

Analysis of Nylon 6 by Semi-micro Scale GPC

Introduction

For the room temperature analysis of Nylon (a polyamide synthetic fiber), sample preparation is made by dissolving samples in hexafluoroisopropanol (HFIP). However, a recent cost increase in HFIP has made this analysis more expensive and HFIP is considered environmentally unfriendly. The use of semi-micro separations has a significant advantage for this type of application by reducing the consumption of mobile phase.

To reduce analysis time and solvent consumption, smaller column sizes for rapid analysis are now available for use with GPC analysis.

In this report, the measurement of Nylon 6 films and fibers was performed using the LC-4000 Series RHPLC system with RI-4035 refractive index detector designed for use with semi-micro GPC columns offering high performance analysis. The solvent used is approximately 8 mL per analysis cycle and represents a 75% solvent saving compared with conventional analysis. The average molecular weight was calculated using ChromNAV-GPC, and a molecular weight calibration curve was constructed using a polymethylmethacrylate (PMMA) standard.

Keyword: Nylon 6, HFIP, semi-micro scale, PMMA, GPC, Refractive index detector, Molecular weight distribution program ChromNAV-GPC

Experimental

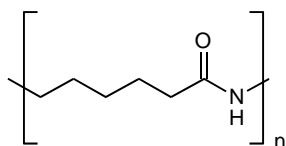
[Equipment]

Pump: PU-4185
 Autosampler: AS-4150
 (100 μ L Sample Loop)
 Column oven: CO-4060
 Detector: RI-4035

[Conditions]

Column: 2x Shodex LF-404 (4.6mmI.D. x 250mmL, 6 μ m each)^{*1}
Eluent: 5 mM Sodium trifluoroacetate in HFIP^{*2}
Flow rate: 0.2 mL/min
Column temp.: 40°C
Injection volume: 20 μ L
 Calibration standards for GPC:

[Structure]



Nylon 6

Sample:

PMMA standards mixture (0.025 (w/v)% each in eluent)
 PMMA STD (1); Mp 1520000, 211000, 21700, 2200
 PMMA STD (2); Mp 772000, 51900, 6900, 645

0.1 (w/v)% Nylon 6 film and fiber in eluent

*1 The column dedicated for use with THF was replaced by HFIP.

*2 Degassing was performed by the batch, not online vacuum degasser.

Results

Fig.1 Chromatograms of PMMA standard.

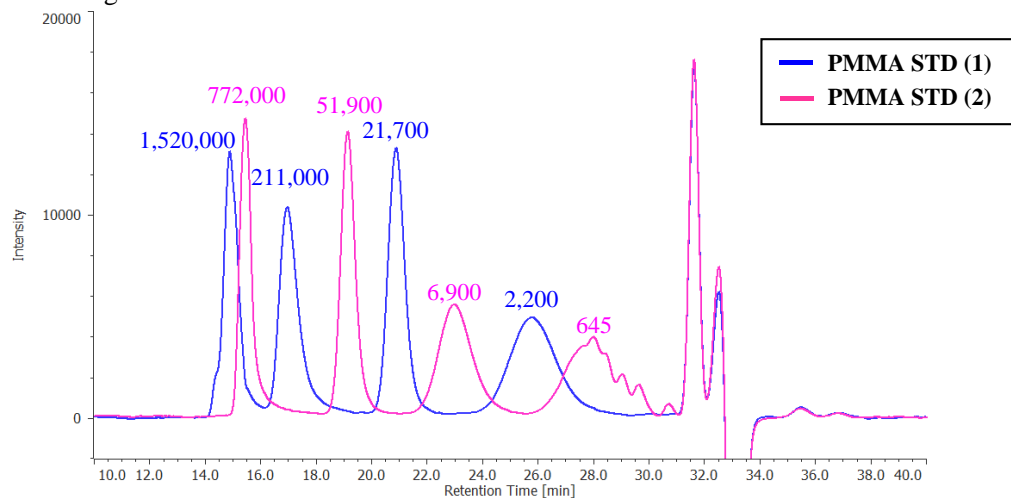


Fig. 1 Chromatogram of PMMA standard

(The values on the chromatogram indicate the Mp, peak top molecular weight, for each PMMA standard)

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Fig. 2 shows the molecular weight calibration curve using PMMA standard. Fig. 3 displays the chromatogram of Nylon 6 film and molecular weight calibration curve, and its molecular weight distribution curve is in Fig. 4. Fig. 5 is the chromatogram of Nylon 6 fiber and molecular weight calibration curve, and its molecular weight distribution curve is in Fig. 6. Table 1 shows the result of each average molecular weight calculation converted to PMMA.

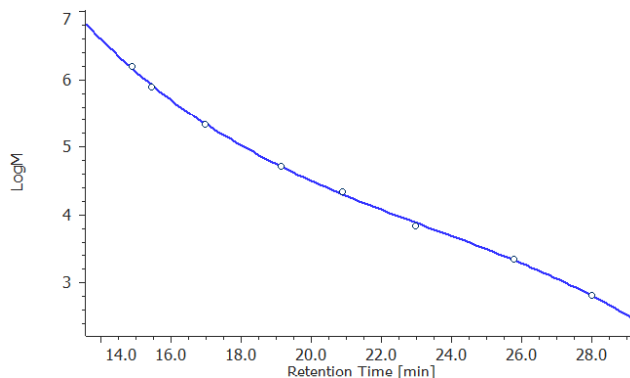


Fig. 2 Molecular weight calibration curve by the PMMA standard.

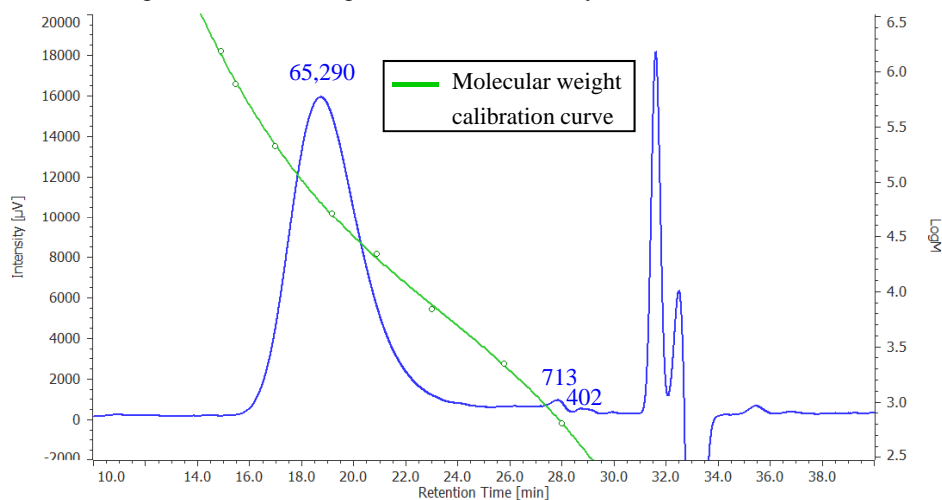


Fig. 3 Chromatogram of Nylon 6 film and molecular weight calibration curve
(The values indicate each M_p converted by PMMA)

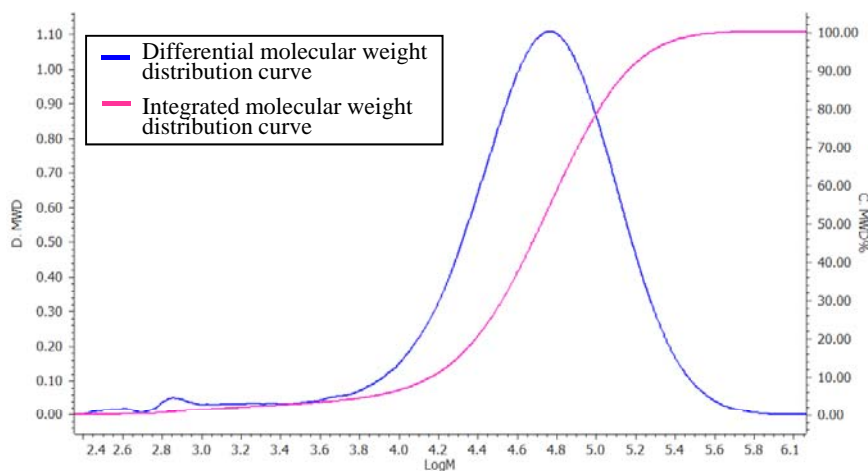


Fig. 4 Molecular weight distribution curve of Nylon 6 film

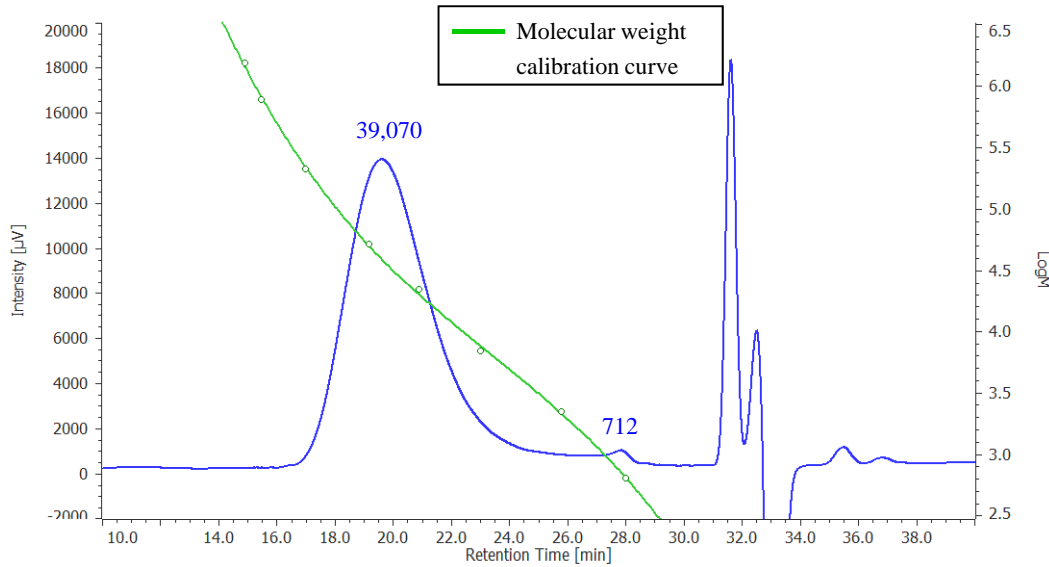


Fig. 5 Chromatogram of Nylon 6 fiber and molecular weight calibration curve
(The integrated indicate each Mp converted by PMMA)

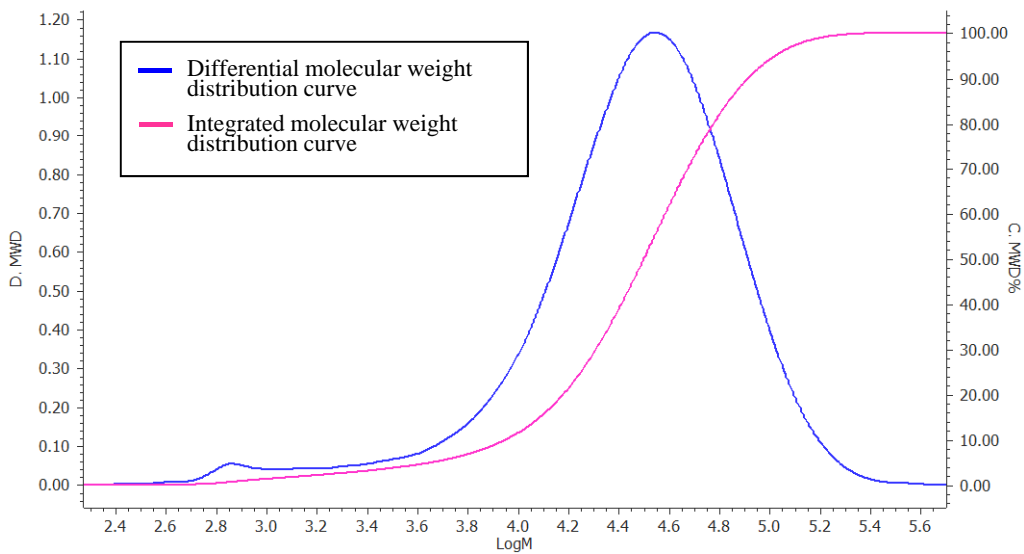


Fig. 6 Molecular weight distribution curve of Nylon 6 fiber

Table 1 Average molecular weight calculation of Nylon 6 film and fiber converted by PMMA

	Mn	Mw	Mz	Mv	Mw/Mn	Mz/Mw
Film	16570	70258	130381	70258	4.24	1.86
Fiber	12854	40210	69148	40210	3.13	1.72



Application Note

310013G

P/N	Description
7003-J014A	PU-4185 RHPLC Semi-micro Pump (Base Unit)
7064-J002A	AS-4150 RHPLC Autosampler
0507-0937	7755-024 Sample loop, 100 μ L
7021-J002A	CO-4060 Column Oven
7031-J002A	RI-4035 Refractive Index Detector, for Semi-micro LC
7058-J011A	BS-4000-1 Bottle stand
6688-H564A	LC-Net CG cable (3x)
7059-J012A	ChromNAV Ver.2 Chromatography Data System
7001-H403A	RHPLC/UHPLC Start Up Kit for LC-4000
7001-H405A	Maintenance tool kit
	2x Shodex GPC LF-404 (4.6 mmI.D. x 250 mmL, 6 μ m)

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