Analysis of Lignins via GPC Viscometry using the Agilent 390-MDS

Application Note

Author

Graham Cleaver
Agilent Technologies, Inc.

Introduction

Lignins impart rigidity to woody plants, achieved by cross-linking different plant polysaccharides that strengthen the cell walls, and are the second most abundant organic polymers on earth. The name itself is derived from the Latin word lignum, meaning wood. Lignins are important in the papermaking process – it is lignin in the pulps used to make newspapers which causes them to yellow with age and it must be removed to produce high quality bleached paper. The base monomers are phenylpropanoids, aromatics with carboxylic acid and hydroxyl functionalities, which form highly complex, amorphous, three-dimensional polymers. It is also used as a raw material for several chemicals, often via lignosulfonates.

Determining the molecular weight distribution and structural properties of lignins is an important step in understanding their behavior in wood pulps. However, the analysis is very challenging, as they are very difficult to extract from wood pulps and often degrade during this process. There is a huge variation in the structure of lignins, with the material showing great heterogeneity from one source to the next.

The accurate molecular weight distributions of four samples of lignin were investigated by GPC with viscometry.

Agilent Technologies

Methods and Materials

Conditions

Columns:	2 x Agilent PolarGel-M, 300 x 7.5mm (part number PL1117-6800)
Eluent:	Dimethylformamide (0.1% LiBr)
Injection Volume:	200 µL
Flow Rate:	1.0 mL/min
Sample Concentration:	2.0 mg/mL
Calibration Standards:	Agilent PMMA EasiVial
Detector Train:	390-MDS incorporating Viscometer and DRI

Results and Discussion

Figure 1 compares responses from refractive index and viscometer detectors obtained from GPC of a lignin sample. Molecular weight distributions of four lignin samples are shown in Figure 2. Figure 3 shows overlaid Mark-Houwink plots for the samples. It is apparent from the figures that the four lignins varied in structure, associated with the different sources from which they were derived.

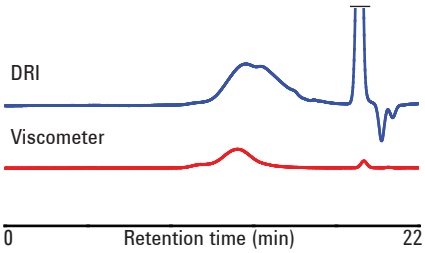

Figure 1. Chromatograms for a lignin sample produced by different detectors

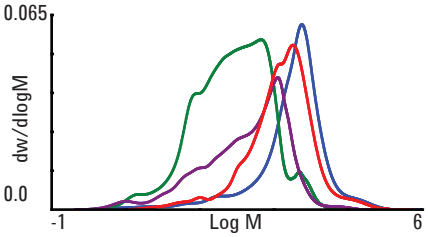

Figure 2. Overlaid molecular weight distributions (MWD) for four lignin samples. The marked differences in MWD suggest the samples were from different sources

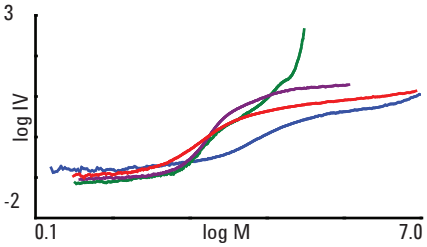

Figure 3. Overlaid Mark-Houwink plots for the four lignin samples. Differences in the traces suggest the lignins possess different structures

Conclusion

The results show that a PolarGel-M column set in conjunction with a 390-MDS detector system comprising a differential refractive index detector and a viscometer can measure the molecular weight distributions of lignin samples by the Universal Calibration approach. The information obtained by employing a viscometer highlighted structural and/or chemical differences between the lignins.

www.agilent.com/chem

Published in UK, April 30, 2015
5991-5829EN

Agilent Technologies