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SEC Analysis of Polylactic Acid Using Advanced Polymer Chromatography

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This is an Application Brief and does not contain a detailed Experimental section.

Abstract

This application brief demonstrates the ability of Advanced Polymer Chromatography (APC) using ACQUITY APC XT Columns to characterize the molecular weight distribution of polylactic acid.

Benefits

Size exclusion chromatography method development for polylactic acid polymers can be accomplished quickly using Advanced Polymer Chromatography.

Introduction

Polylactic acid (PLA) polymers are very useful in many finished products such as landscaping films, medical therapeutics, and food packaging. The popularity of this polymer stems from its versatility, renewable sources and biodegradability. The PLA degree of crystallinity and molecular weight play important roles is its versatility.[1] Crystallinity can affect the polymer solubility, and the polymer must be solubilized for molecular weight determination by size exclusion chromatography (SEC).

These same PLA characteristics can require a co-solvent for complete dissolution of the polymer, and to prevent secondary interactions. A fully dissolved polymer is necessary for characterization by SEC, and true SEC requires no interaction of the polymer with the stationary phase or itself.

The choice for high throughput SEC with a flexible solvent column technology is the Waters ACQUITY APC System.

Experimental

LC conditions

System:	ACQUITY APC
Flow rate:	0.6 mL/min
Mobile phase:	Chloroform (amylene stabilized) with THF (unstabilized): (70/30)
Sample conc.:	2 mg/mL
Sample temp.:	20 °C
Injection vol.:	20 µL
Column temp.:	55 °C
Columns:	ACQUITY APC XT 900Å, 450Å, 125Å, 45Å (4.6 x 150 mm) (P/N 186007254, 186007010, 186007000, 186006995)
Detector:	RI (55 °C)
Data analysis:	Empowe 3 Chromatography Data Software

Results and Discussion

The analysis of PLA was completed using the conditions listed; PLA was dissolved in the mobile phase and analyzed with the APC System and ACQUITY APC XT Columns. Various levels of unstabilized tetrahydrofuran (THF) were added to the amylene stabilized chloroform, until the ideal solvent ratio yielded a complete solution of the PLA and no interaction with the column. The robust BEH column technology and APC solvent flexibility enabled optimizing the mobile phase quickly compared to traditional SEC.

The relative calibration curve in Figure 1 was created using narrow molecular weight polystyrene standards from the ACQUITY APC Polystyrene High MW Calibration Kit (P/N 186007541), and the highest molecular weight standards were excluded as outside the area of interest. The PLA molecular weight values in Table 1, chromatogram (Figure 2) and distribution curve (Figure 3) were obtained through Empower 3 Software with the GPC/SEC option. Table 1 reflects the values in Figure 3. These SEC molecular weight values were key points of interest for polymer analysis: Mn for number average molecular weight, Mw for weight average molecular weight, MP for peak molecular weight, Mz for Z-average molecular weight, and Polydispersity for the ratio of Mw/Mn. The PLA polydispersity index (PDI) value of 2.23 is evidence of a broad molecular weight distribution. A typical polymer PDI is 1 to 3: three being much dispersed and one being mono dispersed.

Sample (Da)	
Mn	6300
Mw	139000
MP	114000
Mz	250000
Polydispersity (Mw/Mn)	2.20

Table 1. Molecular weight values for PLA sample.

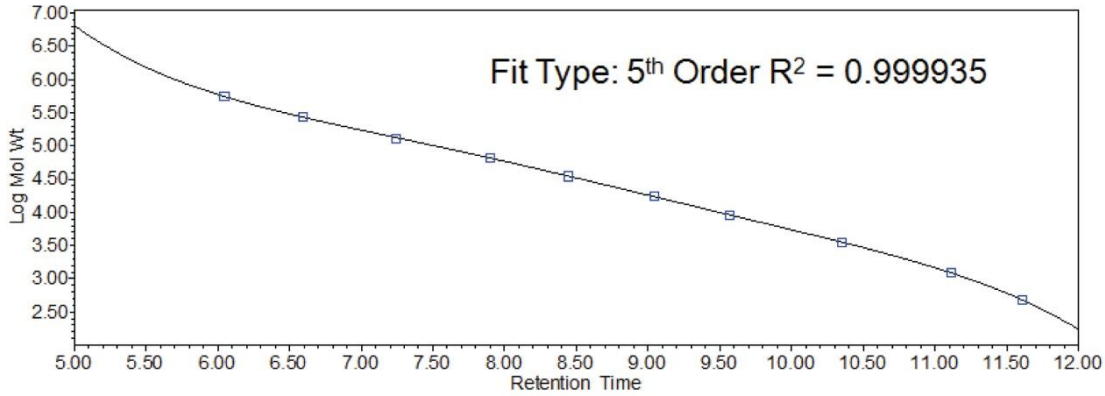

Figure 1. Polystyrene standard calibration curve: 2.3 M and 1.07 M Dalton points excluded.

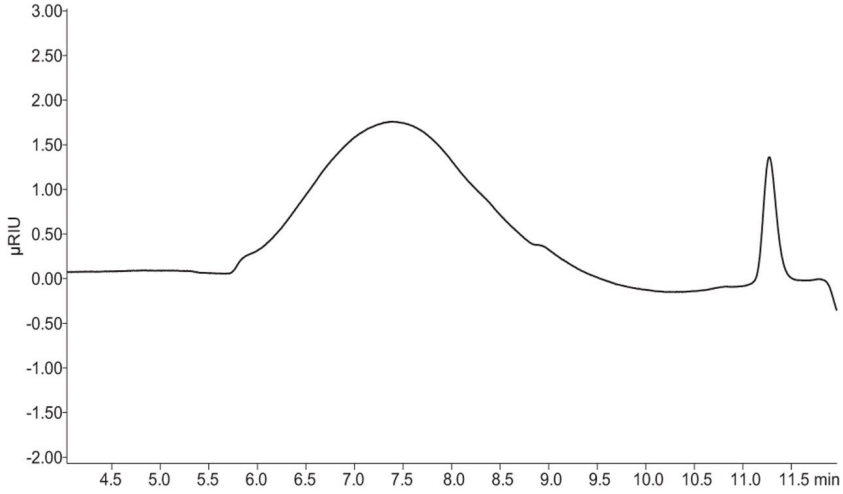

Figure 2. Chromatogram of PLA.

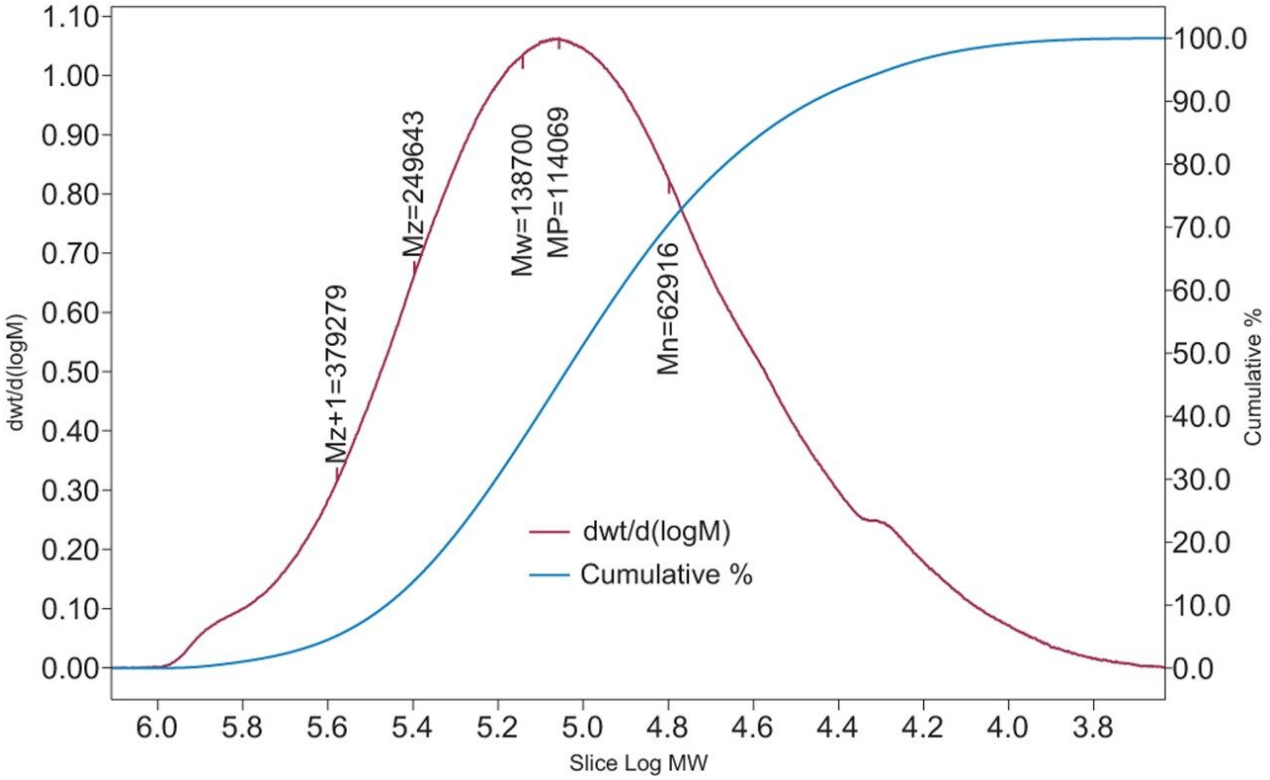

Figure 3. Molecular weight distribution of PLA.

Conclusion

APC was able to chromatographically resolve the separation of PLA by optimizing the solvent blend. The solvent flexibility of the APC BEH columns enabled short equilibration times between solvent blend selections. The APC optional solvent select valve enabled up to six bottles of mobile phase solvent blends for method optimization.

For absolute molecular weight or conformational information, advanced detectors are necessary. The latest low dispersion detectors are now compatible with the ACQUITY APC System as highlighted by Malvern and Wyatt.[2, 3]

References

1. Rafael Auras; Loong-Tak Lim; Susan E. M. Selke; Hideto Tsuji (eds.). Poly (Lactic Acid): Synthesis, Structures, Properties, Processing, and Applications. doi:10.1002/9780470649848. ISBN 9780470293669.
2. “Using Multi-Detector Advanced Polymer Chromatography (APC) to Better Differentiate Samples by Structure”, Malvern Panalytical Limited, 2018, https://www.malvernpanalytical.com/en/learn/knowledge-center/application-notes/AN180606MultidetectorAPCSampleStructure.html.
3. Trainoff, Stephen; Deng, Chris; “Development of a New Online Micro-Viscometer for APC and UHPLC Applications”, Wyatt Technology Corporation, 19 June 2018, http://www.wyatt.com/files/literature/app-notes/sec-mals-proteins/absolute-molar-mass-uHPLC.pdf.

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