Gel Permeation Chromatography with Viscometry for the Molecular Weight Characterization of Epoxy Resins

Application Note

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Introduction

Synthetic resins are a polymeric class of materials designed to mimic the properties of naturally occuring resin materials. Typically viscous liquids that are capable of hardening, they are routinely synthesized via esterification reactions or soaping of organic compounds. First developed in the 1930s and commercialized in the 1940s, epoxy resins are manufactured through the reaction of polyols with epichlorohydrin, such as the largest production epoxy resin, called diglycidyl ether of bisphenol-A (DGEBA). The molecules of epoxy resins are low in molecular weight until cured, when they form a network of large cross-linked structures. Epoxy resins exhibit strong heat and chemical resistance as well as high mechanical strength and therefore find use in many different application areas from adhesives to high performance sports equipment.

The molecular weight and composition of the pre-cured materials is important as these parameters control how the material will cure and the properties of the final product. In their non-cured state epoxy resins may be analyzed by gel permeation chromatography (GPC). Determining the molecular weight of these complex materials is not straightforward as the composition of the material affects the molecular dimensions, which in turn means that molecular weights determined by conventional GPC using only a refractive index detector are inaccurate. However, using viscometry, it is possible to determine the molecular weights of these materials by the universal calibration approach. This note describes the use of GPC with viscometry to determine the molecular weight profiles of two different epoxy resin materials. Employing the universal calibration method, it is possible to determine accurate molecular weights for the materials regardless of composition, and also probe their structure via Mark-Houwink plots.

Verified for Agilent
1260 Infinity
GPC/SEC System

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Materials and Methods

Conditions

Columns:	2 x Agilent PLgel 5 µm MIXED-D, 300 x 7.5 mm (part number PL1110-6504)
Eluent:	Tetrahydrofuran
Flow Rate:	1 mL/min
Inj. Vol:	100 µL
Sample Conc:	2 mg/mL
Temp:	40 °C
Detectors:	Agilent 390-MDS Multi Detector Suite comprising a refractive index detector and a four capillary bridge viscometer
Calibration Standards:	Agilent Polystyrene EasiVials

Results and Discussion

Figure 1 shows overlaid dual detector raw data chromatograms for an epoxy resin sample showing the data collected from the individual detectors. Each polymer eluted as a broad peak.

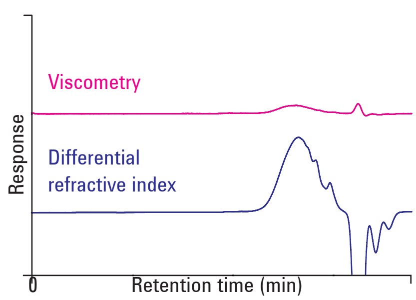

Figure 1. Overlaid dual detector raw data chromatograms for an epoxy resin sample

The samples were all then analyzed by GPC with viscometry, employing the universal calibration method to determine molecular weights that were independent of calibrant chemistry. The overlaid molecular weight distributions are shown in Figure 2.

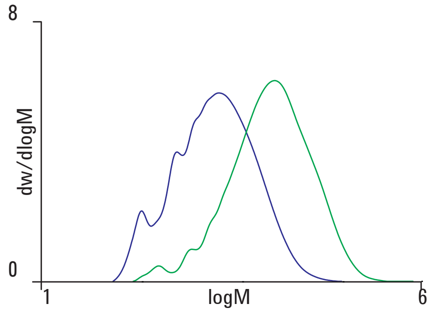

Figure 2. Overlaid molecular weight distributions calculated by universal calibration analysis of the two samples

The Mark-Houwink plots for the two materials are shown in Figure 3.

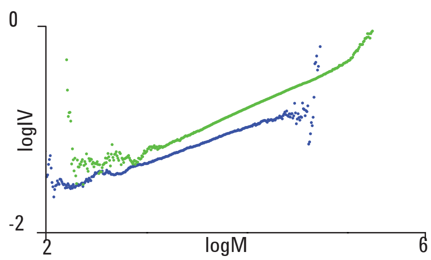

Figure 3. Overlaid Mark-Houwink plots calculated from universal calibration analysis of both samples

Conclusion

The structure of some epoxy resins was elucidated by using gel permeation chromatography with the 390-MDS. The 390-MDS detected differences in the chemistry of the resins, demonstrating the effectiveness of the instrument when investigating the molecular weight and structural properties of polymers.

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