COMPREHENSIVE ANALYTICAL CHARACTERISATION OF LIGNINS OBTAINED WITH DIFFERENT TECHNOLOGIES

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Lignin is the second most abundant organic compound on Earth. It has an amorphous, irregular three-dimensional, highly branched phenolic polymer structure. Because of its complex polymeric structure lignin characterisation is problematic.[1]

The most common process used in lignin production is the kraft process.[2] The obtained kraft lignin has low solubility in many organic solvents. For testing the lignin solubility, we tried different solvents such as water, DMSO, formic acid, acetic acid, ethanol, pyridine etc. In some solvents, lignin got dissolved after shaking with a vortex mixer or after keeping in an ultrasonic bath, whereas in some cases lignin dissolves after reflux with the solvent. The kraft lignin obtained from Sigma Aldrich has good solubility in water, DMSO, pyridine, water/acetonitrile mixture, formic acid and some other solvents.

The characterisation of kraft lignin was carried out using FTIR, ^{1}H NMR (400 MHz) and LCMS techniques. In ATR-FTIR lignin shows many peaks such as peak for O-H group, C-H bond, and for phenolic O-H and aliphatic C-H in methyl group and many more peaks. The lignin dissolved in a solvent which was used for ^{1}H NMR and reference purpose took ^{1}H NMR in DMSO d6. From the ^{1}H NMR analysis, we got information about the presence of acidic proton, aromatic protons, methoxy protons and aliphatic CH_2 groups. We observed some changes in lignin structure after refluxing with some solvents.

Lignin purification is a major issue. We used basic techniques for the purification of lignin such as precipitation and preparative TLC. kraft lignin dissolved in formic acid and added water drop by drop and lignin precipitate out. For lignin purification by prep TLC, we used different acetonitrile in water as eluents.

References

1. *Lu, Y. et al. Structural Characterization of Lignin and Its Degradation Products with Spectroscopic Methods. Journal of Spectroscopy 2017, 8951658.*
2. *Koumba-Yoya, G.; Stevanovic, T. New Biorefinery Strategy for High Purity Lignin Production. ChemistrySelect 2016, 1 (20), 6562–6570*

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