Polybutylene Terephthalate Analysis by Agilent PL HFIPgel and GPC

Application Note

Materials Testing and Research, Polymers

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Introduction

Polybutylene terephthalate (PBT) resins are used in a wide variety of applications in which toughness and resistance to damage are highly advantageous. Mechanical and thermal stress during the production of molded parts can cause degradation, giving a reduction in desirable physical properties. The molecular weight distribution of the resin is a key measure of the onset of degradation and estimating the mechanical strength of the final product. Molecular weight distributions of polymers such as PBT are determined by gel permeation chromatography.

Analysis of Polybutylene Terephthalate

PBT is soluble in 1,1,1,3,3,3-hexafluoroisopropanol (HFIP), a polar organic solvent, which is excellent for dissolving polar polymers such as polyamides and polyesters. The analysis was carried out in HFIP modified by the addition of 20 mM sodium trifluoroacetate to prevent aggregation. Two Agilent PL HFIPgel columns, designed specifically for HFIP applications, were employed for the analysis at a temperature of 40 °C. The Agilent PL-GPC 220 integrated chromatograph was used with differential refractive index and viscometry detection.

GPC, coupled with a molecular weight sensitive viscometer, allowed calculation of molecular weights based on hydrodynamic volume using the Universal Calibration approach, leading to molecular weights independent of the standards used to generate the column calibration. Agilent polymethylmethacrylate standards were employed for the Universal Calibration.

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Table 1 shows the molecular weight averages and intrinsic viscosity for the Valox PBT sample before and after molding as determined by GPC/viscometry. Clearly, the molecular weight distribution indicates that after molding the material has suffered from degradation and is less robust than the virgin material.

Table 1. Molecular Weight Averages and Intrinsic Viscosity of a Polybutylene Terephthalate Resin Before and After Molding

	Mn/g mol^{-1}	Mw/g mol^{-1}	Intrinsic viscosity/g^{-1}
Virgin resin	24,400	48,600	0.535
Molded part	11,200	24,000	0.306

Conditions

Samples	Polybutylene terephthalate resin
Columns	2 × Agilent PL HFIPgel, 300 × 7.5 mm (p/n PL1114-6900HFIP)
Eluent	HFIP + 20 mM NaTFA
Flow Rate	1.0 mL/min
Inj Vol	200 µL
Temp	40 °C
Detectors	Agilent PL-GPC 220 (RI), Viscometer

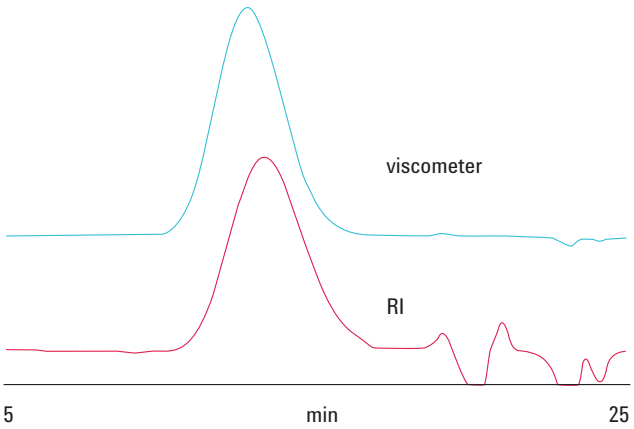

Figure 1. An example overlay of a dual-detector chromatogram of the virgin polybutylene terephthalate resin before molding.

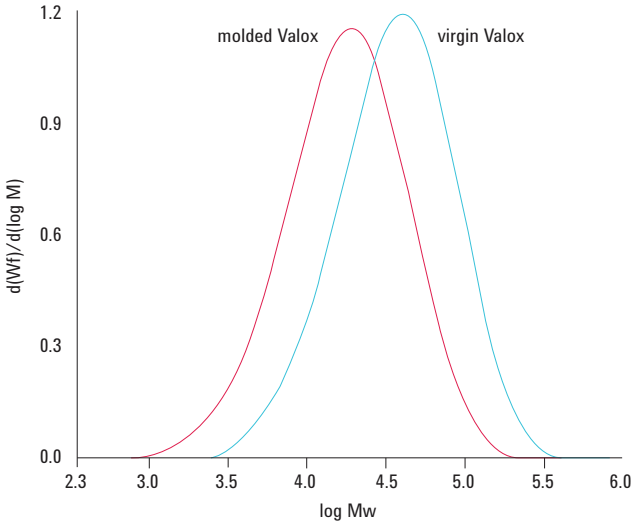

Figure 2. The resulting molecular weight distributions for polybutylene terephthalate.

Conclusions

A sample of polybutylene terephthalate resin was successfully analyzed on an Agilent PL HFIPgel two-column set, revealing degradation of the PBT after a molding process. These columns use a novel dispersion-polymerization process, giving near-uniform bead size and characteristics. This technology avoids the excessive calibration curvature, dislocations, and poor low molecular weight resolution associated with conventional columns that use styrene/divinyl benzene when using HFIP as solvent.

For More Information

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