40th Danubia-Adria Symposium on Advances in Experimental Mechanics

September 24 - 27, 2024, Gdańsk, Poland

Characterization of mechanical properties of five hot-pressed lignins extracted from different feedstocks by load-controlled nanoindentation

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Motivation

Lignin, as illustrated in Fig. 1, stands as a crucial element found in plants. It is extracted in significant quantities as a byproduct of the pulp and paper manufacturing industry [1], establishing it as a primary resource within the domain of renewable raw materials [2]. Understanding the mechanical properties of hot-pressed lignin is crucial for the development of sustainable materials, especially lignin-based composites, as discussed in the WoodComp3D section. Additionally, it's important to note that the composition of lignin's macro-molecules can vary depending on the source material and extraction methods [1]. This variance prompts the question of whether these differences impact its mechanical properties. Consequently, expanding on previous nanoindentation campaigns [3], we undertook a study involving five distinct lignin samples, each characterized by varying feed stocks and extraction processes, employing nanoindentation techniques assisted by light microscopy.

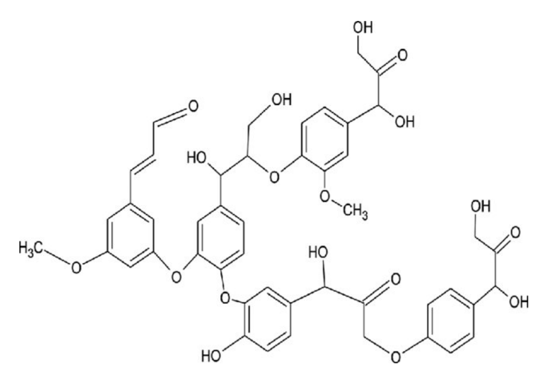


Fig 1. Structure of lignin

Materials and Methods

Fig. 2. Pressed lignin

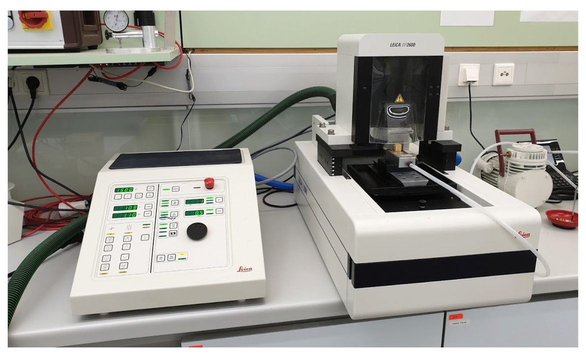


Fig. 3. Ultramiller

The lignin powders were hot-pressed into disc-shaped samples under 108 MPa for 2 min at 90 °C (Fig. 2).

The five types of lignin (Table 1) samples were embedded into epoxy resin.

The embedded lignin samples were polished to a mirror-like finish through a rotating diamond tip attached to an ultramiller (Fig. 3).

Table 1. Types of lignin

Lignin label	E-HW	OS-G	OS-HW	OS-SW	K-SW
Feedstock category	Hardwood	Grass	Hardwood	Softwood	Softwood
Feedstock	Beech	Corn silk, ballon flower	Beech	Spruce	Mixture of spruce and pine
Extraction process	Enz. Hydrolysis	Organosolv	Organosolv	Organosolv	Kraft pulping

We developed a new method that allows for accurate assessment of mechanical properties in porous lignin.

Nanoindentation

4250 double indents (Fig. 4) (8500 total indents) were performed (load controlled) at the five different types of lignins by means of a triboindenter (Fig. 5). The objective of the load function (refer to Fig. 4) was to separate the indentation response into both a plastic component and an elastic-viscoelastic component.The curves of each indent were evaluated according to the Oliver and Pharr method [4].

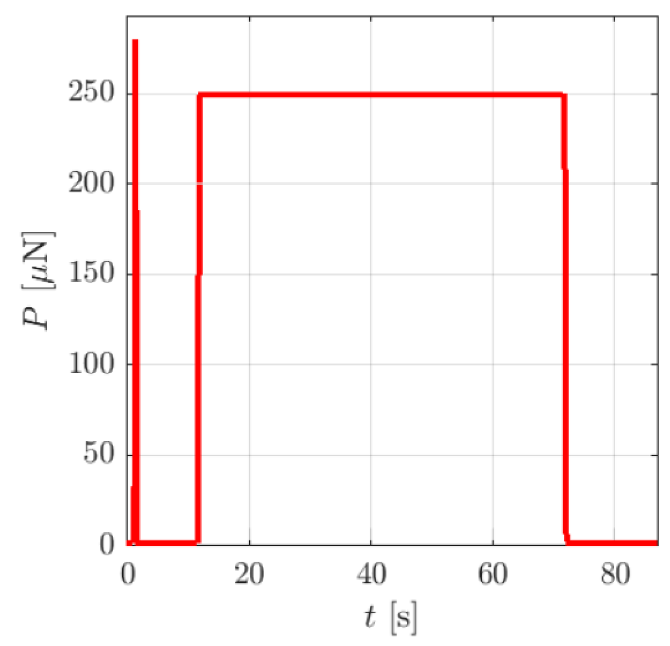


Fig 4. Double indent protocol

Fig 5. Triboindenter

Results

Pores were filtered out by means of light microscopy (Fig. 6).Pores were filtered out by means of light microscopy (Fig. 6).

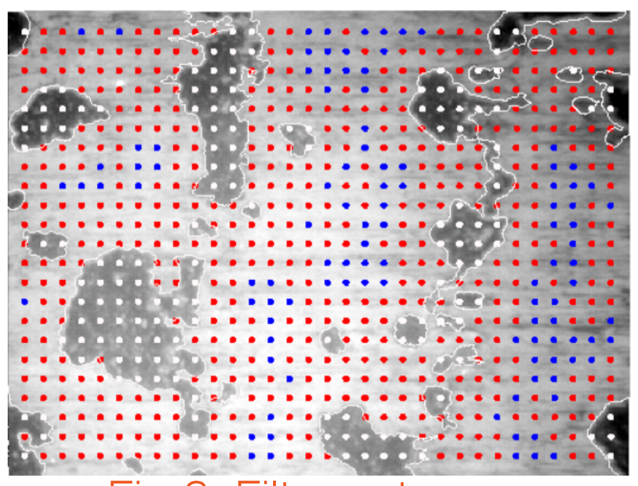


Fig 6. Filter out pores

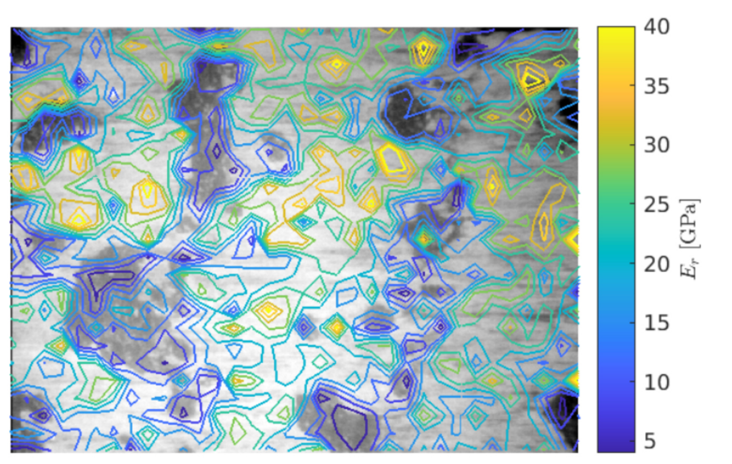


Fig 7. Reduced modulus gradient of OS-HW

The reduced modulus and hardness were identified for each type of lignin (Figs. 7 & 8).

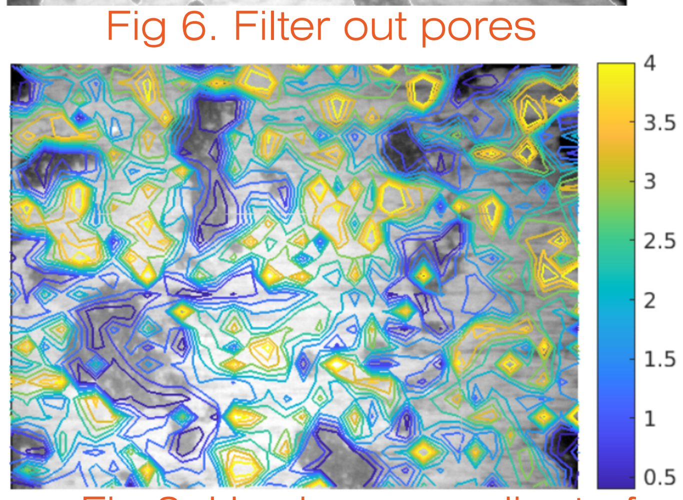
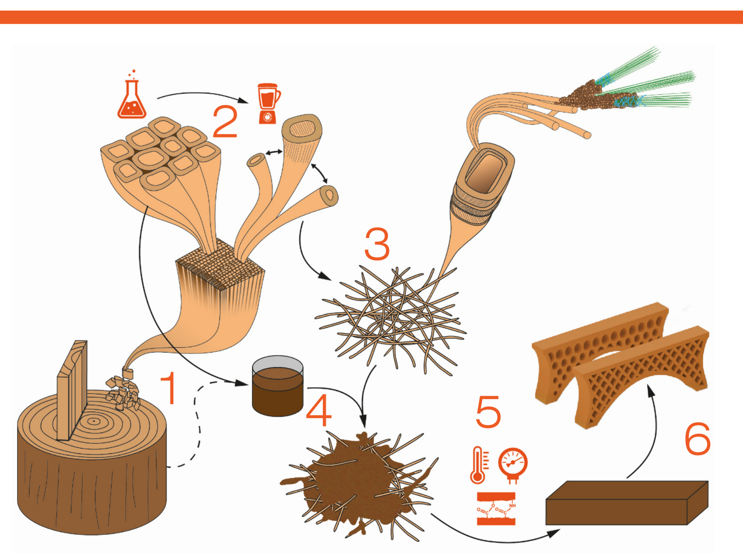


Fig 8. Hardness gradient of OS-HW



Layout of a high performing biocomposite:

WoodComp3D

1. By-products from sawmills, each characterized by different particle sizes.

2. Employing a combination of pretreatment methods involving thermo-chemical-mechanical processes to produce individual fibers with customizable lignocellulosic compositions.

3. Attaining fibers that are both easily accessible and possess a high degree of reactivity without altering their inherent microstructure.

4. Employing solubilized components (such as lignin, hemicellulose, and extractives) known for their strong reactivity and cross-linking capabilities as binding agents.

5. Implementing a reassembly phase that relies on temperature and pressure to facilitate the creation of robust bonds.

6. Crafting a uniform and high-performance biocomposite material suitable for applications in additive manufacturing processes.

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Acknowledgments: The financial support by the Austrian Federal Ministry of Labour and Economy, the National Foundation for Research, Technology and Development and the Christian Doppler Research Association is gratefully acknowledged. Furthermore, the authors would also like to express their gratitude to the CD laboratory company partner HS Timber for their financial support. The authors would also like to thank the support from the Austrian Science Fund (FWF) through the Y1093 START-project.

With the support of:

Federal Ministry Republic of Austria Labour and Economy