Rapid ^{1}H NMR spectroscopy method for lignin monomer quantification

Background/Objective

Reductive catalytic fractionation (RCF) is a promising lignin-first biorefining process wherein lignin is simultaneously extracted from the plant cell wall and depolymerized to a monomer-rich oil. Measurement of aromatic monomer yields from the RCF process is tedious, lacking commercially-available standards for chromatography, and requiring the use of toxic chlorinated solvents for extractions.

Approach

Here, we demonstrate an efficient, reliable, and widely accessible procedure using ^{1}H NMR spectroscopy for measurement of RCF-derived aromatics that does not require chromatography, available standards, and liquid-liquid extraction-based protocols.

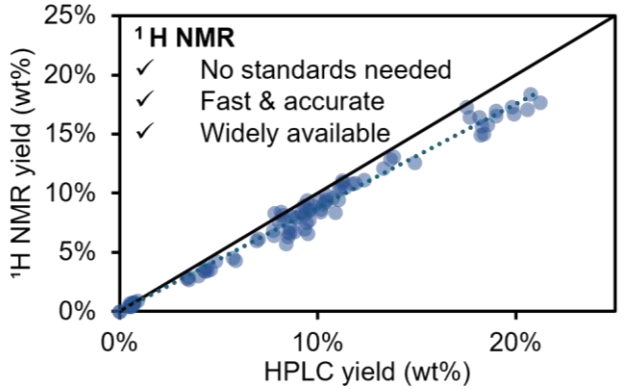
Results

- We compared this NMR method with standard high pressure liquid chromatography (HPLC) for 96 RCF oils with varying monomer selectivity.
- Average absolute deviations of individual monomer yields for the two methods were between 0.5-1.1 wt% (relative 11-17%), and R^2 values were above 0.9.
- The NMR method could be applied to measurement of crude RCF oils without liquid-liquid extraction (required for HPLC methods), providing a convenient way to quantify lignin extraction and aromatic monomer yield.

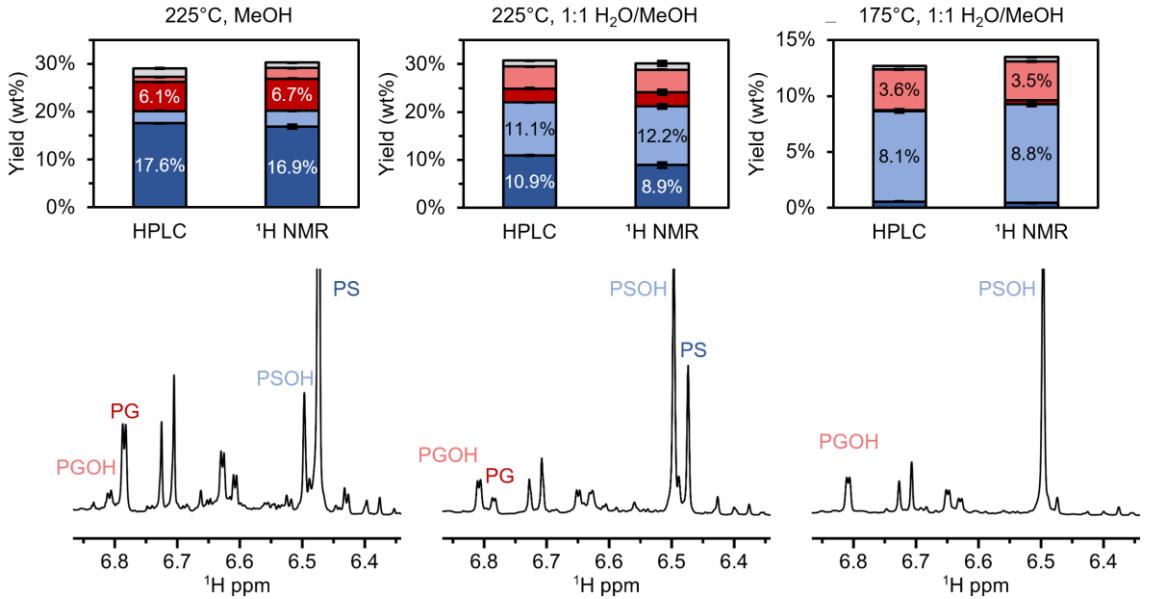
Significance/Impacts

This ^{1}H NMR method enables a rapid, high-throughput, and widely accessible to protocol for RCF monomer yield measurements, improving our ability to evaluate and improve this process for lignin biorefining.

Kenny *et al*. *ACS SusChemEng* (2026), 10.1021/acssuschemeng.5c10351



Correlation of aromatic monomer yield across 96 RCF oils with HPLC yield measurements



Comparison of monomer quantifications obtained from HPLC and ^{1}H NMR for reactions with alternative conditions