Application Brief

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Characterization of Lignin Sulfonate Using GPC/SEC

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Abstract

The GPC/SEC characterization of lignosulfonates in 0.1 M sodium hydroxide solution with Agilent MCX columns as stationary phase is described. The separation of a lignin-based sample on two different MCX 5 µm column sets is compared.

Introduction

Lignin is one of the most abundant natural polymers. It cannot be described with an exact chemical structure, but rather as a class of complex polymers consisting of highly branched phenolic moieties. Lignin is found in the cell walls of most plants and is produced as a by-product in the paper industry on a million tons scale. It is water soluble as an alkaline salt. In recent years, lignin and materials derived from lignin have received increasing interest as renewable raw materials.[1] Lignosulfonic acids are used in several concrete applications as surfactant, while lignosulfonates are the base of many glues and textile additives.

Experimental

See Table 1.

Table 1. Instrument and sample conditions.

	Conditions
Pump	Isocratic pump Flow rate: 1.0 mL/min Mobile phase: 0.1 M sodium hydroxide
Injection System	Autosampler Injection volume: 50 µL
Columns	MCX low MW combination: MCX 5 µm precolumn, 8 × 50 mm (p/n MCA080505) MCX 5 µm 1,000 Å, 8 × 300 mm (p/n MCA0830051e3) MCX 5 µm 1,000 Å, 8 × 300 mm (p/n MCA0830051e3) MCX 5 µm 1,000 Å, 8 × 300 mm (p/n MCA0830051e3) MCX medium MW HR combination: MCX 5 µm precolumn, 8 × 50 mm (p/n MCA080505) MCX 5 µm 1,000 Å, 8 × 300 mm (p/n MCA0830051e3) MCX 5 µm 100,000 Å, 8 × 300 mm (p/n MCA0830051e5)
Temperature	23 °C
Sample Concentration	2 mg/mL
Detectors	Variable wavelength UV-Vis detector (VWD) at λ = 254 nm
Software	Agilent WinGPC

Results and discussion

Lignin-based samples often contain sulfonic acid groups, converted into sodium salts when using 0.1 M sodium hydroxide solution as mobile phase. For highly negatively charged macromolecules, MCX columns typically enable efficient GPC/SEC separation.

Figure 1 illustrates GPC/SEC measurements of a lignin-based sample on two different MCX 5 µm column sets.

The MCX low MW combination, composed of three MCX 5 µm 1,000 Å columns in combination with an MCX 5 µm guard column, is designed for high-resolution GPC/SEC separations of low molar mass polyanions. Using this column set for the separation of the lignin-based sample results in good resolution in the oligomer region.

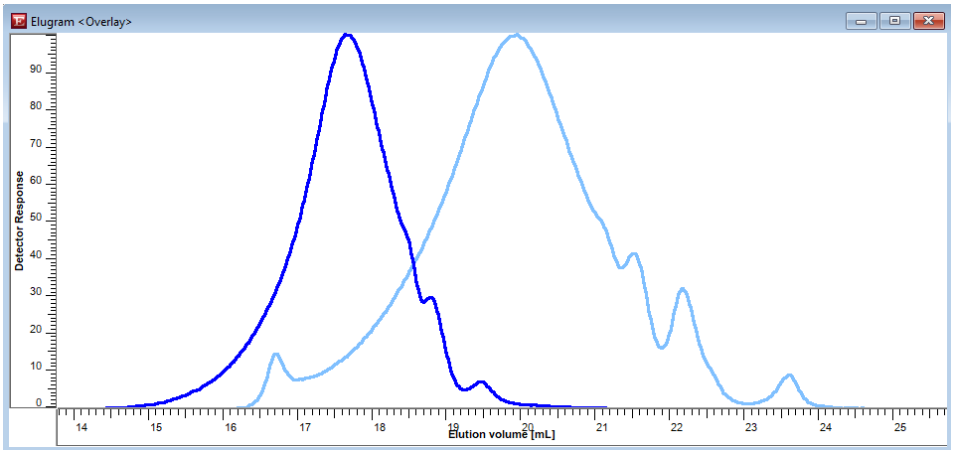

Figure 1. Overlay of a lignin-based sample (UV at 254 nm trace, normalized detector response): MCX low MW combination (light blue line), MCX medium MW HR combination (dark blue line).

However, a small amount of the sample elutes at the exclusion limit as indicated by the small hump at about 16.5 mL elution volume in the light blue chromatogram. Similar peaks were observed for a variety of lignin samples, indicating that the exclusion limit of the MCX 5 µm 1,000 Å column is slightly too low to separate the complete molar mass range of a variety of lignin samples.

Using a column set with a higher separation range (MCX medium MW HR combination) based on an MCX 5 µm guard column in combination with an MCX 5 µm 1,000 Å and an MCX 5 µm 100,000 Å column, the lignin-based sample is fully separated without a shoulder in the lower elution volume region, while some oligomer separation is still visible.

Conclusion

GPC/SEC measurements of a lignin-based sample were achieved by use of Agilent MCX columns as stationary phase and diluted sodium hydroxide solution.

References

1. Calvo-Flores, F. G.; Dobado, J. A. Lignin as Renewable Raw Material. *Chem. Sus. Chem.* **2010**, *3*, 1227–1235.

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