

Separation of lignin phenols with supercritical High Performance Chromatography (scHPLC)

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Introduction

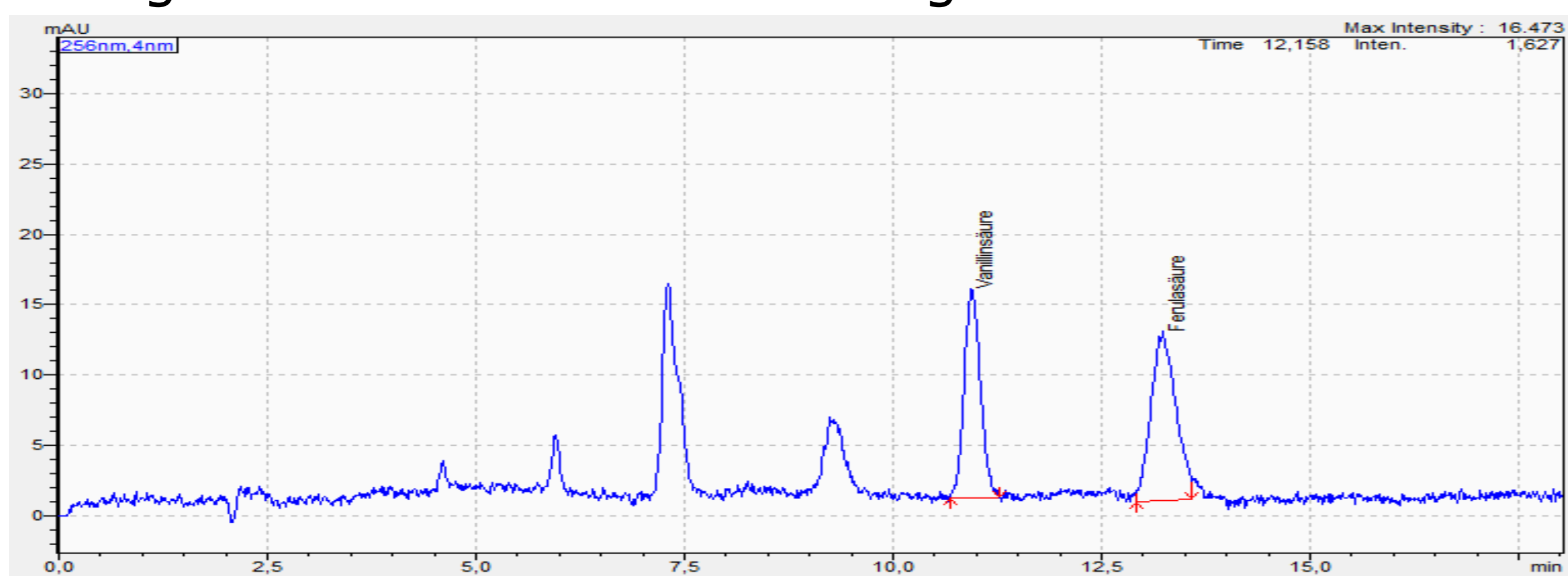
- Extraction and chromatographic separation of lignin phenols with supercritical CO₂
- Investigation of the separation mechanism for suitability to separate lignin phenols
- Variation of the measurement parameters and optimization of the separation stage number on the basis of vanillin
- Design of experiments for supercritical parameters optimization (pressure, temperature, modifier, flow rate)
- Analysis of enzymatic lignin degradation products with the optimized method

Methodology

- Hydrophilic Silica (SiO₂) column with a supercritical CO₂ chromatography system and methanol modifier
- Variation of pressure (100-155 bar), temperature (40-55 °C, flow rate (0.2-1.6 ml/min), methanol content (5-30%)
- Standards used for optimization: Acetosyringone, Acetovanillon, Ferulic acid, para-coumaric acid, Syringaldehyde, Vanillin, Vanillic acid, 4-Hydroxyacetophenon, 4-Hydroxybenzaldehyde, 4-Hydroxybenzoic acid

Results | Lignin degradation

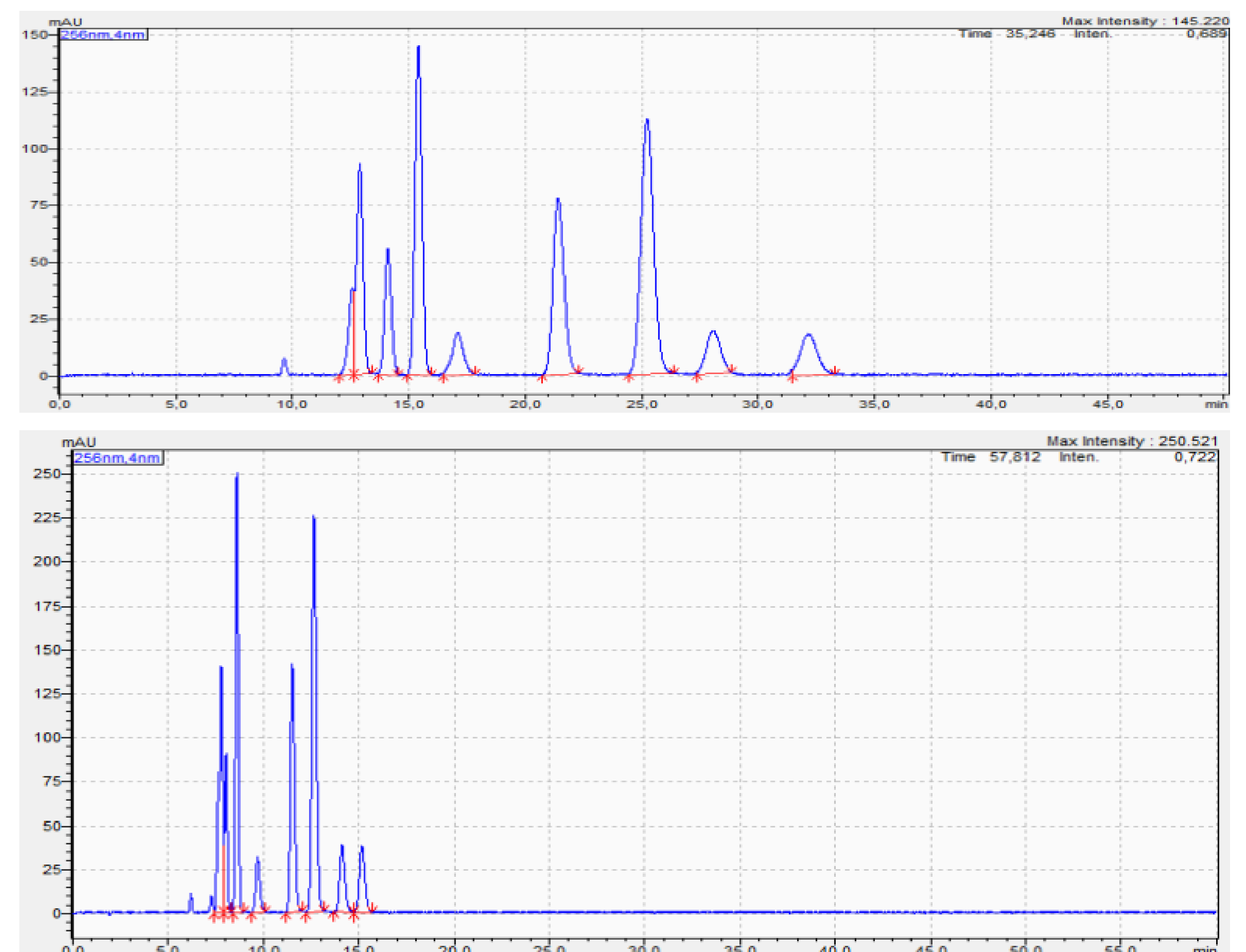
- Organosolv and Acetosolv lignins were used
- Chemical degradation took place during extraction
- Degradation with white-rot fungi in an airlift reactor



Vanillic acid and ferulic acid as primary phenolic products from lignin

Results | Supercritical Parameter Optimization

- Maximum of the separation stage number at a methanol content of 13.95%.
- According to Van-Deemter evaluations, the optimal flow rate value is 1.2 mL min⁻¹
- Number of separation stages have been increased to 12,822
- Separation time reduced to 17 minutes



Comparison of the chromatograms of the standard separation method (top) and aDoE optimized method (bottom) of ten lignin phenols

Summary | Outlook

- scHPLC separation of lignin phenol is possible with a resolution of $R > 1.5$ and separation stage number of over 10,000
- Flow rate and the methanol content of the mobile phase have the greatest influence on separation
- Yields of 2.6% for ferulic acid and 0.5% for vanillic acid were determined after degradation of beech lignin
- In addition, 4-hydroxyacetophenone was detected as a product in a microbial lignin degradation was detected