Enhancing Lignin Functionality through Targeted Process Optimization

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BERGISCHE UNIVERSITÄT WUPPERTAL

epf GRONINGEN 2025

I. Background

Lignocellulosic feedstock

- 2022: The annual global production of lignocellulosic biomass in nature is estimated at 181.5 billion tons - of which only 8.2 billion tons of biomass are currently used. [1]

Organosolv

- In addition to maximizing delignification and/or lignin purity, varying the process parameters of the acidic ethanolic organosolv process can also affect lignin structure. [2]

Motivation

- Lignins with high phenolic content could act as antioxidants, while lignins of low M_w are excellent precursors for the production of carbon fibers. [3]

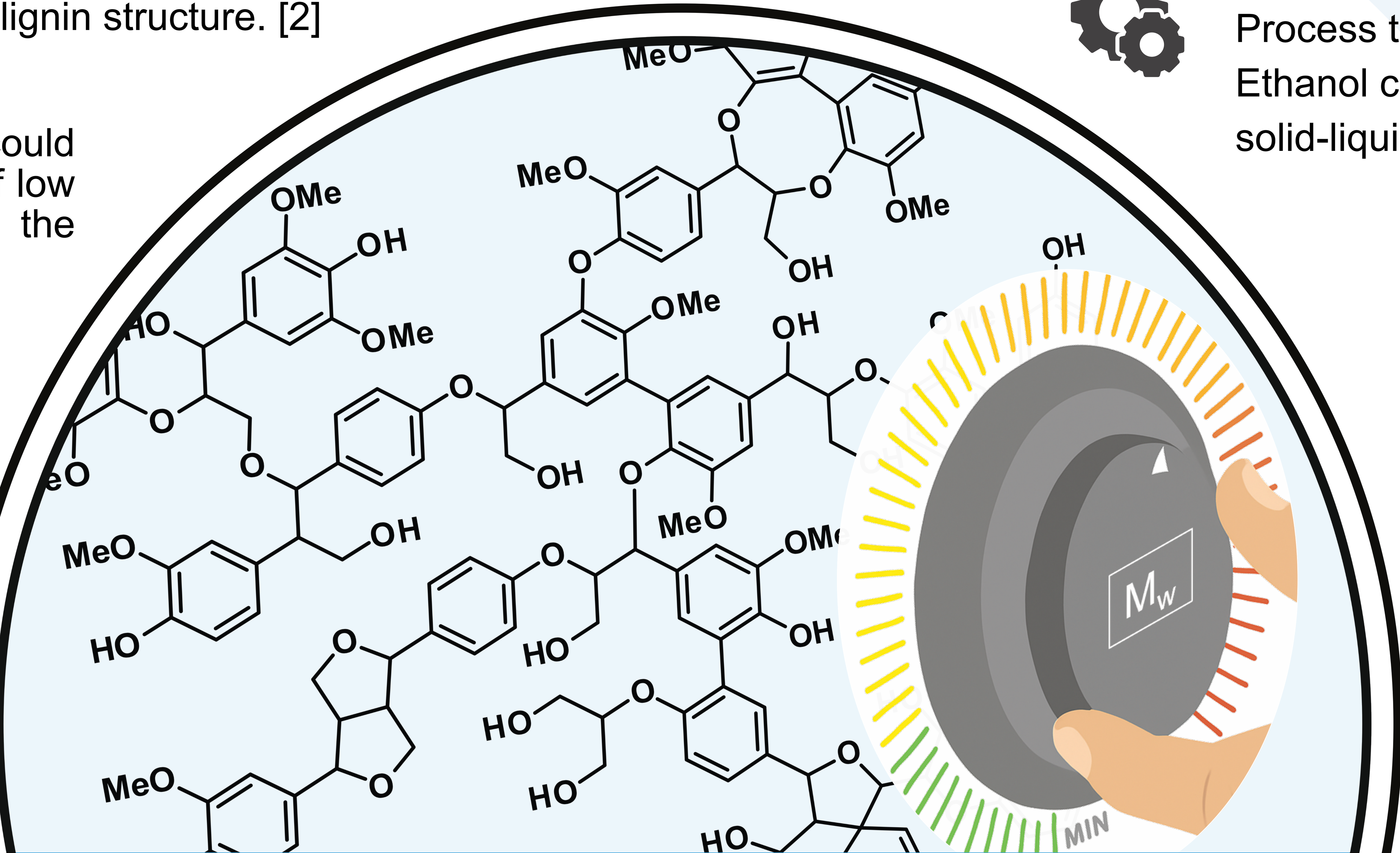
Fig 1. Tomato stems, vine prunings, grape pomace, and reeds were used as biomass.

Fig 2. Parr 4570 autoclave used for Organosolv process.

II. Organosolv Process Design

- The **aim of the experiments** was to determine how the pre-treatment and process parameters influence the structure of lignin, particularly its weight-average molecular weight (M_w) and total phenolic content (TPC).
- The significance of the process parameters was investigated and correlated with potential condensation and depolymerization reactions.

Particle size	2.0 - < 0.25 mm
Temperature range	150 - 240 °C
Process time range	60 - 120 min
Ethanol concentration	50 - 96 % (*v/v*)
solid-liquid ratio	1:8

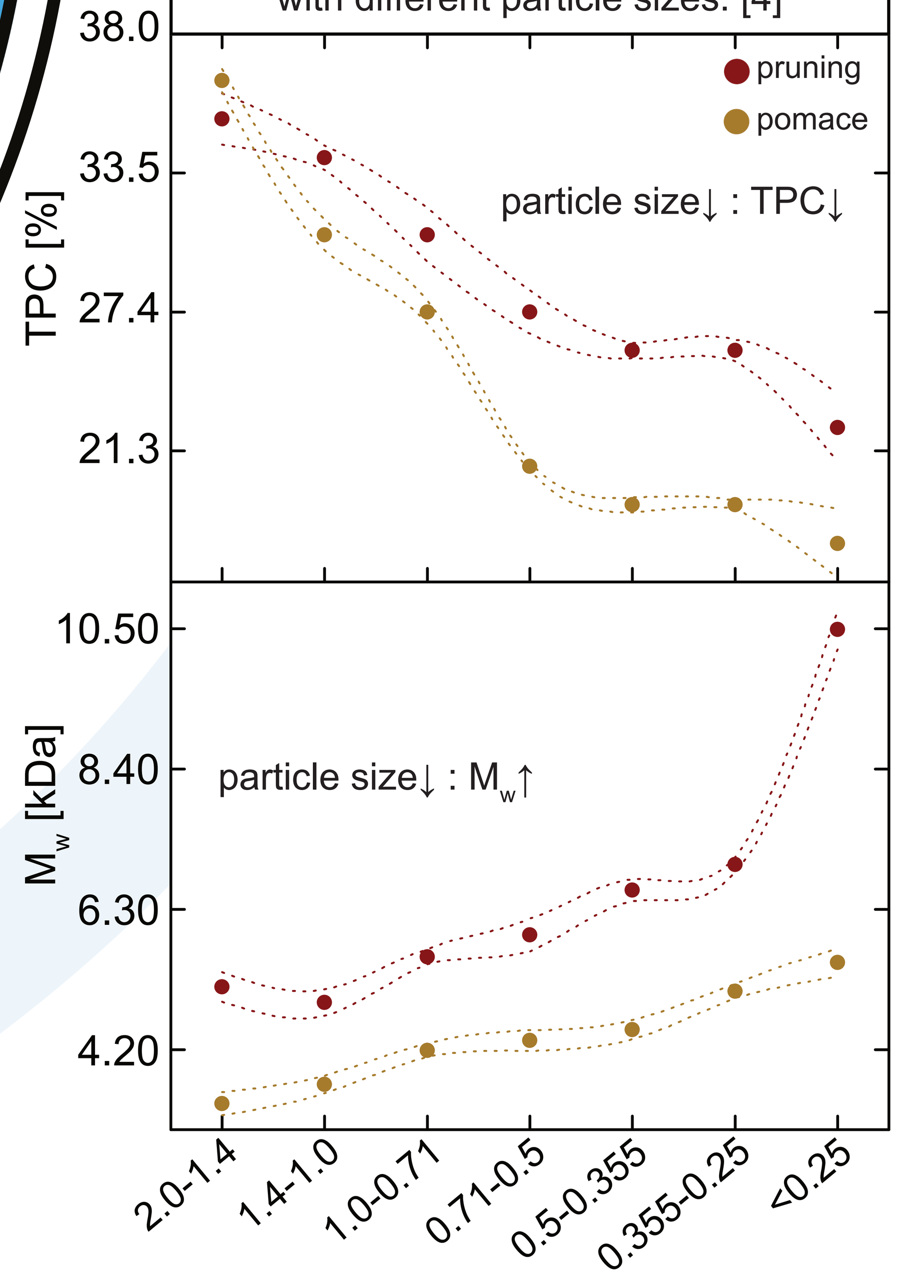


Tuning Lignin Structure via Targeted Organosolv Processing

III. Lignin Analysis

The purity of the lignins (> 96 wt%) was determined using NREL (TP-510-42618). The lignin was analyzed by SEC (0.1 M NaOH) and UV/Vis (FC reagent) and additionally by HSQC-NMR (DMSO-d6) and FTIR.

Fig 3. Dependence of TPC and M_w on lignins from two different biomasses with different particle sizes. [4]



Ethanol conc., temperature, and particle size significantly affect TPC and M_w. These effects were consistently observed across different biomass types.

- Reducing particle size led to decreased TPC and increased M_w, indicating enhanced condensation during the Organosolv process.
- HSQC-NMR confirmed this by showing fewer β-O-4′ linkages and more condensed structures.
- This suggests that mechanochemical effects during size reduction may trigger condensation, leading to increased M_w and reduced TPC.

IV. Outlook

Broadening the study to other process parameters (e.g., catalysts, solid-liquid ratios) to assess their effect on lignin structure, and to combine these findings with yield optimization efforts.

- Ethanol concentration had the strongest impact on lignin M_w; lower levels significantly reduced it (see **Fig 5.**).
- Temperature showed a nonlinear effect, suggesting an optimal range for controlled depolymerization (minimum around 200°C).
- Time had no significant impact on M_w.

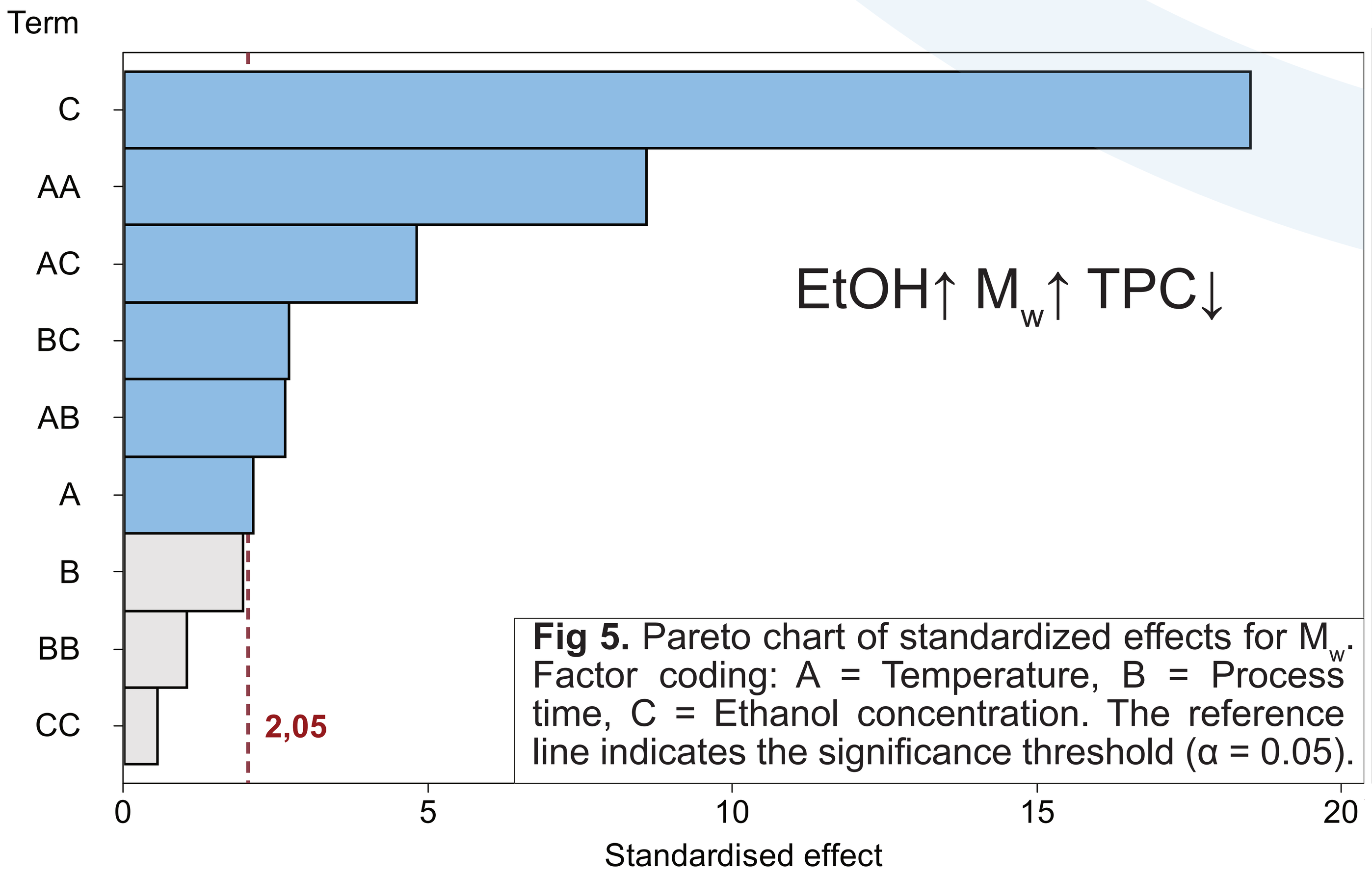
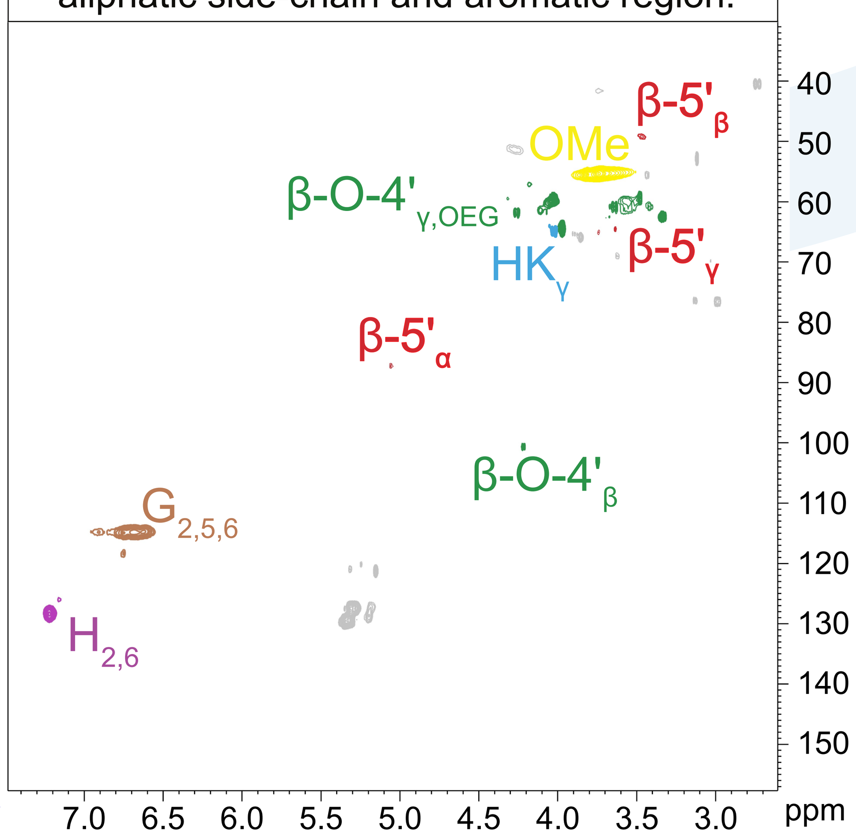


Fig 5. Pareto chart of standardized effects for M_w. Factor coding: A = Temperature, B = Process time, C = Ethanol concentration. The reference line indicates the significance threshold (α = 0.05).

Fig 4. HSQC NMR spectrum of a grape pomace (2.0-1.4 mm) lignin, showing the aliphatic side-chain and aromatic region.



Acknowledgment

Funding by BMBF (13FH102PX8 & 13FH522KB2), BRSU Graduate Institute and TREE Institute. The authors gratefully acknowledge Hochschule Geisenheim University and University of Bonn for providing the biomasses.

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