fibrillation measurement
- Optical imaging method

$$\text{Fibril perimeter} = \frac{\text{Perimeter of fibrils}}{\text{Perimeter of fibre} + \text{fibrils}}$$

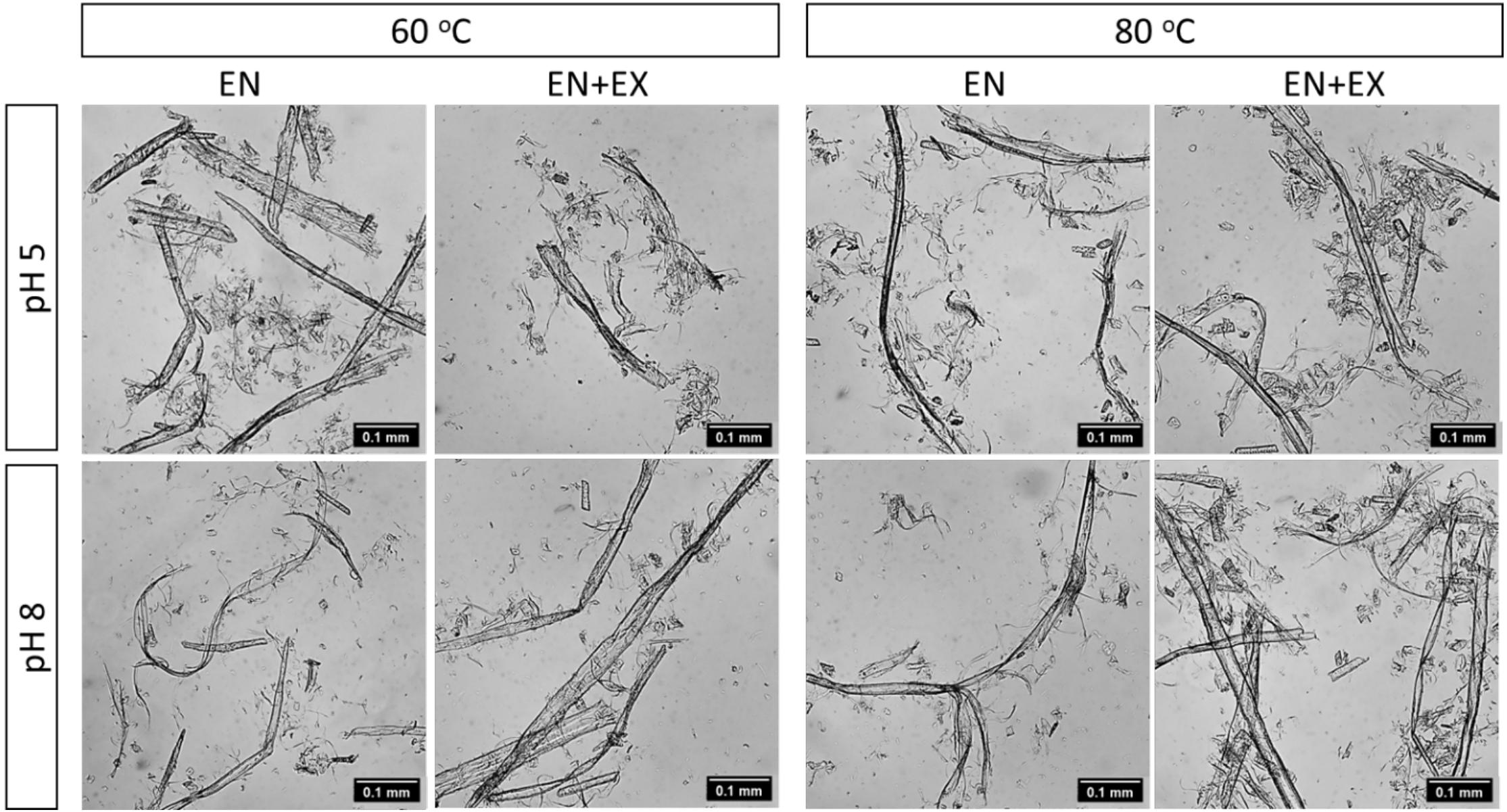
FIBRE MORPHOLOGY

- ↑ fibril perimeter with refining: EN hinders fibrillation
- Fibrils are more accessible
  - Higher surface area
- EN+EX at pH 8 and 80°C: ↑ fibrillation
- Interaction: kinetics, pulp and incubation conditions

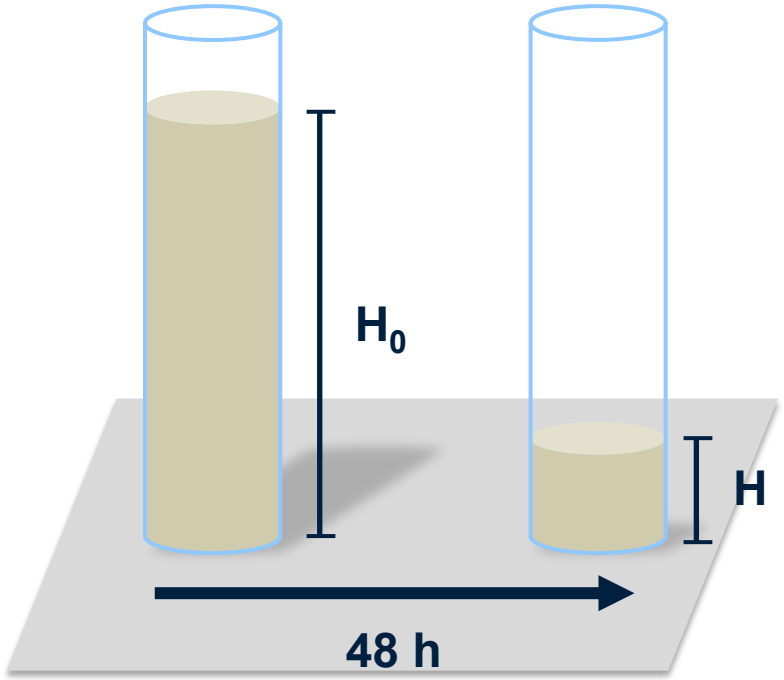


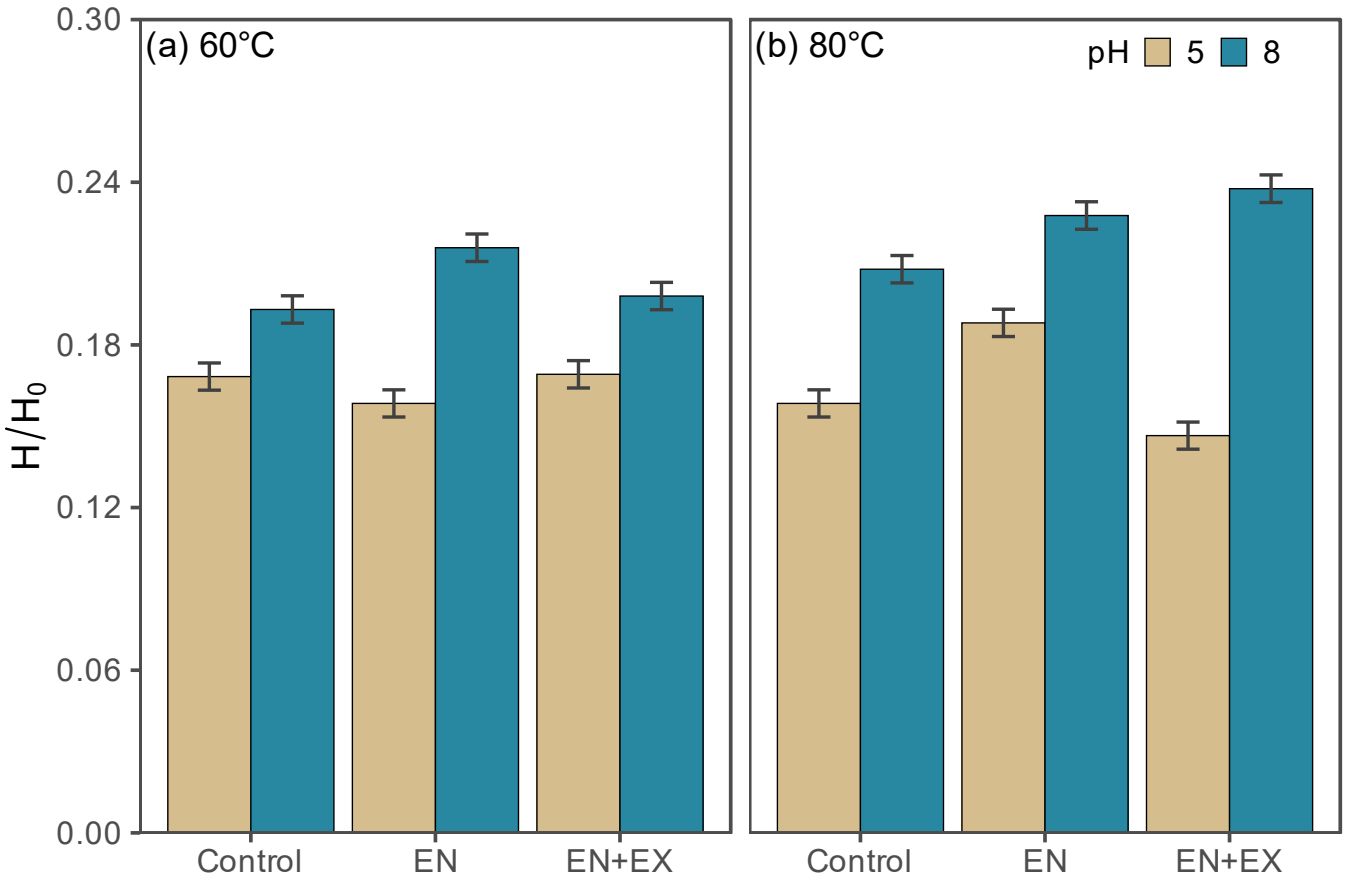
OPTICAL MICROSCOPY

- Enzyme digestion pattern at pH 5 and 60°C
- Fibrils and cut fibres at pH 8 and 80°C



SEDIMENTATION

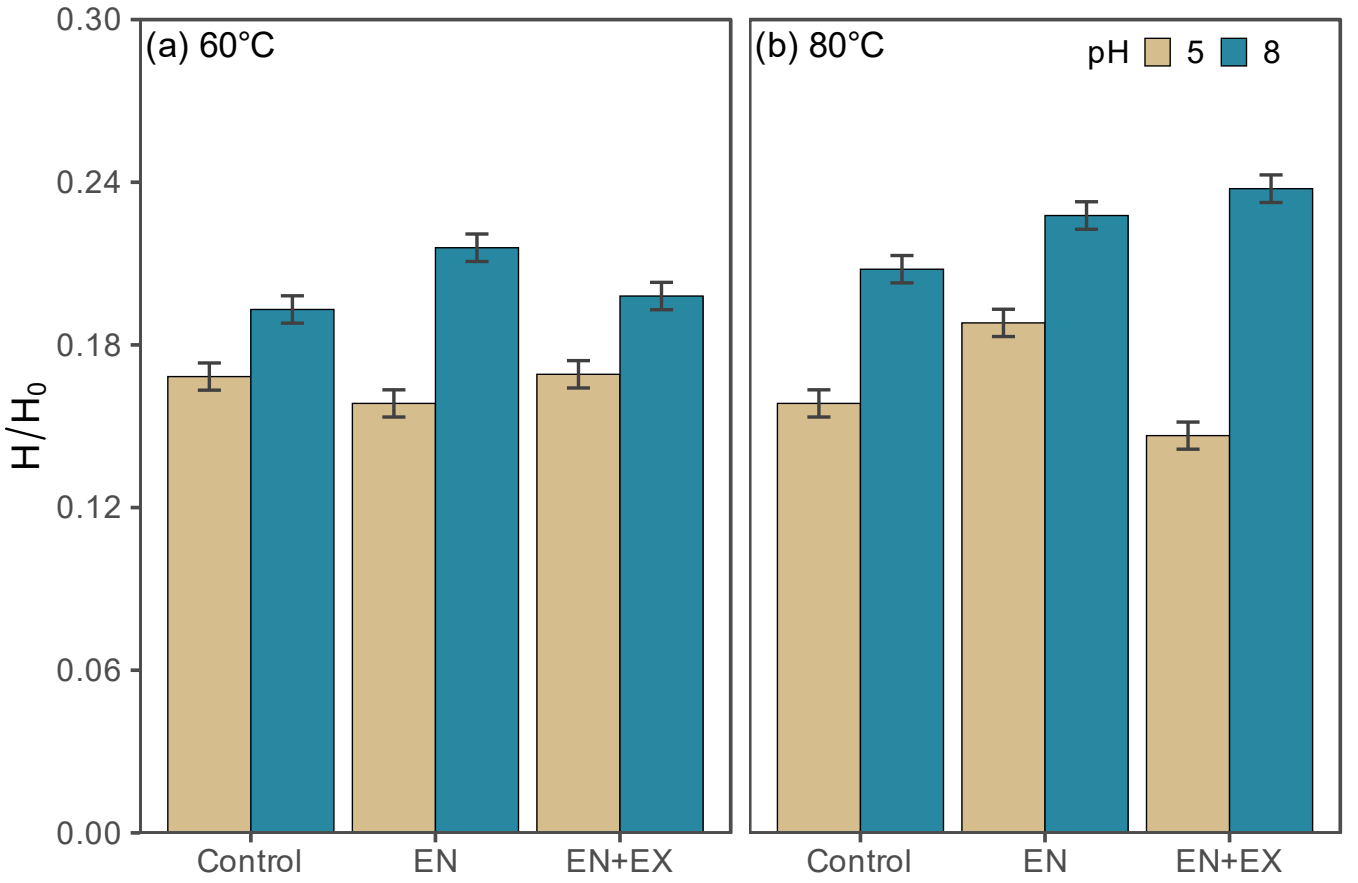




- Incubation at pH 8 ↑ settling height
- Higher settling height seen at pH 8, 80°C hydrolyzed with EN+EX
- Process governed by crowding factor
- Similar morphology, same concentration
- Surface charge modification?

SEDIMENTATION

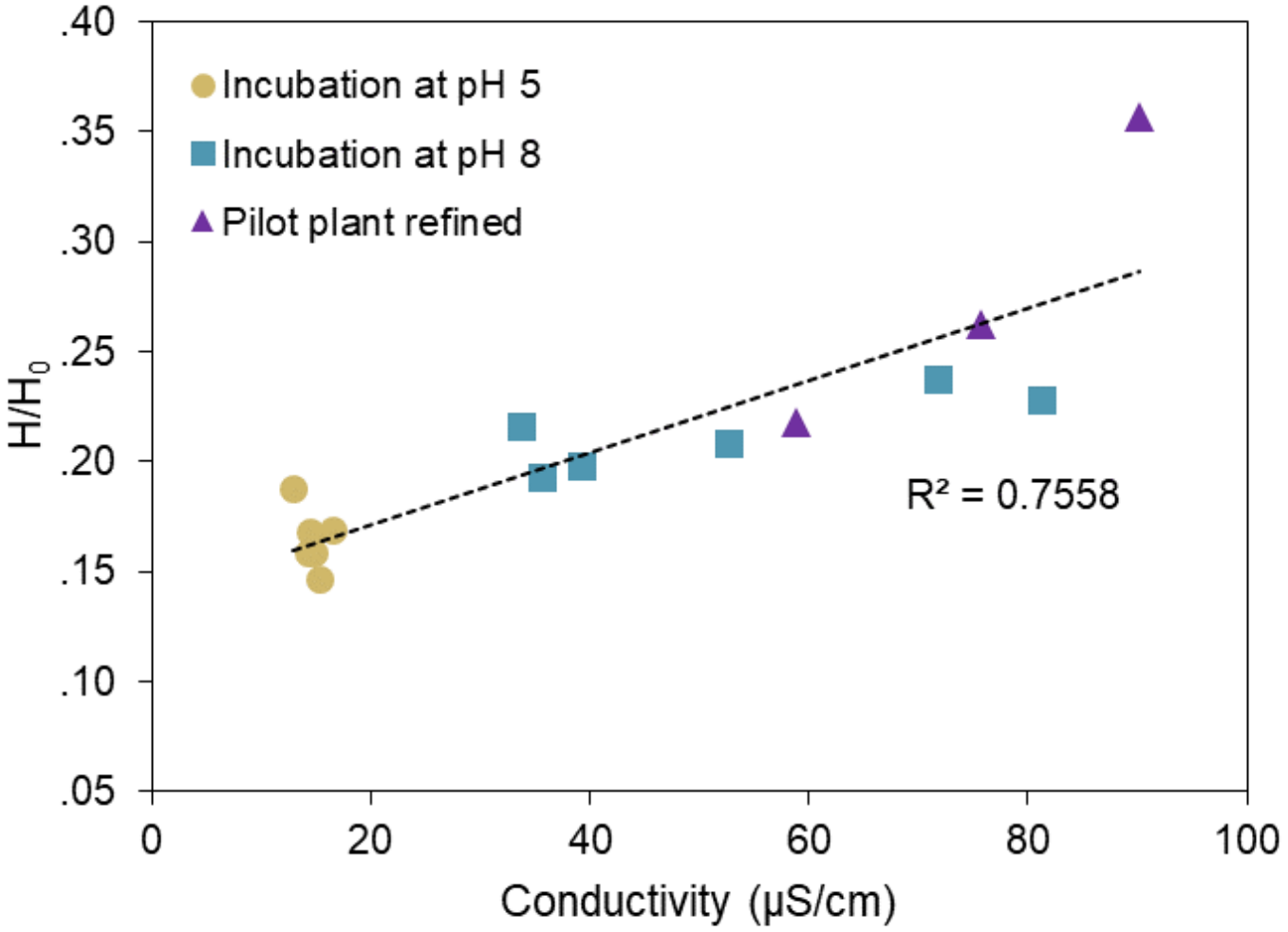
Sample	pH	Temperature (°C)	Coarseness (mg/100m)
Control	5	60 80	6.00 ± 0.02 5.46 ± 0.02
	8	60 80	5.39 ± 0.04 5.30 ± 0.01
EN	5	60 80	5.02 ± 0.03 5.50 ± 0.02
	8	60 80	5.17 ± 0.04 6.17 ± 0.03
EN+EX	5	60 80	4.73 ± 0.01 5.60 ± 0.02
	8	60 80	4.43 ± 0.01 4.21 ± 0.03



- Coarseness ↓ with temperature and pH ↑
- Enzymes mostly help ↓ coarseness
- EN+EX treated fibres showed ↓ ↓ ↓ coarseness

CONDUCTIVITY

- Incubation at pH 5 showed lower conductivity
  - Fibres collapsed
  - Surface charge neutralized
- Incubation at pH 8 ↑ repulsion
- Direct correlation with settling height
- Including short fibres refined at the pilot plant does not undo the correlation



CONCLUSION

- Incubation at pH 8 and 80°C increases fibrillation
- Adding EN+EX at 1 wt.% further helps the process
- Incubation at pH 8 shows higher settling height
- Conductivity could be linearly correlated to settling height

NEXT STEPS

- Investigation of cellulase dosage
- Determine the enzyme activity on a standard substrate
- Evaluate handsheet properties and correlate to H/H_0
- Investigation of hemicellulase inclusion

ACKNOWLEDGEMENTS

TMP mills:

Alberta Newsprint Company

CANFOR

CATALYST A PAPER EXCELLENCE COMPANY

HOLMEN PAPER

MILLAR WESTERN

MEADOW LAKE MECHANICAL PULP INC. A PAPER EXCELLENCE COMPANY

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NSERC CRSNG

Universities:

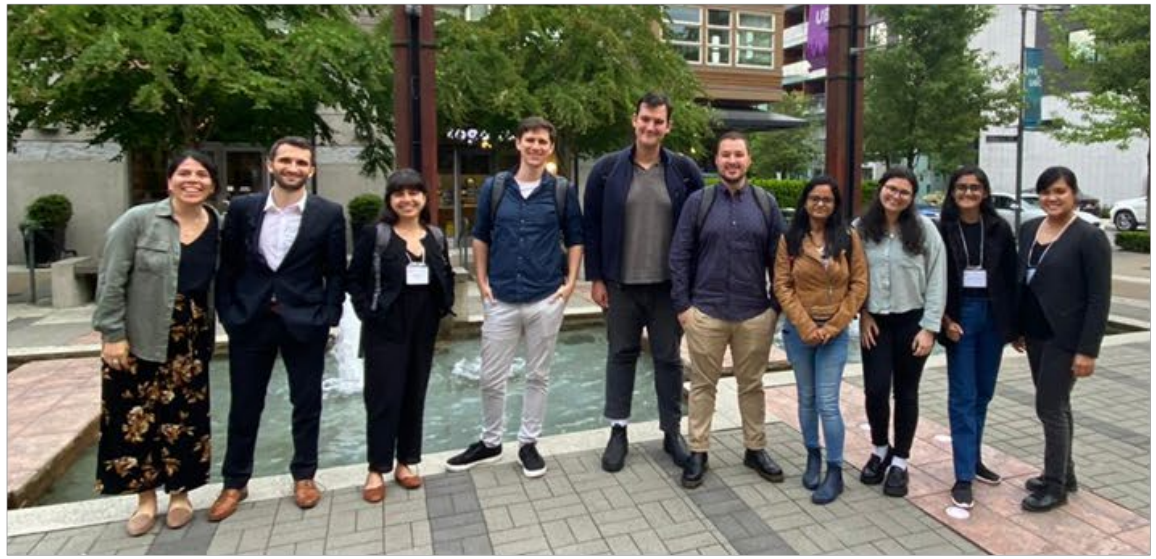
UBC Pulp and Paper Centre

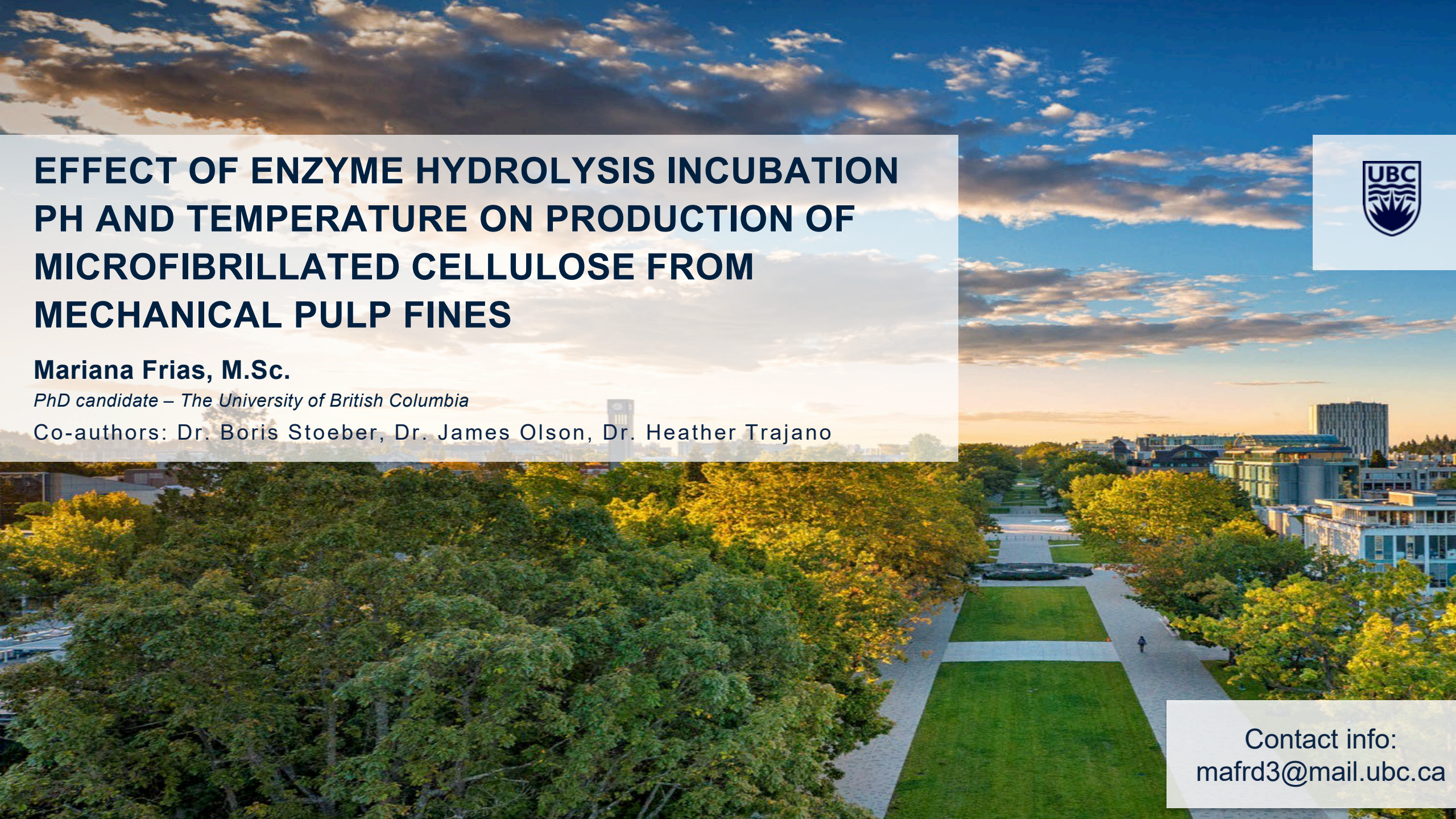
University of Victoria

BCIT

McMaster University

ERMP team & Stoeber Lab





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