International Conference on Nanotechnology for Renewable Materials

Iron Oxide Nanoparticles for Enhanced Visualization of Pulp Fibres by X-Ray Micro-Computed Tomography

Anderson T. V. Veiga, Elisa S. Ferreira,
James Drummond, Samuel Brown, Lewis Mason,
Andre Phillion, D. Mark Martinez, Emily D. Cranston

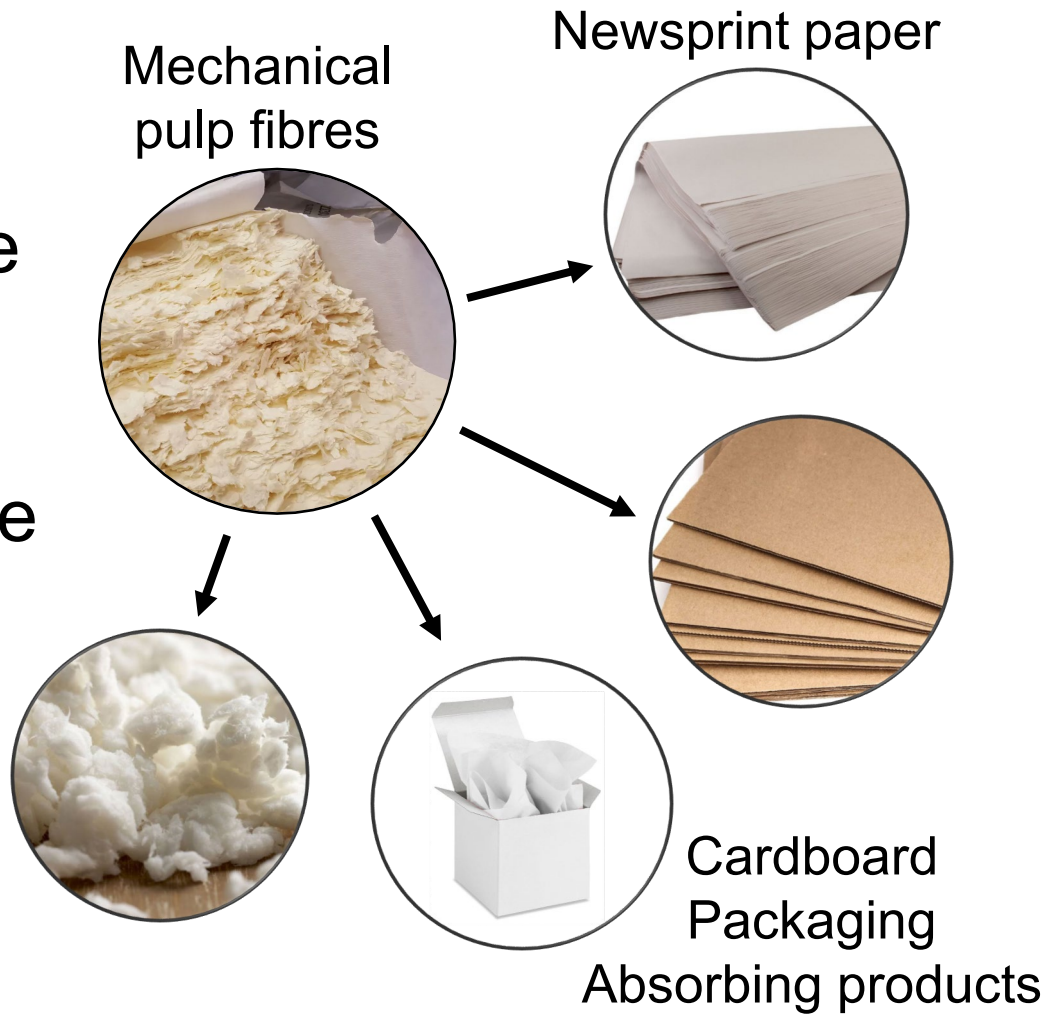
12-16 JUNE 2023 • VANCOUVER, B.C. CANADA

Nanotechnology to Advance Mechanical Pulp Applications

- Adaptability to changing product demand and supply (labour and materials)
- Understanding of products affected by the pulping process
- Explore new applications → high value-added products and advanced sustainable materials

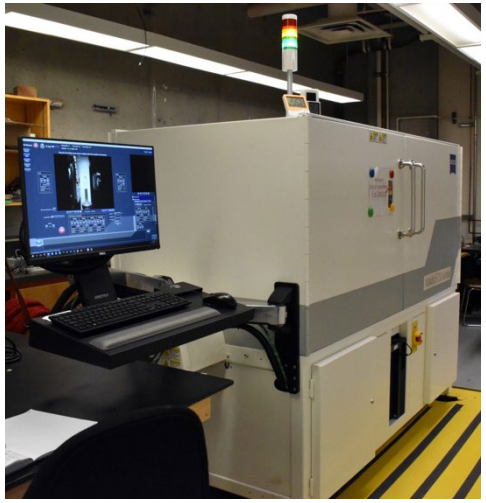
Advanced characterization method

✓ X-ray micro-computed tomography (MicroCT)



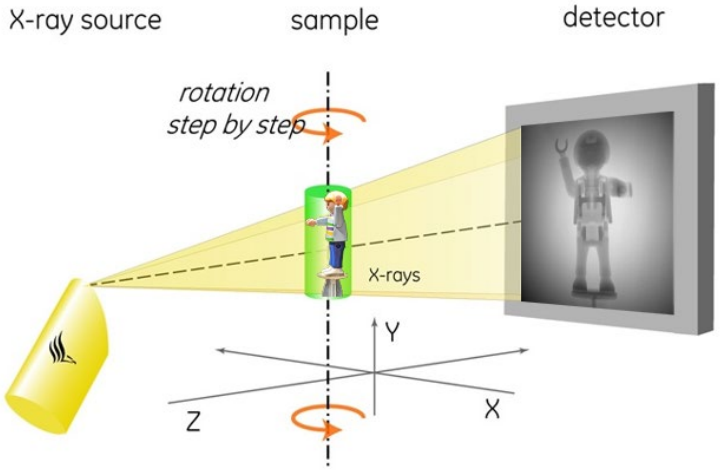
MicroCT

- X-ray imaging in 3D (same as clinical CT scans)
- Smallest pixel size = 0.3 µm

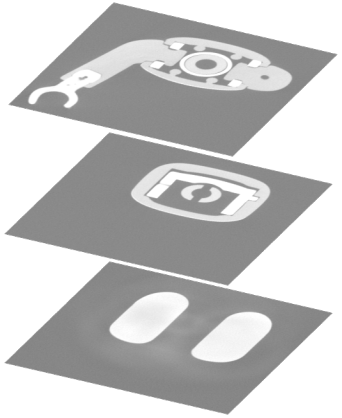


ZEISS Xradia 520 Versa
X-ray microCT
(Pulp and Paper Centre, UBC)

Image acquisition

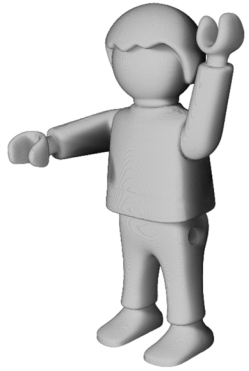


Reconstruction

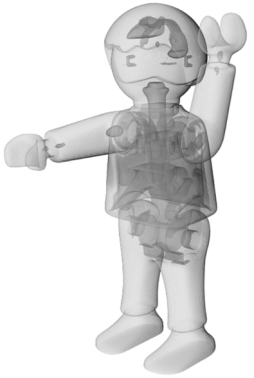


Cross-section
“slices”

3D volume rendering

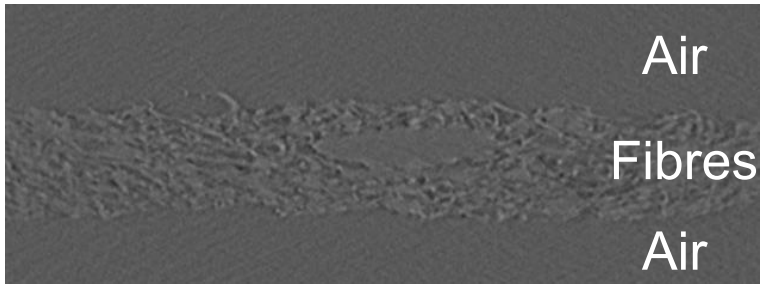


Interior details



Pulp Fibres Visualized by Micro-CT

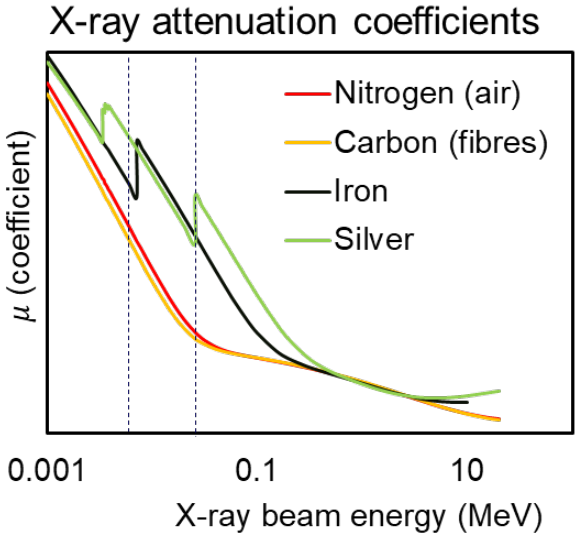
Cross section of a paper sheet



Pulp fibres by microCT

× Low-contrast image

× No fibre segmentation



$$I = I_0 e^{-\mu d}$$

I = X-ray intensity reaching the detector

I_0 = primary X-ray intensity

μ = attenuation coefficient

d = thickness

Iron oxide nanoparticles for enhanced contrast!

Why Enhance Contrast?

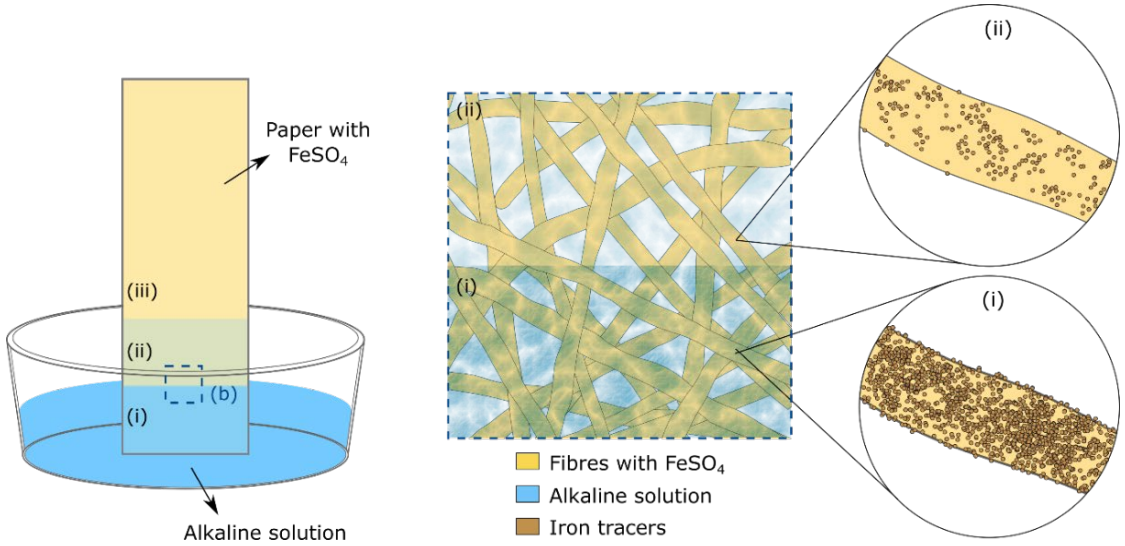
- Easier and more effective image processing
- Full fibre segmentation
- Visualize fibre morphology and fibre-fibre contacts
- With and without chemical treatment/modification
- Track the labelled fibres in 3D over time in microCT

- ❑Absorption of water
- ❑Swelling
- ❑Drying
- ❑Mechanical deformation

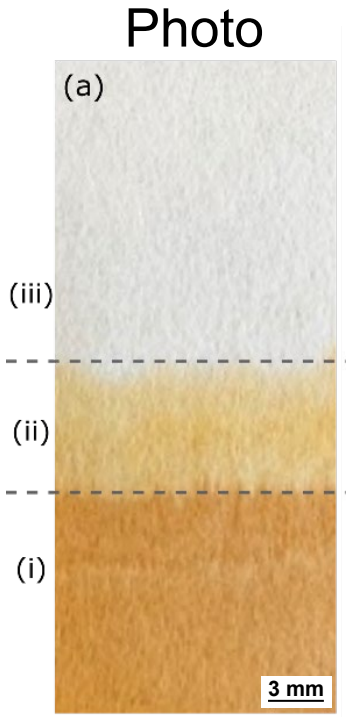
Enable new lignocellulosic products and markets!

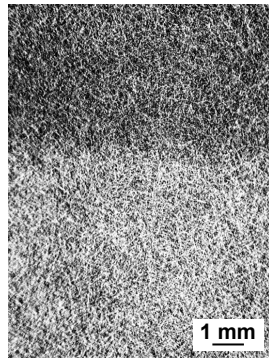
Mapping Water Absorbency in Paper with Iron Nanoparticles

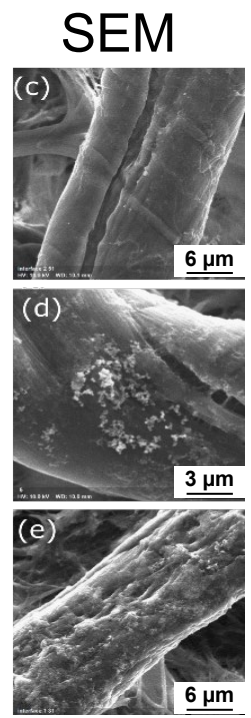
Liquid absorption test – iron oxide nanoparticles as tracers



(i) Immersed in NaOH: dark orange bottom
(ii) Wet through capillary flow: light orange middle
(iii) No contact: white top area







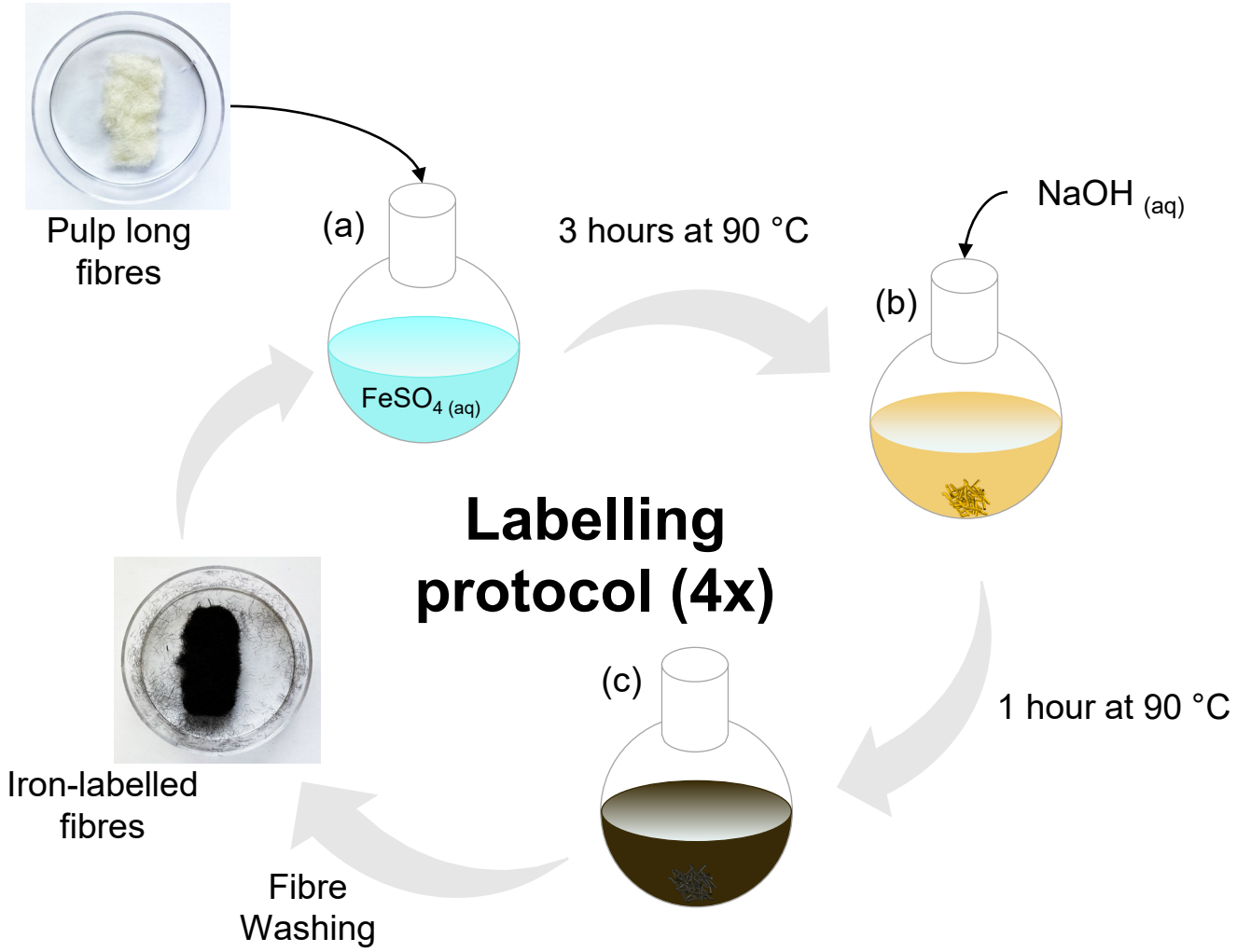
0% Fe

0.3% Fe

0.8% Fe

Ferreira et al. *Carbohydrate Polymers* **2023,** *311*, 120785

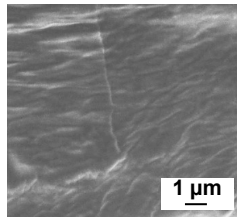
Iron Oxide Nanoparticle Labelling



- ❑Fully labelled fibres
- ❑Handsheets with 1 wt.% labelled fibres
- ❑Various fibre types
- ❑Image analysis

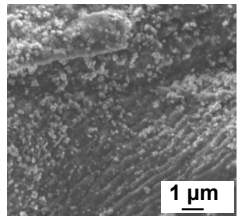
Fe-Labelled Fibres

Pulp fibre



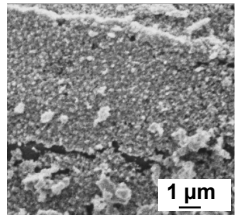
✓ 0 wt.% Fe

Labelled 1x

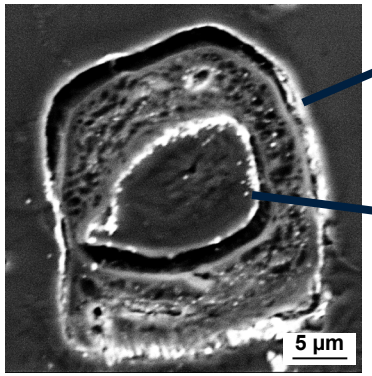


✓ Iron oxide NPs
✓ 4.4 wt.% Fe

Labelled 4x



✓ Robust iron oxide NPs coating
✓ 14.1 wt.% Fe



Fibre surface coated

Lumen surface coated

Magnetic fibres

*Primarily magnetite (Fe_3O_4) – $Fe^{(II)}$ + $Fe^{(III)}$ – brown/black; iron oxy(hydroxi)des

Enhanced Image Processing

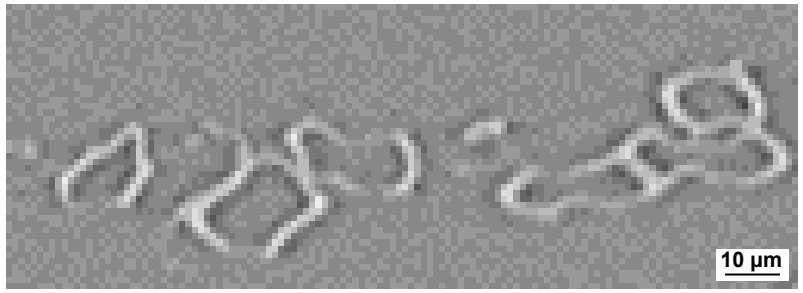
✓ Cross section microCT images

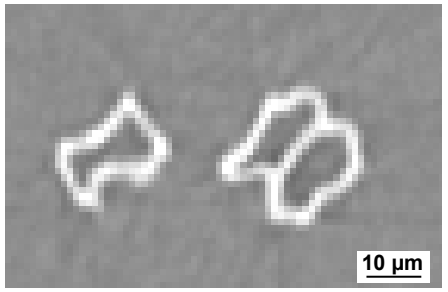
Unlabelled Fibres

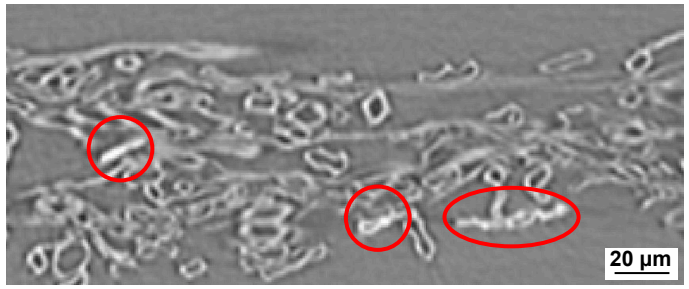
Labelled Fibres

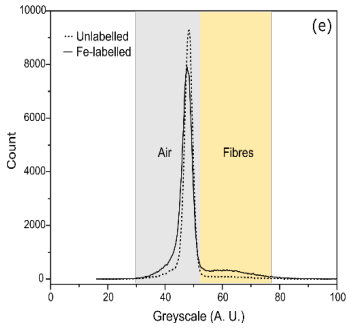
Unlabelled and labelled fibres

Raw images





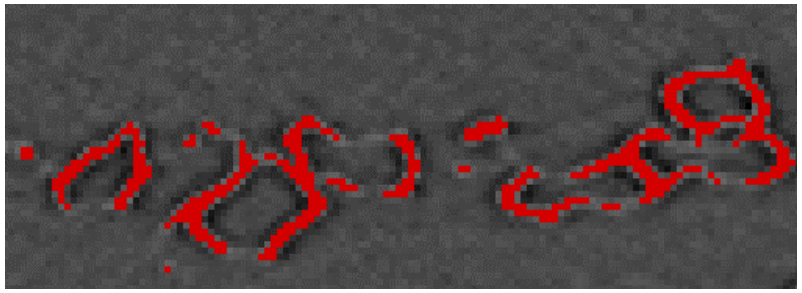


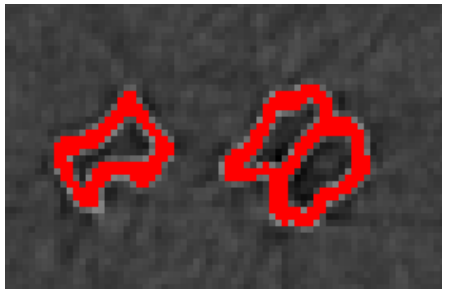


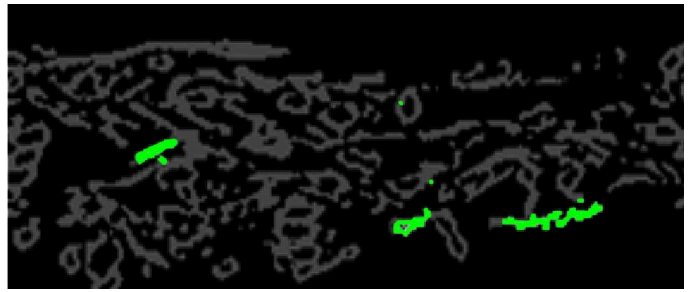
Intensity-based thresholding

Advanced segmentation

Processed image

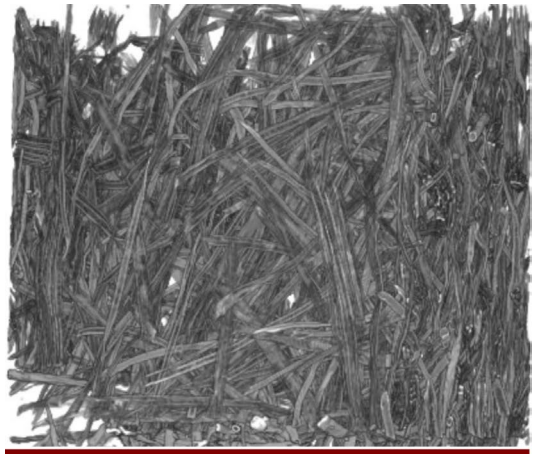




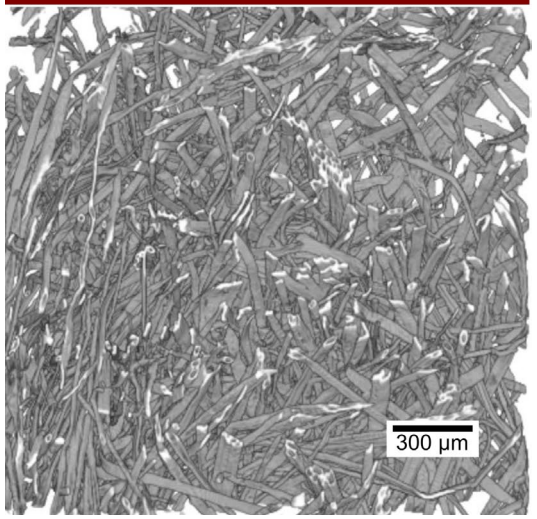


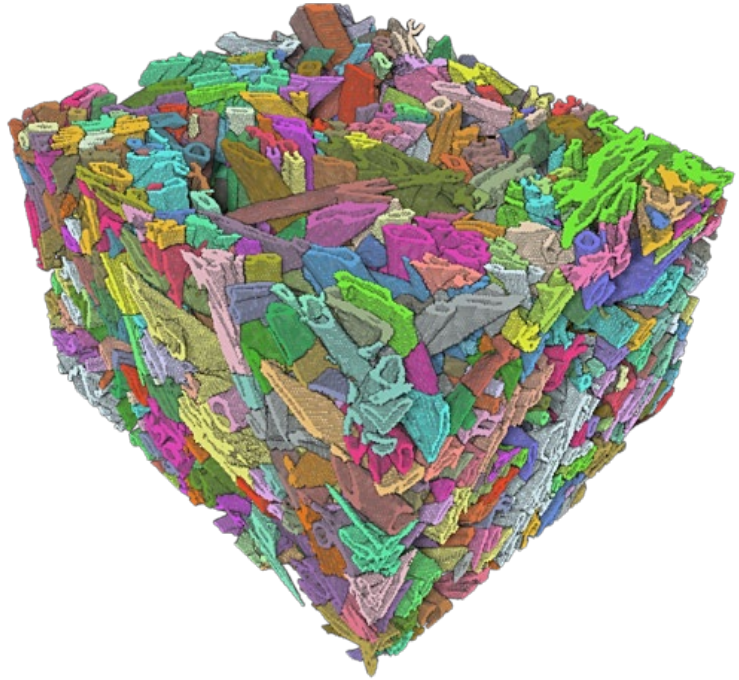
Improved MicroCT Contrast

Unlabelled Fibres

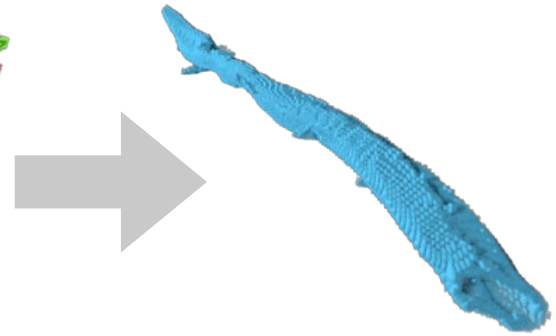


Labelled Fibres



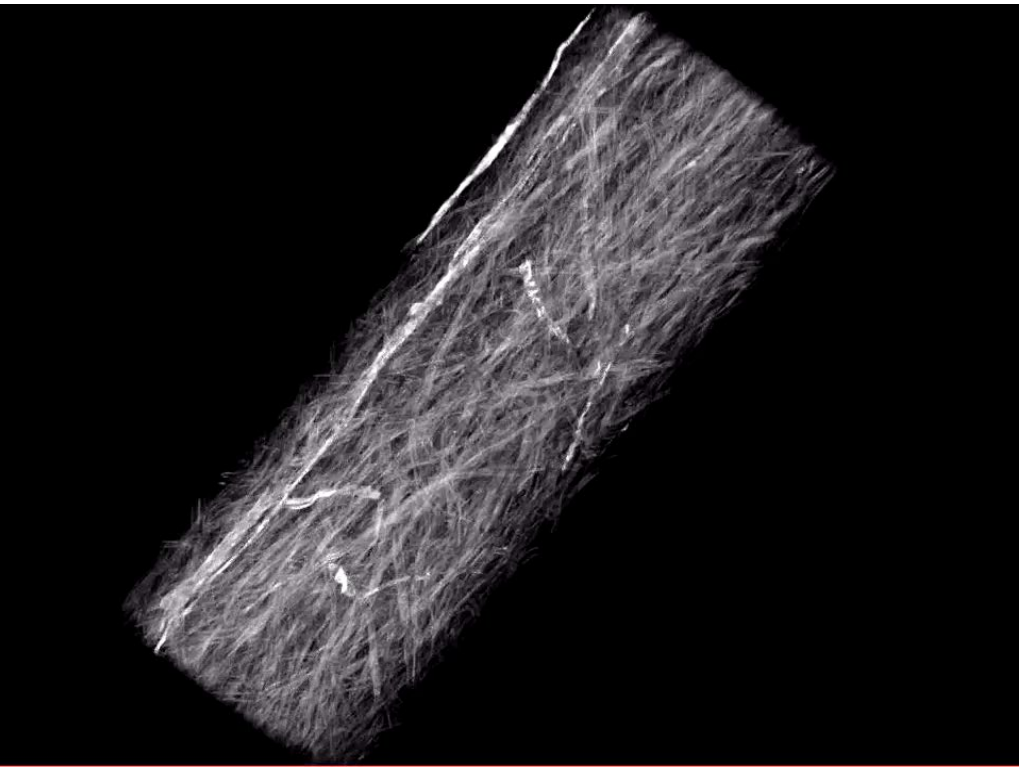


3D segmentation



Fibre segmentation

Unlabelled and labelled fibres



2.5 µm voxel scan - 3D render

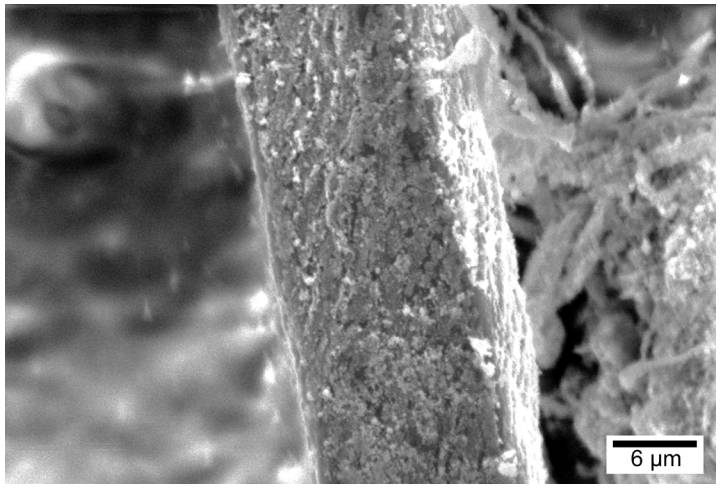
Labelled fibres can be distinguished and segmented

- Tensile stiffness of handsheet is statistically higher than unlabelled handsheet
- Loading 1 wt.% > theor. percolation of 0.5 wt.%

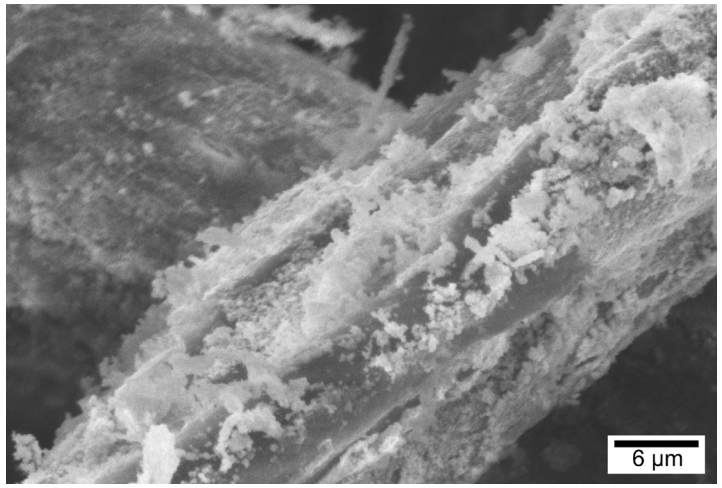
❑Optimize loading to not impact the mechanical properties significantly

Labelling Other Fibre Types

Iron oxide nanoparticle labelling protocol works with different fibre types



Fe-labelled TMP
23 wt.% Fe



Fe-labelled **oxidized** TMP
28 wt.% Fe

How does the surface of fibers affect nucleation and growth of NPs?

- ❑ Kinetics studies
- ❑ CNF model films

Fibre surface chemistry affects labelling efficiency

- ❑ Useful to spatially identify chemical modifications and fibre morphology

Conclusions

Pulp fibres labelled with iron oxide nanoparticles

✓High-contrast microCT images

✓Fibre segmentation

✓Potential to be applied as tracers in paper products

✓Support the development of lignocellulosic products

Acknowledgments – THANK YOU!

NSERC CRSNG

INNOVATION.CA
CANADA FOUNDATION FOR INNOVATION | FONDATION CANADIENNE POUR L'INNOVATION

BioProducts Institute

ERMP Network
(Energy Reduction in Mechanical Pulping)

Alberta Newsprint Company

CANFOR

PAPER EXCELLENCE

MILLAR WESTERN

HOLMEN PAPER

West Fraser

This research was conducted on the UBC Point Grey (Vancouver) campus, which sits on the traditional, ancestral, unceded territory of the xʷməθkʷəy̓əm (Musqueam) First Nation.