FACILITIES

NREL researcher Renee Happs loads samples and sets up experiments on NREL's 300 MHz NMR spectrometer. *Photo by Dennis Schroeder, NREL 62922*

R&D with **NREL'S Nuclear Magnetic Resonance (NMR) Facility**

NREL's state-of-the-art NMR Facility provides scientists the ability to analyze both solid and liquid samples on four NMR spectrometers.

NREL's NMR instruments provide distinct capabilities for analyzing a variety of samples from biomass to plastics to battery components. NREL scientists can monitor reactions—even in real time—to further their understanding of the fundamental chemical processes that may drive the development of next-generation sustainable fuels, materials, and products.

Common types of analyses in NREL's NMR Facility include:

- Rapid quantitation of monomeric sugars in biomass hydrolysates
- Bio-oil characterization and quantitative analysis
- Lignin composition and quantitative determination of both aromatic and side chain/bond content
- Temperature-dependent reaction rate determination
- Characterization of lignin-derived oils
- ^{13}C metabolic flux analysis and ^{13}C isotope tracing.

CORE CAPABILITIES AND APPLICATIONS

Sample Automation

Three NMR spectrometers offer sample automation for liquid-state analysis. The high-throughput Sample Jet on the 600 MHz spectrometer lets NREL scientists load up to 480 samples at a time. Additional sample changers on the open-use instruments enable NREL scientists to maximize their efficiency.

Cutting-Edge Solid-State Analysis

A dedicated solids instrument facilitates nondestructive, quantitative analysis of lignin and cellulosic composition of biomass feedstocks. The instrument also lets researchers analyze multiple cell wall components in a single spectrum. NREL's solid-state probes allow for unique studies of many biomass-related materials and polymers.

Open-Use Instruments

At NREL, scientists have access to two different NMR spectrometers for performing fundamental structural and chemical compositional analysis on a wide variety of liquid-state samples.

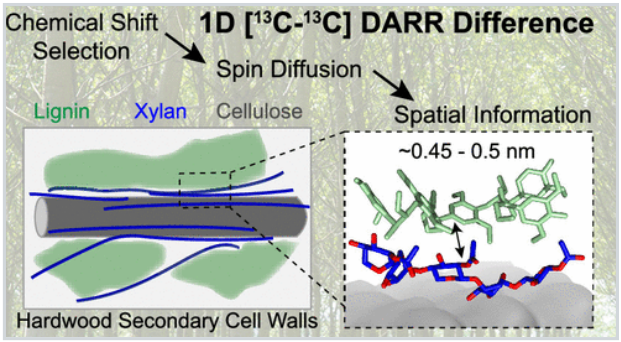
High-Resolution Magic Angle Spinning Spectroscopy

NREL's high-resolution magic angle spinning probe, equipped with Z-gradient, enables researchers to perform liquid-like NMR experiments on insoluble materials. As NREL scientists delve into materials and polymer chemistries, this specialized capability allows them to achieve much greater resolution to support their analysis.

RECENT SUCCESSES

Selective One-Dimensional ^{13}C–^{13}C Spin-Diffusion Solid-State NMR Methods Probe Spatial Arrangements in Biopolymers

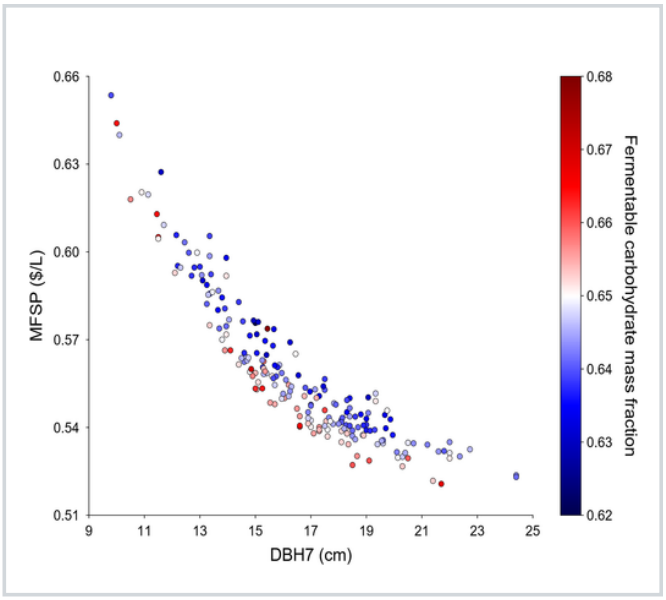
NREL and a team of scientists used a solid-state NMR technique on a variety of ^{13}C-enriched biopolymers using a selective one-dimensional version of a more common two-dimensional ^{13}C–^{13}C correlation experiment. By collapsing the 2D experiment into a much faster 1D version using selective shaped radio-frequency pulses (prior to a ^{13}C–^{13}C spin-diffusion period), they could conduct the experiment as much as 10–30 times faster. That accomplishment let them access spin-diffusion buildup curves, which contain distance information, in a reasonable timeframe. Using this "1D DARR difference" method, the researchers, for the first time, were able to measure the lignin/xylan inter-polymer distance at roughly 0.45–0.5 nm between xylan acetyl carbons and lignin aromatic groups. These measurements suggest that lignin/xylan inter-polymer interactions—relevant to biomass recalcitrance and the structural integrity of wood—arise from tight surface packing between those polymers, driven by xylan acetyl decorations.



Researchers were able to measure the lignin/xylan inter-polymer distance at roughly 0.45–0.5 nm between xylan acetyl carbons and lignin aromatic groups.

Researchers Point to Sugars as Key Factor in Ideal Feedstock for Biofuels

NREL researchers used ^{1}H NMR to analyze the sugar composition of over 900 poplar trees. By combining NMR and high-performance liquid chromatography data to build partial least squares models, they could perform high-throughput analysis of the biomass hydrolysates. The resulting sugar compositions were used for techno-economic analysis of poplar as a major feedstock, which revealed that growers could breed the trees for both size and sugar composition without trading one for the other.



NREL researchers plotted the minimum fuel selling price (MFSP) against diameter at breast height measurements at age 7 (DBH7) with total mass fraction of fermentable carbohydrates (glucan, xylan, arabinan) shown for each sample by color. For large DBH7, composition becomes an equally important driver of MFSP.

Highlighted Publications

B. Addison, et al. "Selective One-Dimensional ^{13}C–^{13}C Spin-Diffusion Solid-State Nuclear Magnetic Resonance Methods to Probe Spatial Arrangements in Biopolymers Including Plant Cell Walls, Peptides, and Spider Silk." *The Journal of Physical Chemistry B*. DOI: 10.1021/acs.jpcb.0c07759.

R. M. Happs, et al. "Economic Impact of Yield and Composition Variation in Bioenergy Crops: *Populus trichocarpa*." *Biofuels, Bioproducts and Biorefining*. DOI: 10.1002/bbb.2148.

C. A. Downes, et al. "Electrocatalytic CO_2 Reduction over Cu_3P Nanoparticles Generated via a Molecular Precursor Route." *ACS Applied Energy Materials*. DOI: 10.1021/acsaem.0c01360.

Find Out More

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