Analysis of Epoxy Resins by GPC Viscometry using the Agilent 390-MDS Multi Detector Suite

Application Note

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Introduction

Epoxy resins are complex materials manufactured by the reaction of a 'resin' with a 'hardener'. This produces a cross-linked polymer that is extremely strong, tough and may be used to bond materials together. The resin component of the mixture is an epoxide pre-polymer, typically produced by the reaction of an epoxide with bisphenol-A. The resulting short-chain material contains pendant –OH and epoxide functionalities and is polar in nature. The molecular weight of the epoxide chains in the resin is important as this influences the curing rate of the final epoxy resin formulation when mixed with the hardener, however, accurate molecular weights are difficult to obtain due to the complex, inhomogeneous nature of the epoxide polymer chains.

Gel permeation chromatography (GPC) is a well-known technique for assessing the molecular weight distribution of polymers, a property that influences many physical characteristics such as the curing time of epoxy resins. GPC viscometry employing a viscometer in combination with a differential refractive index detector has the advantage of allowing the determination of accurate molecular weights for structurally complex polymers and co-polymers via the Universal Calibration approach. Using this methodology, the same molecular weights are determined for samples regardless of the standards used to create the calibration. This application note describes the analysis of two samples of epoxide pre-polymers by GPC viscometry.

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Methods and Materials

Conditions

Samples:	Epoxide pre-polymers
Columns:	2 x Agilent PolarGel-L, 300 x 7.5 mm (p/n PL1117-6830)
Injection Volume:	200 µL
Eluent:	Dimethyl formamide + 0.1% LiBr
Flow Rate:	1.0 mL/min
Detector Train:	390-MDS incorporating Viscometer and DRI
Detector Temp:	All detectors set at 60 °C

Epoxide pre-polymers are polar in nature and can be run in polar organic solvents such as dimethyl formamide using suitable columns such as a set of PolarGel-L columns. The 390-MDS was chosen as part of the system as it is capable of multi-detector GPC in polar solvents.

Results and Discussion

Figure 1 shows an example overlaid multi-detector chromatogram for the sample of one of the epoxide pre-polymers. The appearance of oligomers resolved by the PolarGel-L columns is clearly apparent.

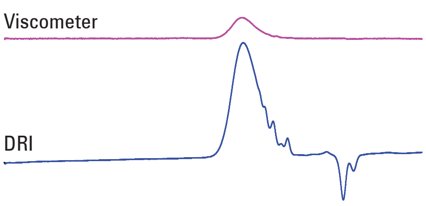

Figure 1. Overlaid multi-detector chromatogram for an example epoxide pre-polymer

Figure 2 shows an overlay of the accurate molecular weight distributions of the two samples under investigation. As can be seen, they have very different distributions indicating that the materials are two different grades of epoxide pre-polymer that will display differing hardening rates when mixed with the hardener component of the epoxy resin mixture.

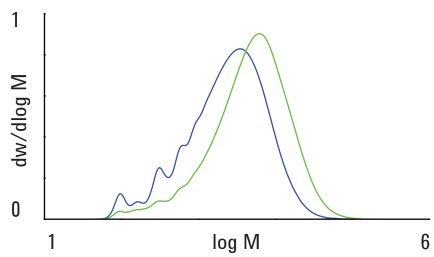

Figure 2. Overlaid multi detector molecular weight distributions of two samples of epoxide pre-polymer

Figure 3 shows the overlaid Mark-Houwink plot of log intrinsic viscosity as a function of log molecular weight for the two samples. Both grades of epoxide pre-polymer display a similar relationship only showing deviation at high molecular weight. This would indicate that the molecular dimensions of the two materials are approximately the same at low molecular weight, however, as the molecular weight increases there is a structural disparity between the two materials, most likely due to a change in the chemistry of the samples.

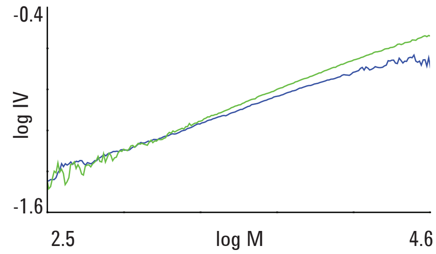

Figure 3. Overlaid Mark-Houwink plots for the two samples of epoxide pre-polymer

Conclusion

The structure of some epoxide pre-polymers was determined using gel permeation chromatography with the Varian 390-MDS Multi Detector Suite. Mark-Houwink plots provided by the 390-MDS revealed 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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