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Chemical derivatization techniques for lignin analysis



On January 30, 2020, Sudha Esakkimuthu defended her doctoral thesis of the University Grenoble Alpes, prepared under the supervision of the Professor Gérard Mortha, and of Nathalie Marlin, Associate Professor HDR (Grenoble INP-Pagora / LGP2).

She presented the results of her research work entitled *Study of new chemical derivatization techniques for lignin size exclusion chromatography characterization*.

Lignin is the second most abundant biopolymer on earth after cellulose and it consists of highly-branched, three dimensional aromatic structures with variety of functional groups.

This research work was to establish lignin derivatization methods for lignin analysis, to quantify functional groups and to determine lignin molar mass distribution (MMD) by size exclusion chromatography. Five technical lignin samples are considered: Protobind 1000, Organosolv, Pine Kraft, Eucalyptus Kraft and Indulin. They are derivatized through classical acetylation method and new methods such as fluorobenzoylation and fluorobenzoylation. The number of hydroxyl present in the lignin samples are quantified through GC and NMR (^1H , ^{13}C , ^{19}F and ^{31}P) techniques. The molar mass distribution of derivatized lignin samples are calculated using different SEC columns with different solvents (DMAc and THF). Conventional and universal calibration methods are used for MMD calculations.

With this approach, new derivatization methods significantly enhance lignin solubility in THF and improve chromatographic results. Universal calibration leads to about three times higher molar mass values than by conventional calibration.

Contacts

Gerard.Mortha@pagora.grenoble-inp.fr – Nathalie.Marlin@pagora.grenoble-inp.fr

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