CHARACTERISATION OF ORGANOSOLV LIGNINS ISOLATED FROM DIFFERENT BIOMASSES

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Introduction

Lignin is a heterogenic phenylpropanoid biopolymer found in most terrestrial plants, which gives the plant its mechanical strength. The p-hydroxyphenyl unit (H), guaiacyl unit (G), and syringyl unit (S) are the building blocks when integrated into the lignin macromolecule (Fig. 1), there is a difference in the distribution of monolignol units depending on the type of plant biomass.¹

The purpose of this study was to optimize and implement analytical procedures for lignin analysis by size-exclusion chromatography (SEC) and Fourier transform



infrared spectroscopy (FTIR), as well as investigate the effect of the different solvent usage in Organosolv extraction method on lignin properties and structure. The pretreatment of lignocellulosic biomass with an organic solvent (Organosolv pulping) allows to isolate high-quality lignin with inherent structure, which is crucial for its further valorization. This method can also thrive with low input since the solvent is easily recovered by distillation.

> Figure 1. (a) Lignocellulosic biomass structure, (b) monolignol structural units of lignin.

Methods

A study was conducted on lignin extracted from three different biomass types: hardwood (aspen chips), softwood (pine sawdust), and grassy biomass (barley straw). From each biomass, the lignin was extracted with usage of ethanol or 1,4-dioxane as the solvent and analysed using SEC and FTIR methods (Fig. 2). The absorption bands corresponding to bond vibrations in FTIR spectra were assigned based on previously reported data ^{2,3}.



The usage of different solvents in the extraction showed variability in the yield (Table 1) and of the color of the lignin. Dioxane extraction resulted in 2-3 times higher yield and darker color of lignin. Lignins extracted with ethanol were of lighter color, where pine wood lignin had a pinkish hue.

The results from SEC (Table 1) showed, that lignin extracted with ethanol has slightly higher molecular weight (avg. M_n≈2300 g/mol, M_w≈3400 g/mol), compared to lignin extracted from the same biomass using dioxane as the extraction solvent (avg. $M_n \approx 1900$ g/mol, $M_w \approx 2800$ g/mol). The highest and lowest molecular weights belonged to the aspen ethanol lignin and barley straw dioxane lignin, respectively. The polydispersity indices ranged from 1.37 to 1.76.

The FTIR spectral data and semi-quantitative monolignol content are presented in Fig. 3 and Fig. 4, respectively.

Table 1. Lignin yield and molecular weight distribution results.

	Biomass	*Extraction solvent	Lignin yield per biomass [%]	**Lignin yield per lignin content [%]	M _N [g/mol]	M _w [g/mol]	PI
	Aspen	E	4.6	17.0	2601±27	3743±43	1.44
		D	13.6	50.4	1925±6	2670±32	1.38
	Pine	E	7.0	21.2	2343±27	3391±28	1.45
		D	20.4	61.8	1959±24	3450±5	1.76
	Barley straw	E	5.9	28.1	2187±15	3211±29	1.47
		D	11.7	55.7	1809±16	2469±23	1.37

Figure 3. Superimposed FTIR spectra of extracted lignins.



* E – ethanol, D – 1,4-dioxane

**Total lignin content obtained from previous study in our group ⁴.

Conclusions

Figure 4. Semi-quantitative monolignol content ratio in extracted lignins, E – ethanol, D – 1,4-dioxane, S – syringyl unit, G – guaiacyl unit.

Based on current work, it can be concluded that the choice of the biomass feedstock and extraction solvent influence molecular weight distribution of lignins, as well as on the content of monolignols. For further implementation and valorisation of lignin, the optimal biomass and extraction procedure can be selected based on this study.

References

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