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LEWIS-ACID CATALYSED ORGANOSOLV LIGNIN EXTRACTION FROM WHEAT STRAW

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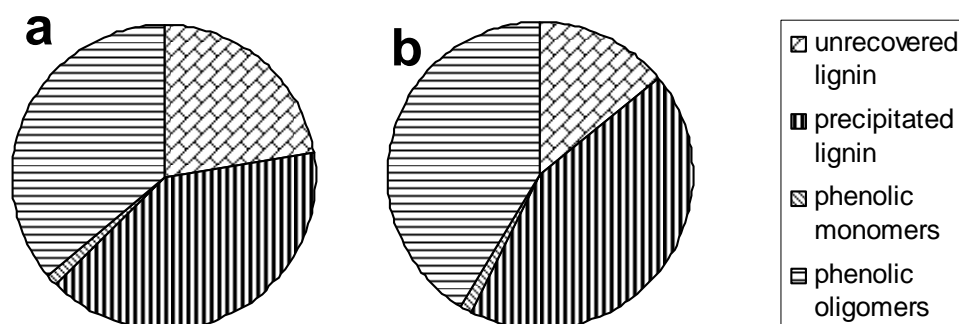
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The recovery of lignin-derived phenolic products by fractionation of lignocellulosic biomass is a promising way to replace high-value added oil-derived products by specialties issued from renewable resources. Catalytic control of the fractionation of biomass is the ideal tool to achieve the selectivities needed for economically sustainable processes.

In this project, Lewis acids have been studied as catalysts in the organosolv treatment of wheat straw. Fractionation of the lignocellulosic biomass and fragmentation of lignin have been performed in aqueous ethanol in the presence of FeCl₂, CuCl₂, FeCl₃, Ga(OTf)₃, ZrOCl₂ or Sc(OTf)₃ [1]. The lignins were characterised in terms of molecular weight, β-O-4 linkage content and chemical functions through size exclusion chromatography, thioacidolysis, ³¹P and ¹³C NMR and FT-IR spectroscopies.



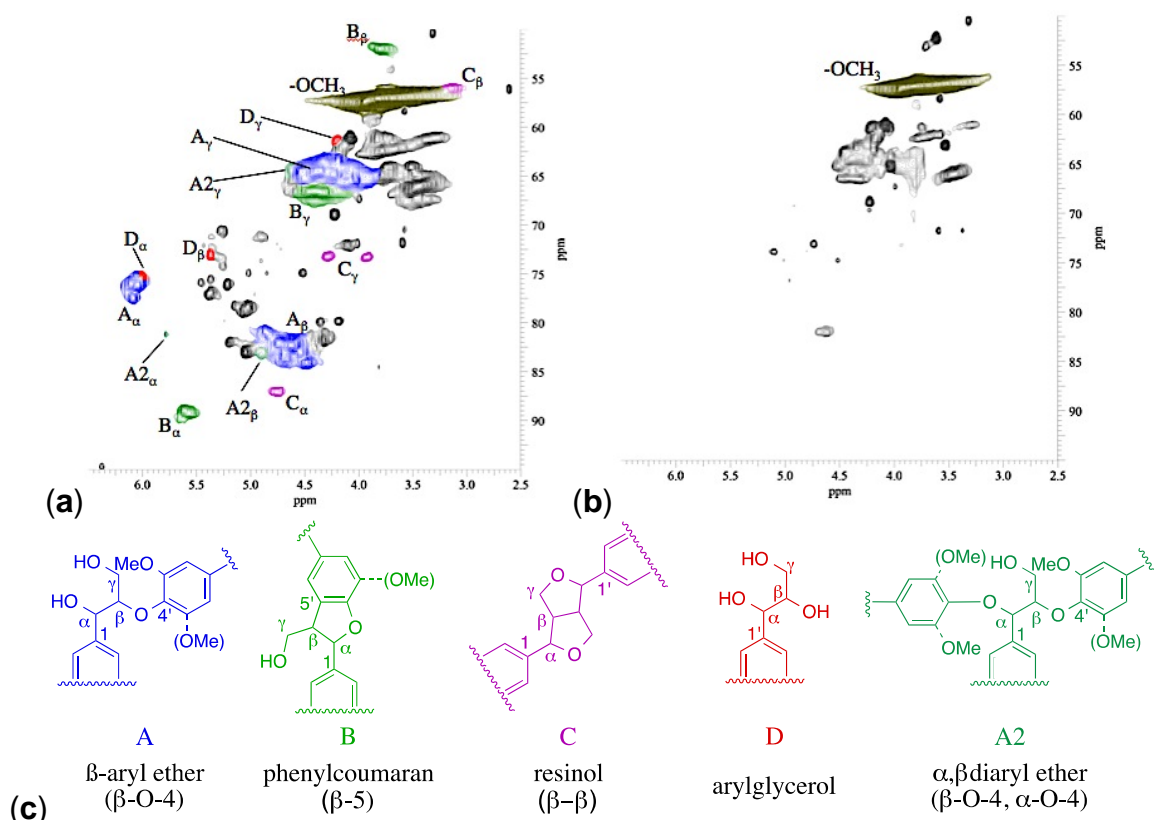
Selectivity of delignification of wheat straw at 160 °C in 66 % aqueous ethanol in the presence of 8 mmol/L Lewis acids with cation hardness (a) <math><10\text{ eV}</math> and (b) $>20\text{ eV}$

The degree of delignification and the yield of precipitated Klason lignin increased with the hardness of the Lewis acid. About half of the delignification products were

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water-soluble monomers and oligomers. Phenolic oligomers were targeted as intermediates in polymer chemistry.

The precipitated lignins were significantly different from the parent lignin in straw and their properties were tailored by the choice of the Lewis acid. The molecular mass, the amount of OH groups and of aliphatic C-O bonds decreased as the hardness of the acid cation increased. The hardest Lewis acids produced condensed, g-units enriched and aryl ether linkage-free lignins with molecular mass lower than 6 KDa. The control of the properties of lignin is especially appealing for the elaboration of additives for the textural control of suspensions.



^1H - ^{13}C HSQC NMR spectra (aliphatic region, $\delta_{\text{C}}/\delta_{\text{H}}$ 45-95/2.5-6.5, ppm) of (a) H_2SO_4 issued lignin and (b) $\text{Sc}(\text{OTf})_3$ issued lignin and (c) aryl ether linkages found herein

References

- [1] S. Constant, C. Basset, C. Dumas, F. Di Renzo, M. Robitzer, A. Barakat, F. Quignard, *Ind. Crop. Prod.* **2015**, 65, 180-189.

Acknowledgements

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