TECHNISCHE UNIVERSITÄT WIEN

Influence of the lignin content as a natural adhesive in biocomposites using holocellulose and Asplund fibers from spruce wood

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DI Cornelia Hofbauer

In cooperation with…

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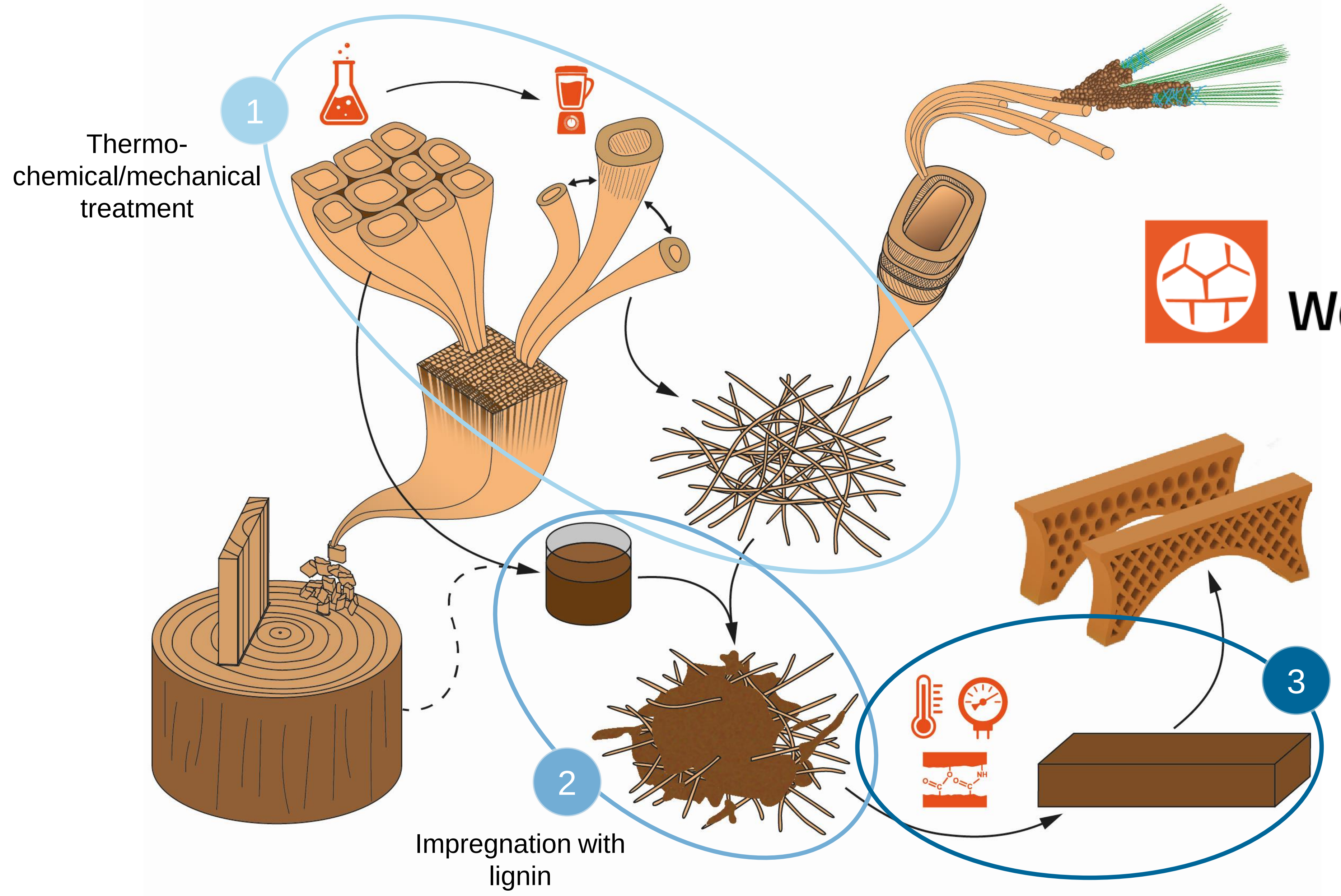


WoodComp3D

Christian Doppler Forschungsgesellschaft

What is the purpose…

- Long-term CO_2 storage
- Composites without nondegradable synthetic adhesives
- Biodegradable and environmentally friendly building material
- Improve the value chain of sawmill by-products (wood chips, sawdust, etc.)

Agenda

Study Part I – Pretreatment and Impregnation

Introduction and Materials
- Purpose and raw materials

Methods
- Asplund (Thermo-mechanical)
- PAA (Peracetic acid)

Results
- Microscopy
- Chemical composition

Conclusions

Study Part II – Pressing

Introduction and Materials
- Pressing

Methods
- Pressing conditions
- Strength testing

Results - PAA
- Density
- Bending Tests

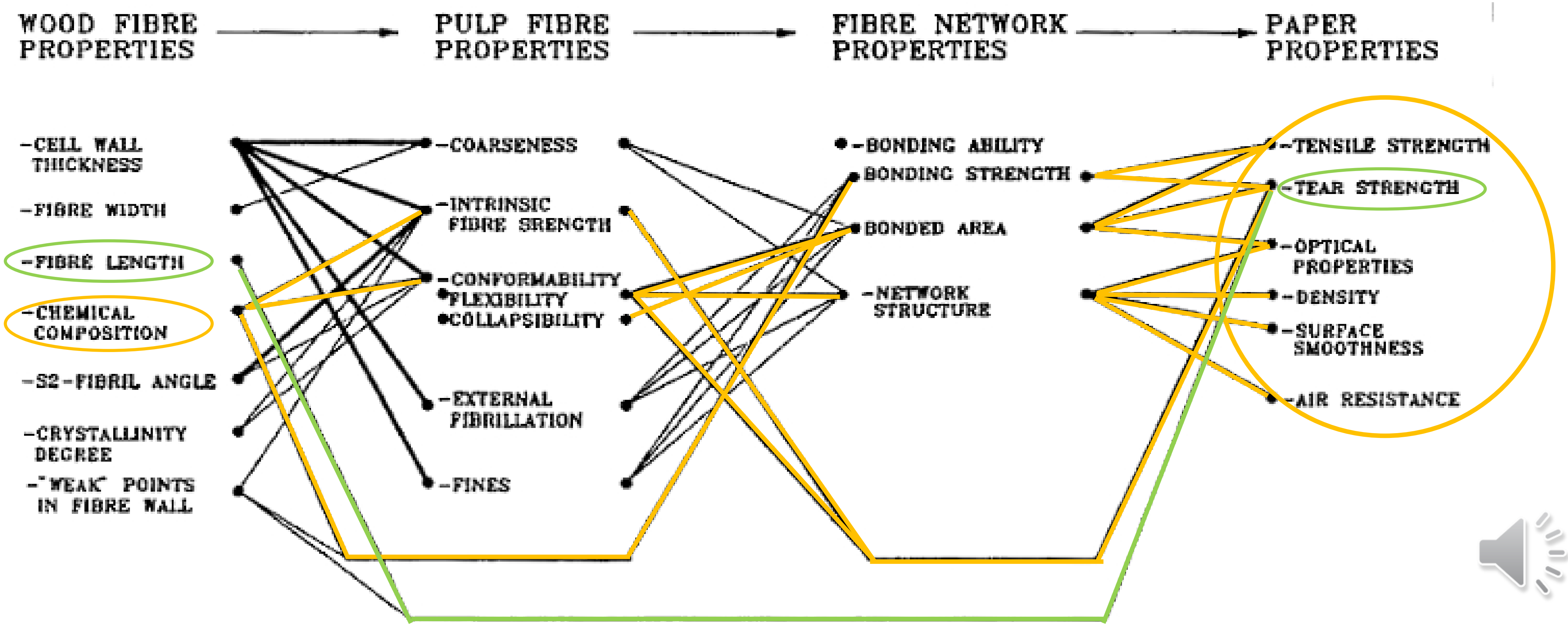
Conclusions

Study Part I

- Pretreatments
- Impregnation
- Chemical and optical analysis

Why PAA-pulping? Why Asplund pulping?

[1]



[1] PAAVILAINEN, Leena. Influence of fibre morphology and processing on the softwood sulphate pulp fibre and paper properties. 1993.

Why PAA-pulping?

- Fibrillation process without severe mechanical treatment
- Initial fiber length almost completely preserved
- Pulping at 80-100°C – moderate temperatures
- Selective extraction of lignin

Why Asplund[2] pulping?

- Fibrillation process with minor chemical effects → initial composition almost preserved
- Native lignin and hemicellulose for enhanced bonding abilities
- Low energy process (80 kWh/ton)

[2] PÉREZ, Alan D.; FISKARI, Juha; SCHUUR, Boelo. Delignification of low-energy mechanical pulp (asplund fibers) in a deep eutectic solvent system of choline chloride and lactic acid. Frontiers in chemistry, 2021, 9. Jg., S. 688291.

Raw material - PAA

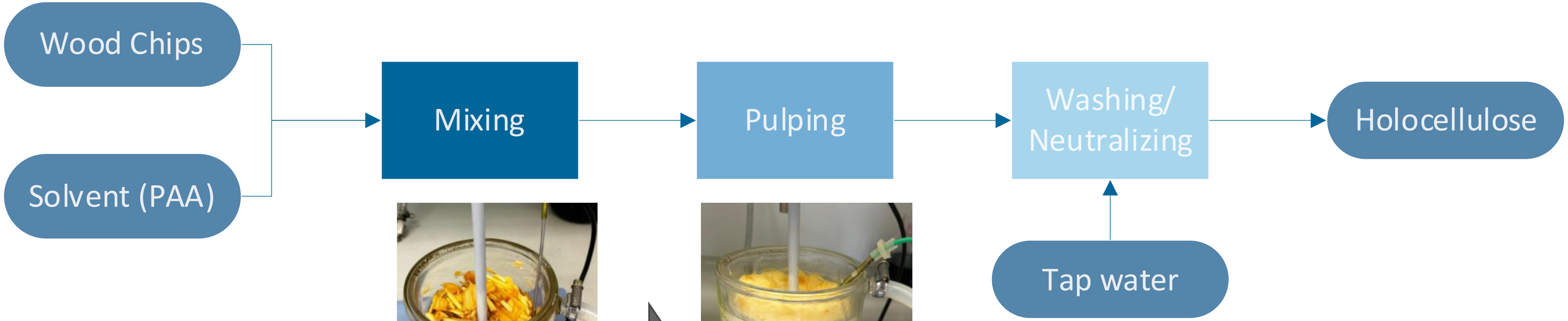
- Spruce wood chips
- Different particle sizes (1mm < x < 5cm)
- Air dried and stored open in a bag

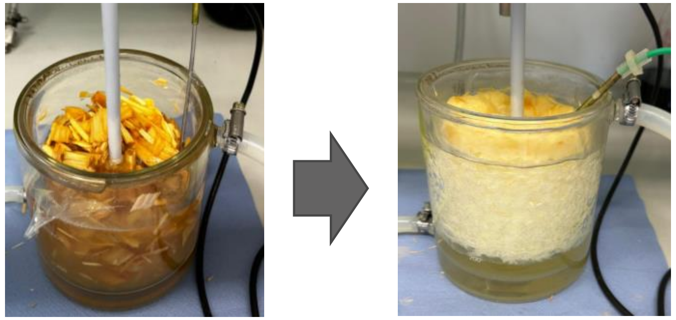
Asplund fibers[2]

- Mid Sweden University
- Norway spruce chips
- Pilot scale refiner
- Similar to the industrial process related to thermomechanical pulp (TMP)
- High moisture content for refining process
  - Heating was provided by steam (165°C)

[2] PÉREZ, Alan D.; FISKARI, Juha; SCHUUR, Boelo. Delignification of low-energy mechanical pulp (asplund fibers) in a deep eutectic solvent system of choline chloride and lactic acid. Frontiers in chemistry, 2021, 9. Jg., S. 688291.

PAA – process
Peracetic Acid[3]



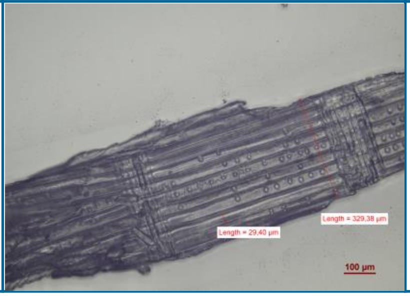
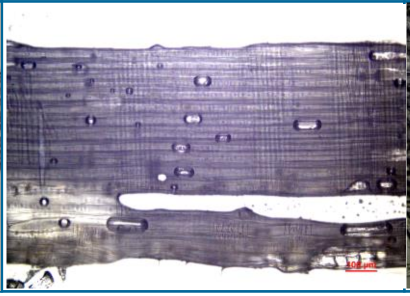
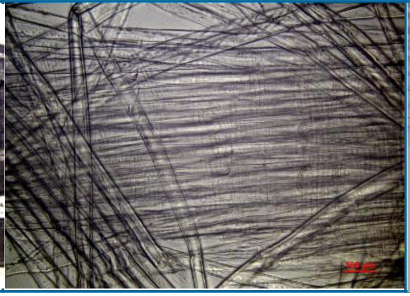
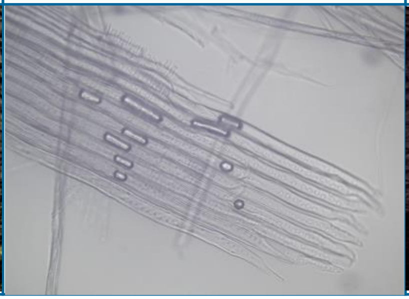


- PAA - concentrations
  - 99% Acetic acid
  - 35% Hydrogen peroxide
- Mixing rate - reagents: 1:1 in wt%

[3] WESTIN, Per-Oskar, et al. Single step PAA delignification of wood chips for high-performance holocellulose fibers. Cellulose, 2021, 28. Jg., Nr. 3, S. 1873-1880.

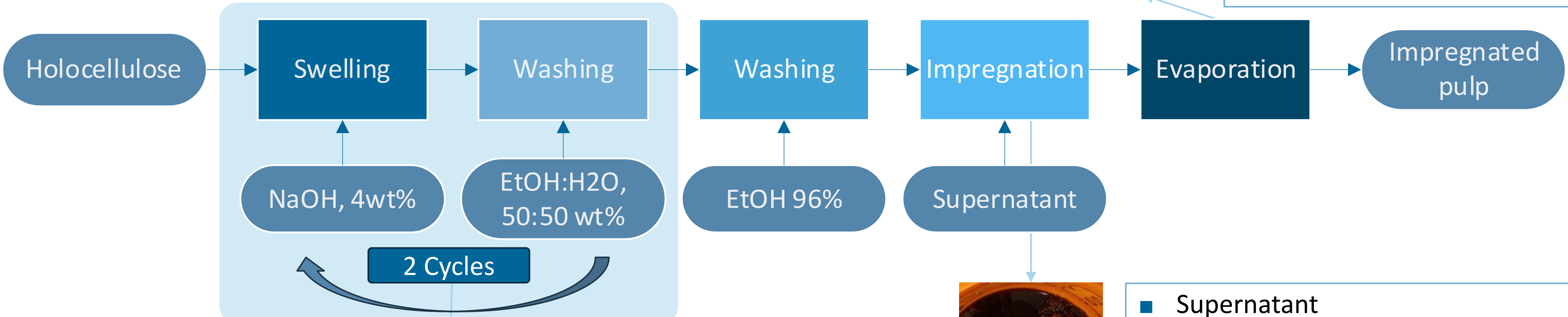
PAA – pulping

- Mixing wood chips and solvent at room temperature
  - Mixing ratio: 1:9 (wt%)
- 2 pulping cycles (50 min) with stirring at 100°C
- Between cycles mechanical treatment (3 min)

	1 – After cooking time of 50 min	2 – After first mechanical treatment	3 – After 100 min (2. Cycle)	4 – After second mechanical treatment	5 – After third mechanical treatment
100°C					

ISL-process
Impregnation with soluble lignin[4]

- Product
  - Ethanol was evaporated to the most possible during mixing
  - Lignin remains inside
  - Approx. 90 wt% moisture content

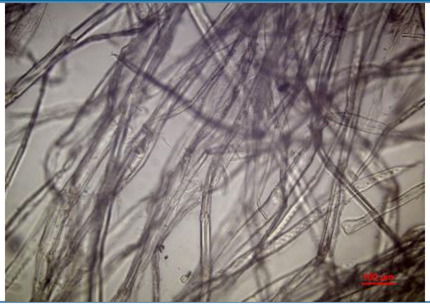
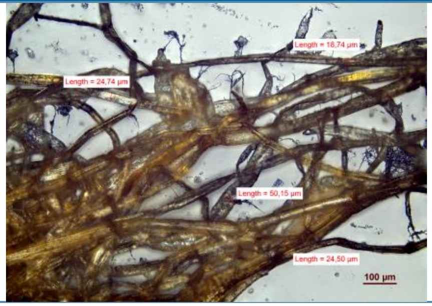
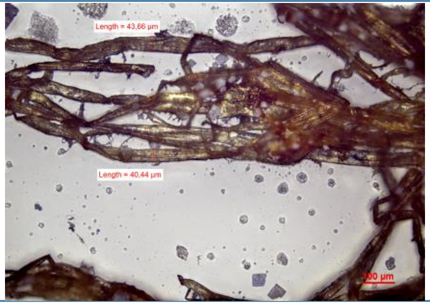


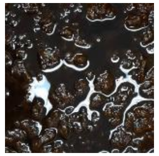
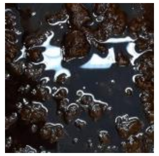
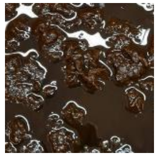
- Feedstock
  - Holocellulose pulp
  - Asplund fibers

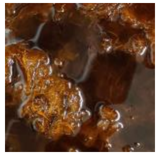
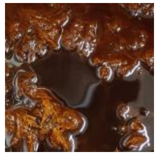
- Supernatant
  - Industrial kraft-lignin powder
  - Dispersed into pure ethanol
  - Centrifuged and separated

[4] JIANG, Bo, et al. Lignin as a wood-inspired binder enabled strong, water stable, and biodegradable paper for plastic replacement. *Advanced Functional Materials*, 2020, 30. Jg., Nr. 4, S. 1906307.

ISL-process steps

	1. Cycle step – ISL	2. Cycle step – ISL	3. Fibers directly after blending	4. Impregnated fibers after 24h
100°C				

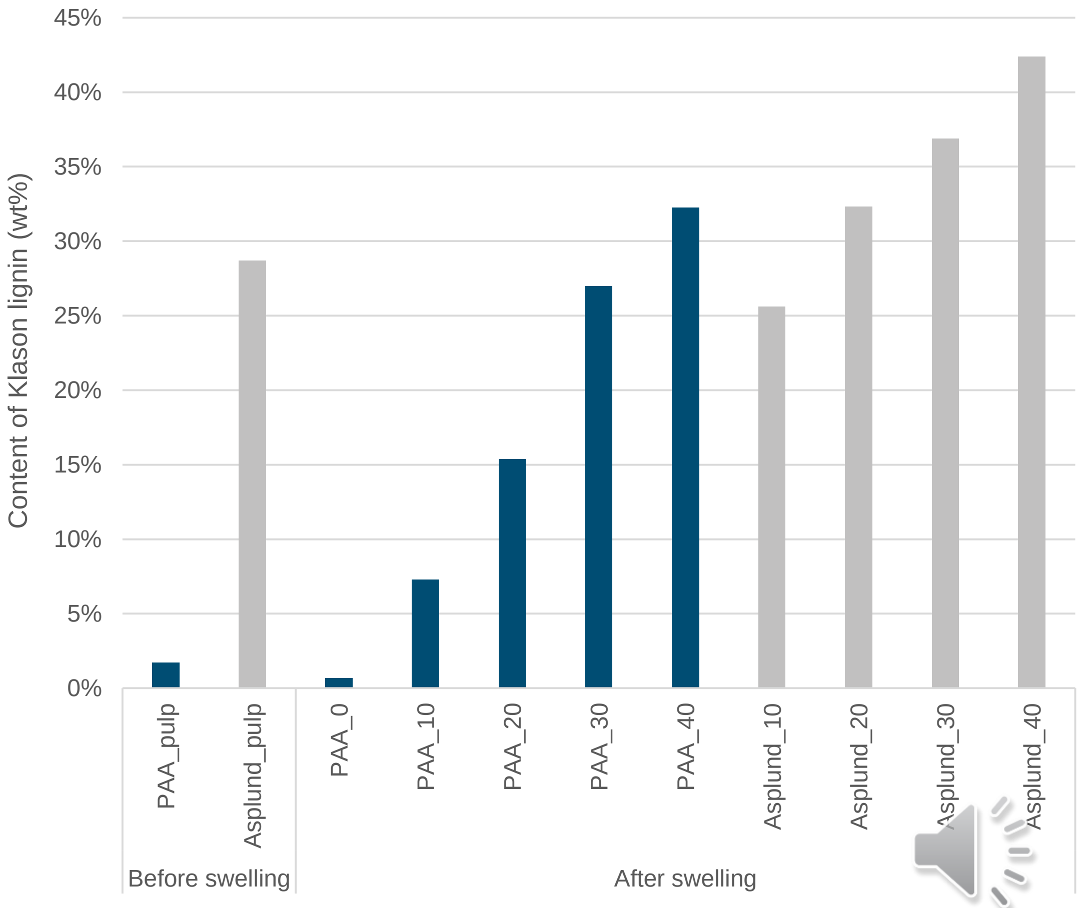
Impregnated PAA-pulp				
0wt%	10wt%	20wt%	30wt%	40wt%
				

Impregnated Asplund-pulp				
0wt%	10wt%	20wt%	30wt%	40wt%
				

Chemical analysis

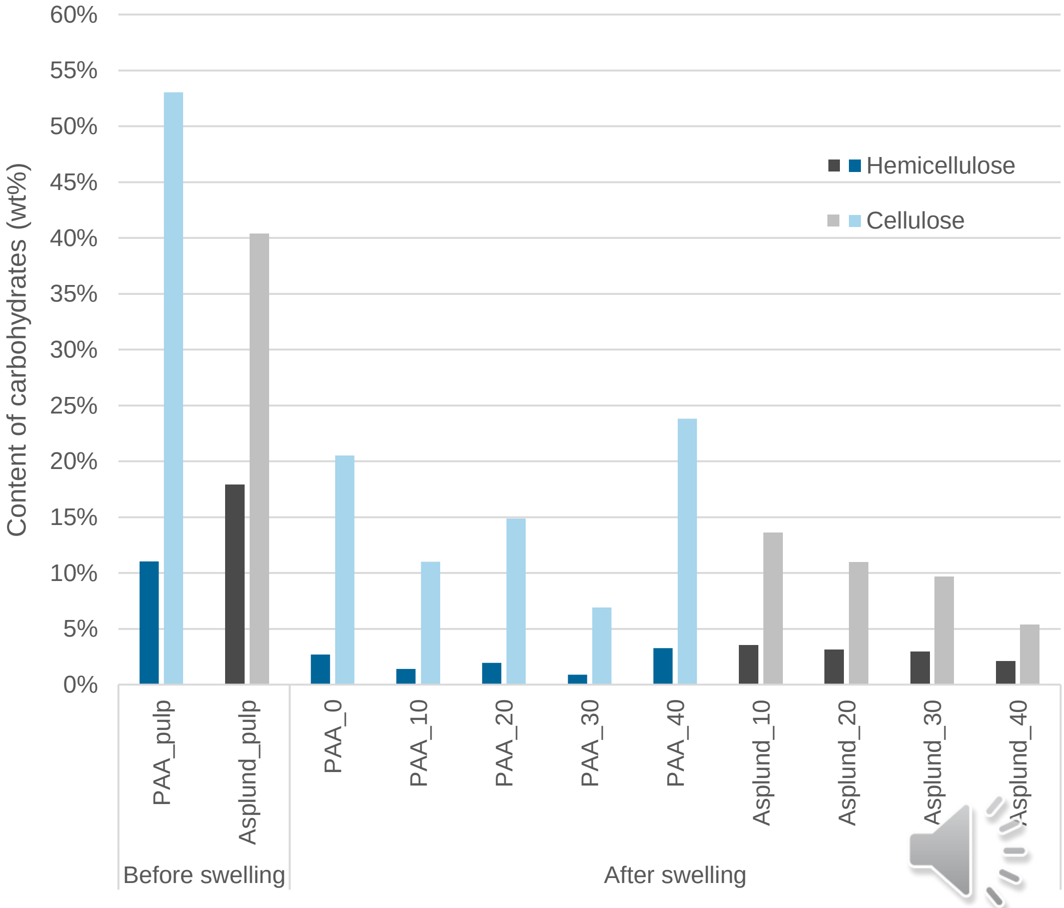
Klason lignin content

- Impregnation with different lignin contents:
  - 2 batches: Holocellulose (PAA_pulp) and Asplund fibers (Asplund_pulp)
  - Lignin contents: 0, 10, 20, 30 and 40wt%
  - (PAA_ 0 - 40 and Asplund_0 - 40)
- Losses of lignin during the ISL process in both pulps



Chemical analysis
Carbohydrates in solids

- The PAA-pulp has the highest glucose (cellulose) content
- After swelling the glucose content decreased significantly
- For the impregnated PAA-batch, no specific trend is visible – inhomogeneity of the pulping process
- The impregnated Asplund batch shows a trend



What we learned…

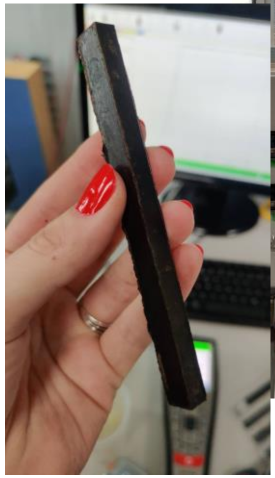
- Good fibrillation during the PAA-pulping process
- During the impregnation lignin is further extracted
- Swelling decreases the glucose content significantly
- Homogeneous distribution of the supernatant on the fibers
- Lignin content sufficient controllable in the pulp

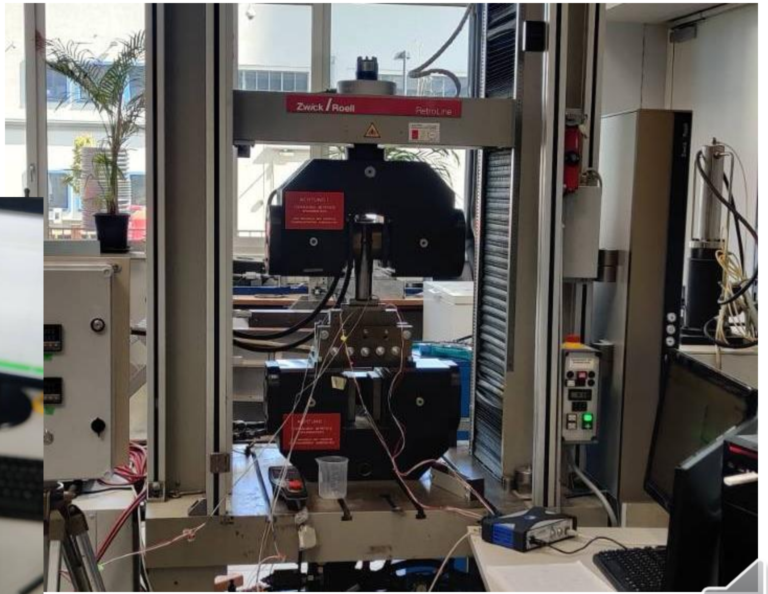
Study Part II

- Pressing
- Mechanical analysis

Why do we press?

- Chemical and physical bonding between the components under heat and pressure
- Mobilization of lignin in the fiber network
- Production of specimen called “beams”
  - Dimensions: 120x10x10 mm





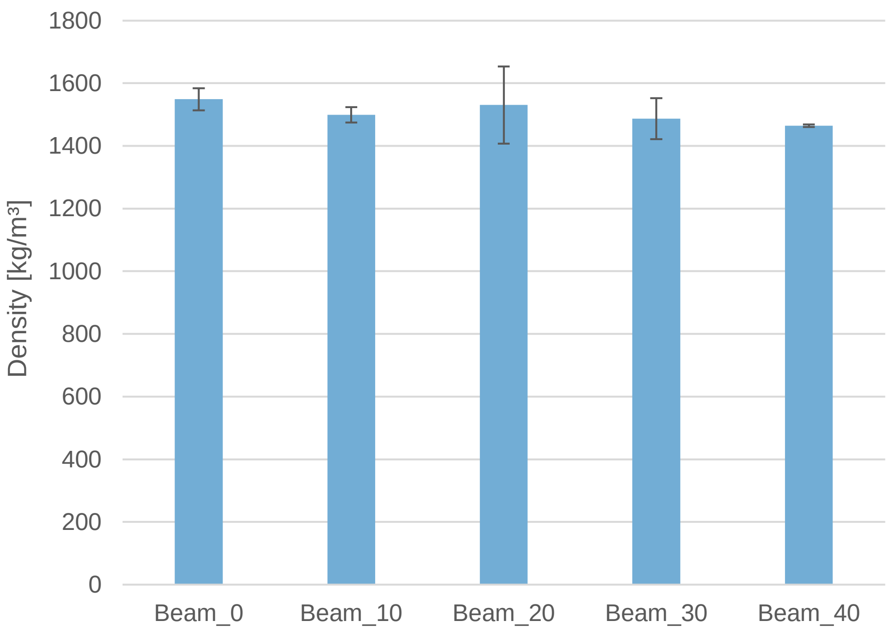
Pressing conditions

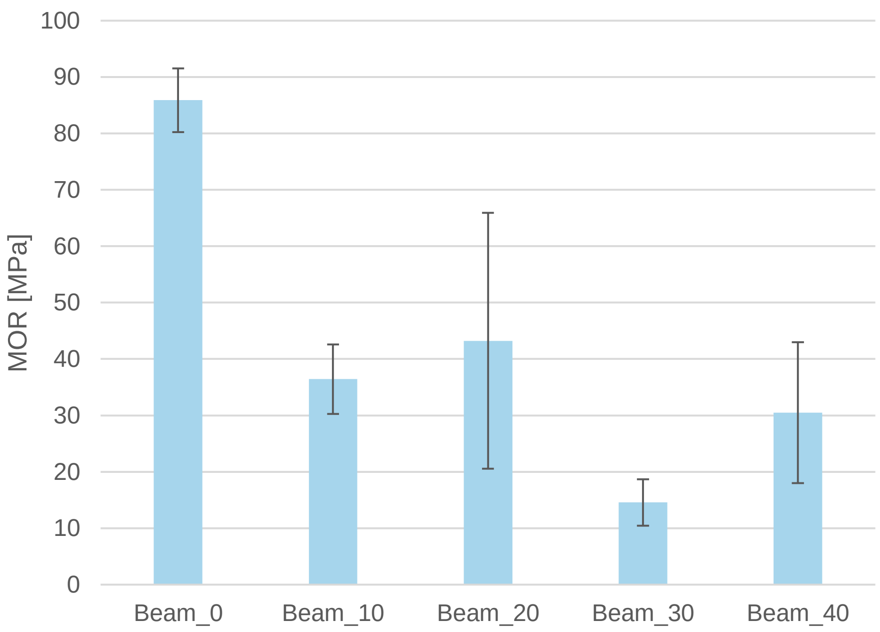
- Displacement controlled
- Max. temperature: 200 °C
- Holding time: 20min
- Force: 54kN
- Cooled after holding time to 40°C

Mechanical and physical analysis

- Density and dimensions of the “beams”
- Bending test
  - Until failure of the material
- Modulus of rupture (MOR)

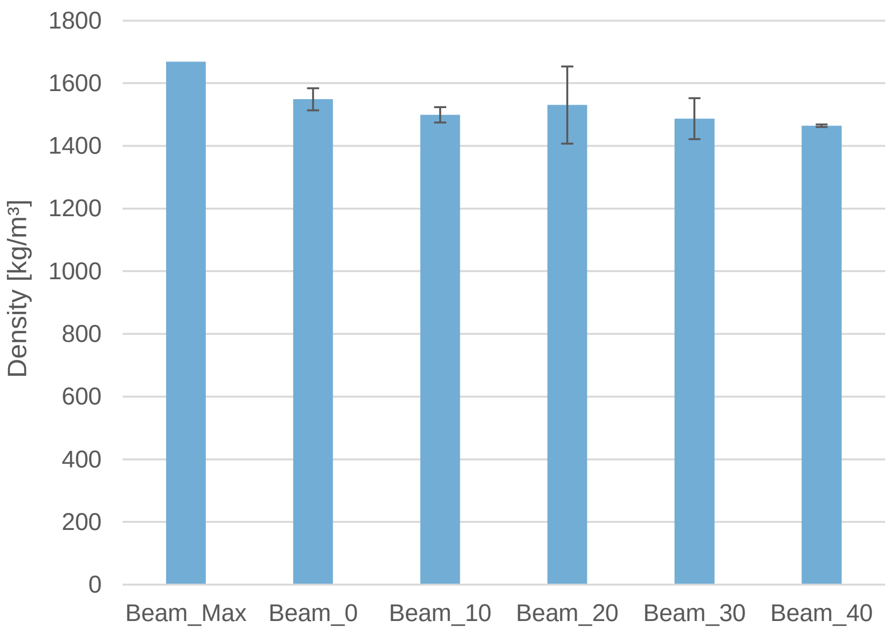
PAA – Physical and mechanical properties

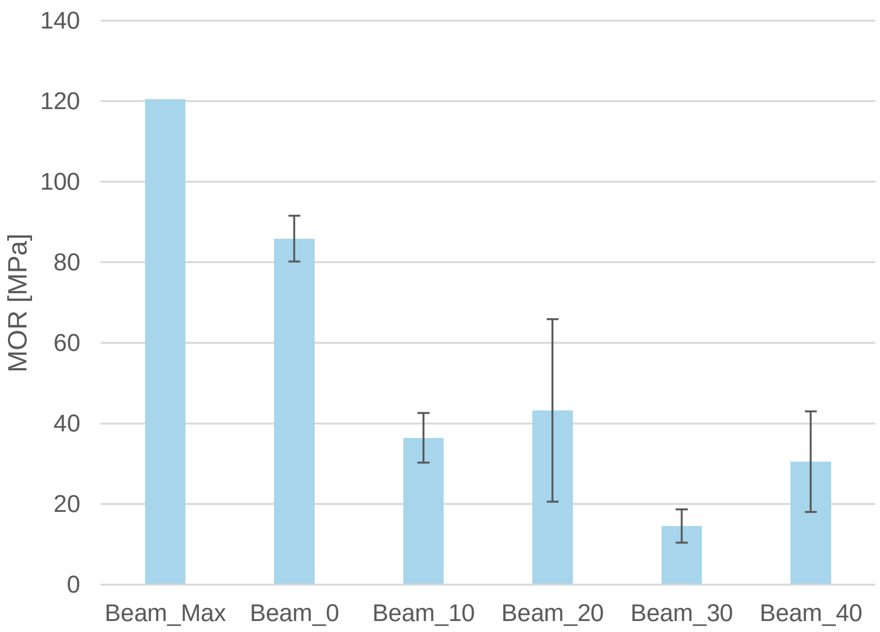




- No correlation visible between density and MOR
- High values can be achieved

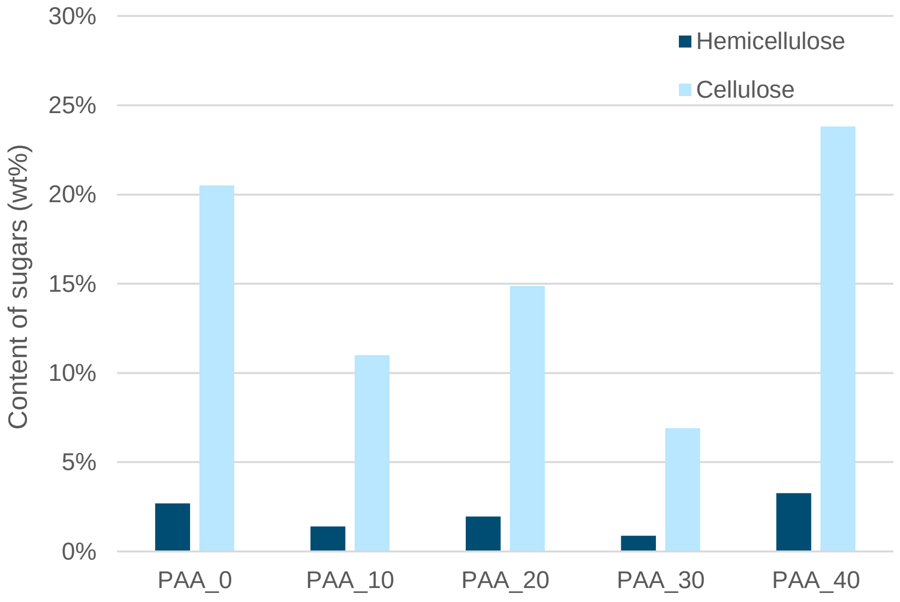
PAA – Physical and mechanical properties

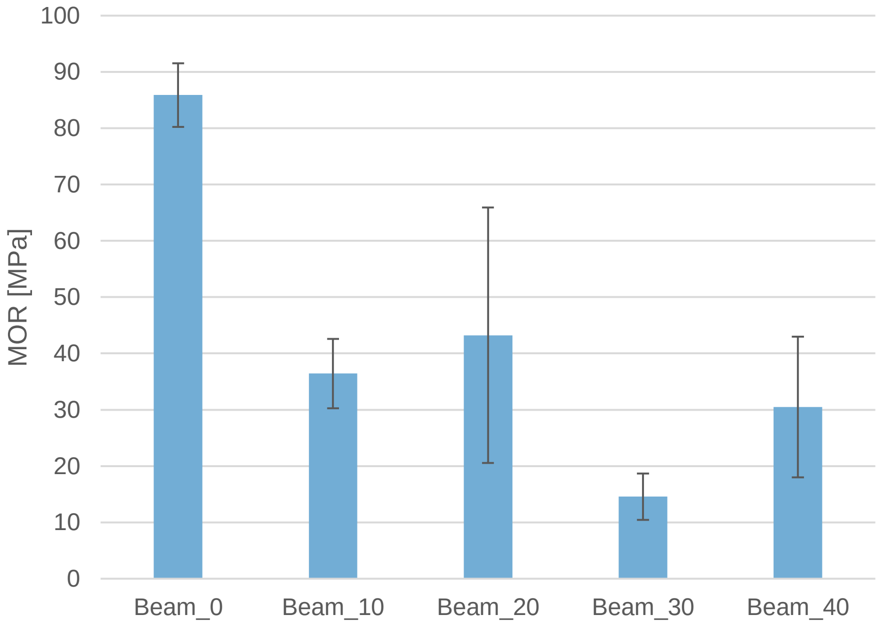




- Beam_Max from pre-studies
    - 16 wt% lignin

PAA – physical/chemical properties





- Control of the fiber composition crucial

What we learned…

- High values (90-120 MPa) can be achieved
- The MOR of the beams vary because of inhomogeneous fiber composition
- PAA-pulping needs to be more controllable
- The bonding ability and strength of the composite is very depending on the composition of the fiber and homogeneity of the pulp
- Lignin is a suitable natural binder

Outlook

- Press and evaluate the Asplund pulp
- More controllability over the whole process (pulp composition, homogeneity, losses)
- Optimization of the pulping and pressing conditions
- Study of different lignins for better binding abilities

Thank you!

- A big thank you to my colleagues:
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- And to the Mid Sweden University:
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