Researchers create green solvent to boost lignin applications

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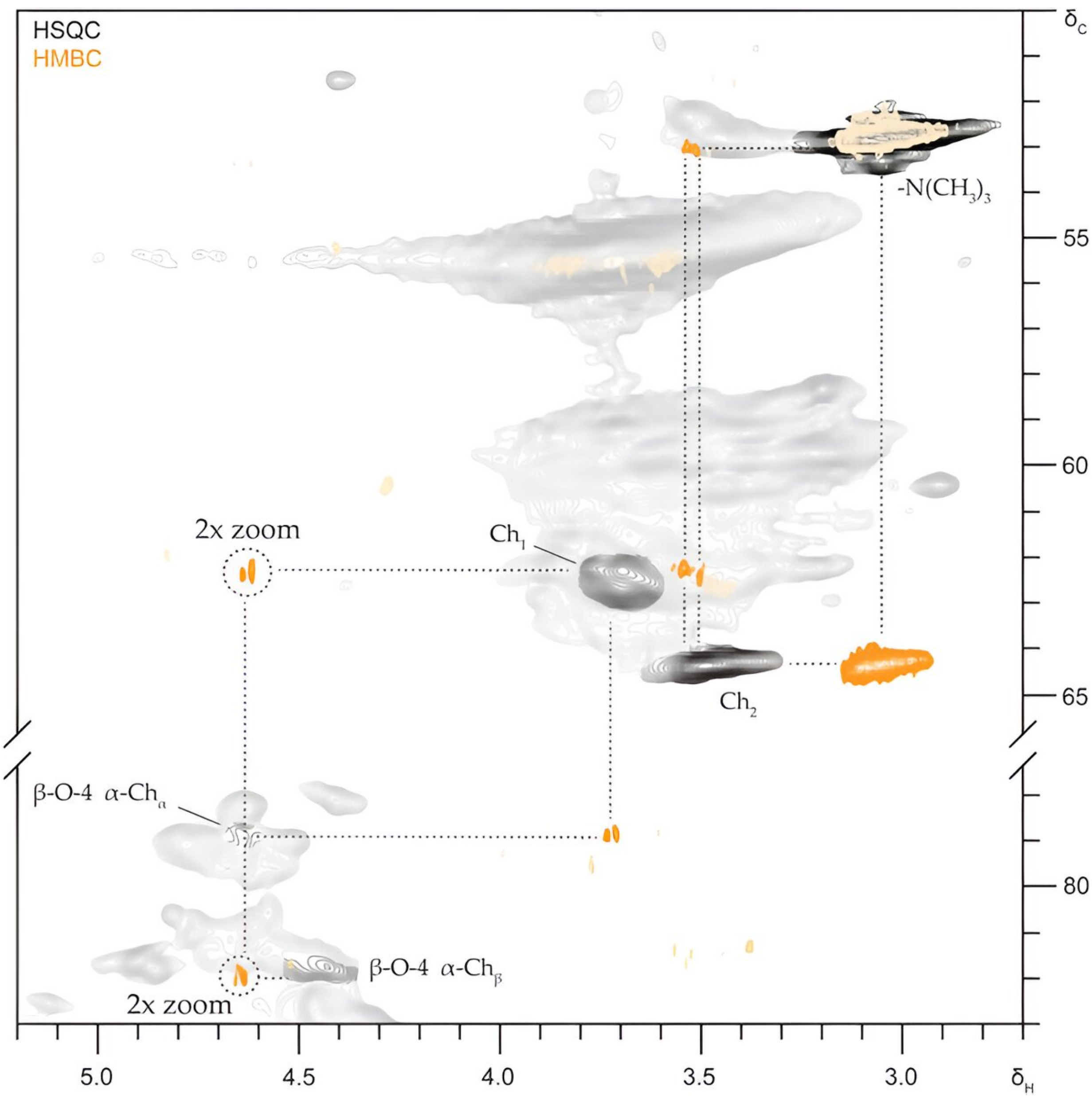

Selected regions of overlayed selective HSQC and HMBC NMR spectra (600 MHz, DMSO-d_6) of saponified DES lignin R9, with annotated HSQC signals in black and unassigned in gray, and annotated HMBC signals in orange and unassigned in light orange. Credit: *Green Chemistry* (2024). DOI: 10.1039/D4GC00909F

Lignin, the glue that holds fibers together in trees and plants, is one of the most common yet one of the most complex biocomposites, because there are so many variants and qualities. Its heterogeneous chemical structure makes it difficult to investigate its exact composition.

Using NMR spectroscopy, researchers from Wageningen University & Research (WUR) have successfully characterized, for the first time at molecular level, lignin extracted from Miscanthus (Silvergrass) with a green solvent.

This makes it possible to identify applications for which the material is suitable, thus bringing closer the total upgrading of the raw materials in a biorefinery process. The researchers have published their study in the journal *Green Chemistry*.

Residual stream

In industry, lignin is usually considered a residual stream that can mainly be used for energy and energy recovery. This is done, for example, through the Kraft pulp process in the paper industry. In this process, wood chips are traditionally cooked in a pressure vessel and treated with aggressive chemicals to extract cellulose: the fiber for paper.

"The quality of the lignin is so degraded by such an intensive process that it is difficult to come up with a valuable application for it," says Gijs van Erven, researcher Biorefinery and Sustainable Value Chains at Wageningen Food & Biobased Research.

"There is therefore growing interest in developing milder separation processes that do less damage to the lignin structure, making it suitable for high-value applications."

One of these mild processes is the use of Deep Eutectic Solvents (DES). These solvents are non-toxic and relatively easy to produce from (partly) biobased chemicals. The process takes place under milder conditions, especially in terms of temperature, which saves a lot of energy. In addition, the lignin remaining in this process is reactive, so of higher quality and usable for more high-value applications.

Balance

Van Erven states, "We looked at a mixture of lactic acid and choline chloride to separate Miscanthus. This northern European crop is a model for other grass-like crops, such as maize, straw and sugar cane.

"DES based on lactic acid and choline chloride is also applicable for hard and softwood, but this requires a higher temperature or a longer digestion time. This has an impact on the lignin structure you are eventually left with. We look for a balance between the amount of lignin we extract from the raw material, its purity and the intactness of its structure."

The starting point is to extract as much value as possible from raw materials. In this case, that was successful: Analyses using the NMR showed that some of the lactic acid and choline from the solvent binds covalently to the lignin. Demonstrating these chemical compounds at the molecular level was a challenge because of the heterogeneous structure of lignin—it is actually a complex mixture of diverse polymers.

New applications

"Wageningen University & Research has the analytical toolkit to tackle such an issue. We were thus able to prove that the structure in DES lignin samples is unique: lactic acid and choline are built in, so to speak. Therefore, the properties of this material are actually different from those of traditional lignins.

"This opens up new opportunities. For example, we can make better biocomposites and coatings from PLA (biobased plastic) mixed with DES lignin because they both contain lactic acid.

"The positive charge of choline also makes our lignin more suitable for aqueous applications, for example as a flocculant or surfactant for water processing. We see all kinds of directions where this lignin has new applications," says van Erven.

Follow-up project

A follow-up project with the exotic-sounding name AmphiphiLig is studying such lignin applications further. This involves working with industry to find modifications that can make lignin more applicable in both oily and aqueous environments. It also investigates how solvents can be recovered in the process.

"Further research on this and improving the recovery process is definitely worthwhile. I think our follow-up research will really boost the development of applications with lignin," claims van Erven.

More information: Gijs van Erven et al, Choline and lactic acid covalently incorporate into the lignin structure during deep eutectic solvent pulping, *Green Chemistry* (2024). DOI: 10.1039/D4GC00909F

Provided by Wageningen University

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