

Evaluation of Polycarbonate (PC) Degradation by Molecular Weight Distribution Measurements

Introduction

Polycarbonate (PC) is a polymer commonly used in electronic devices and construction materials. PC undergoes degradation due to factors, such as exposure to ultraviolet light or heat and repeated recycling. Quantitatively evaluating the associated changes in physical properties is important for developing products made from PC and controlling their quality. One method for performing such evaluations of polymer raw materials involves the use of gel permeation chromatography (GPC) to determine their molecular weight distribution. When analyzing PC by GPC, tetrahydrofuran (THF) is typically used as an eluent.

In this application note, PC test samples were exposed to irradiation in a xenon accelerated weathering tester, and the resulting changes in molecular weight distribution were evaluated using a GPC system equipped with an RI-4030 refractive index detector and a high-performance analytical GPC column. Data analysis was performed using the molecular weight distribution calculation program in ChromNAV. A calibration curve generated from polystyrene (PS) standards was used to determine the molecular weight distribution of the degraded PC samples.

Keywords

PC, polycarbonate, GPC, gel permeation chromatography, molecular weight distribution, molecular weight calibration curve, THF, tetrahydrofuran, refractive index detector, RI detector

Experimental

Instruments		LC Conditions	
Pump:	PU-4180*	Column:	GPC KF-806L x2 (8.0 mm I.D. x 300 mm L, 10 μ m)
Autosampler:	AS-4150	Eluent:	THF (stabilizer-free)
Column oven:	CO-4060	Flow rate:	1.0 mL/min
Detector:	RI-4030	Column temperature:	40 °C
* with option units		Injection volume:	100 μ L

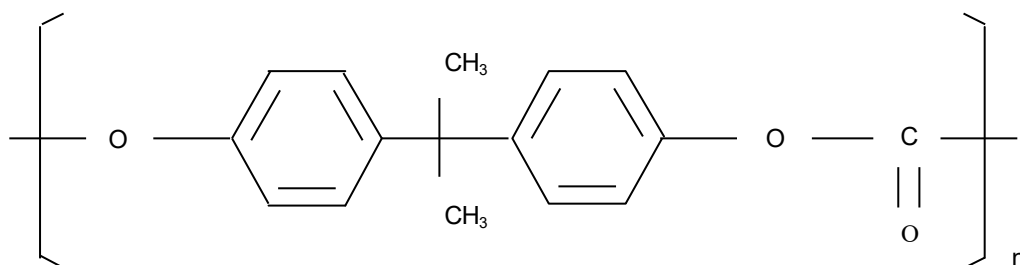


Fig. 1 Structure of polycarbonate (PC)

Sample

Standard samples for creating molecular weight calibration curve

PS mixed sample

(two samples were prepared for different molecular weight peaks (M_p))

Standard sample 1: M_p 1,470,000; 257,000; 18,100; 3,090

Standard sample 2: M_p 778,000; 64,500; 6,320

Each standard sample was dissolved and diluted in THF to 0.025 % (w/v).

Test samples for evaluation

PC test samples (approximately 3 mm, pellet-shaped, Standard Test Piece Co., Ltd.)

Results

Figure 2 illustrates the photodegradation test procedure. PC test samples were irradiated using a xenon accelerated weathering tester (SOLARBOX 1500e, manufactured by Cofomegra, provided by JASCO INTERNATIONAL Co., Ltd.) at an irradiance of 60 W/m² and a temperature of 65 °C. Three samples were sequentially introduced into the weathering tester at different times to achieve total irradiation times of 10 days, 5 days, and 1 day for Samples 1, 2, and 3, respectively. Following irradiation, the degraded surface layer of each sample was removed using a plane slicer (Slice Master KS-10, provided by JASCO Engineering Co., Ltd.) and dissolved in THF at a concentration of 0.1% (w/v) for GPC analysis.

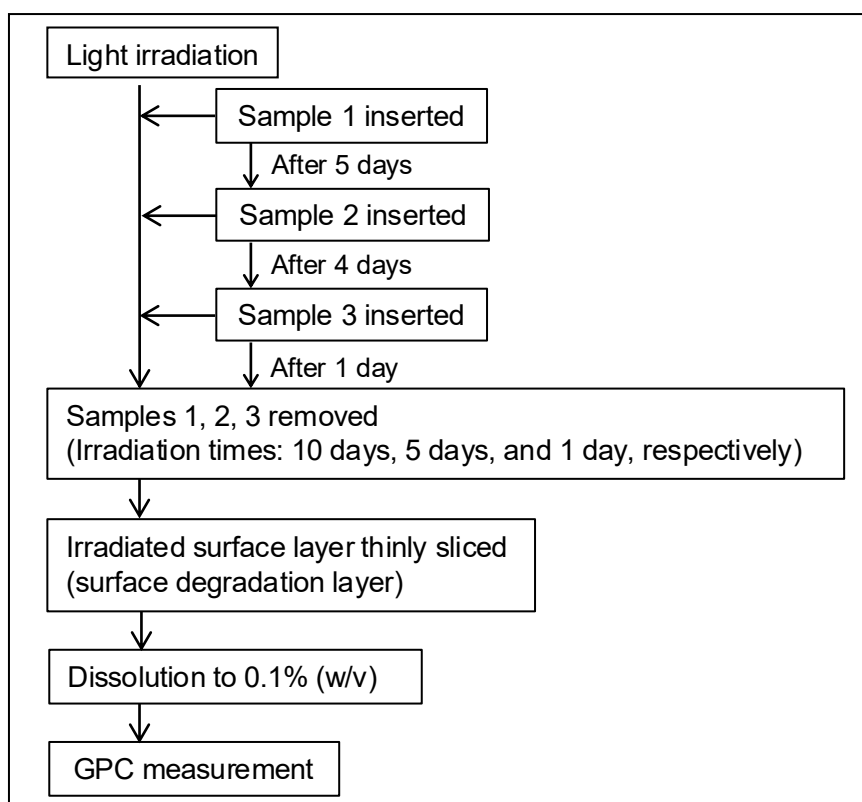


Fig. 2 Photodegradation test procedure

Figure 3 shows the chromatograms of the two PS standard samples used for the molecular weight calibration. The resulting calibration curve is shown in Figure 4.

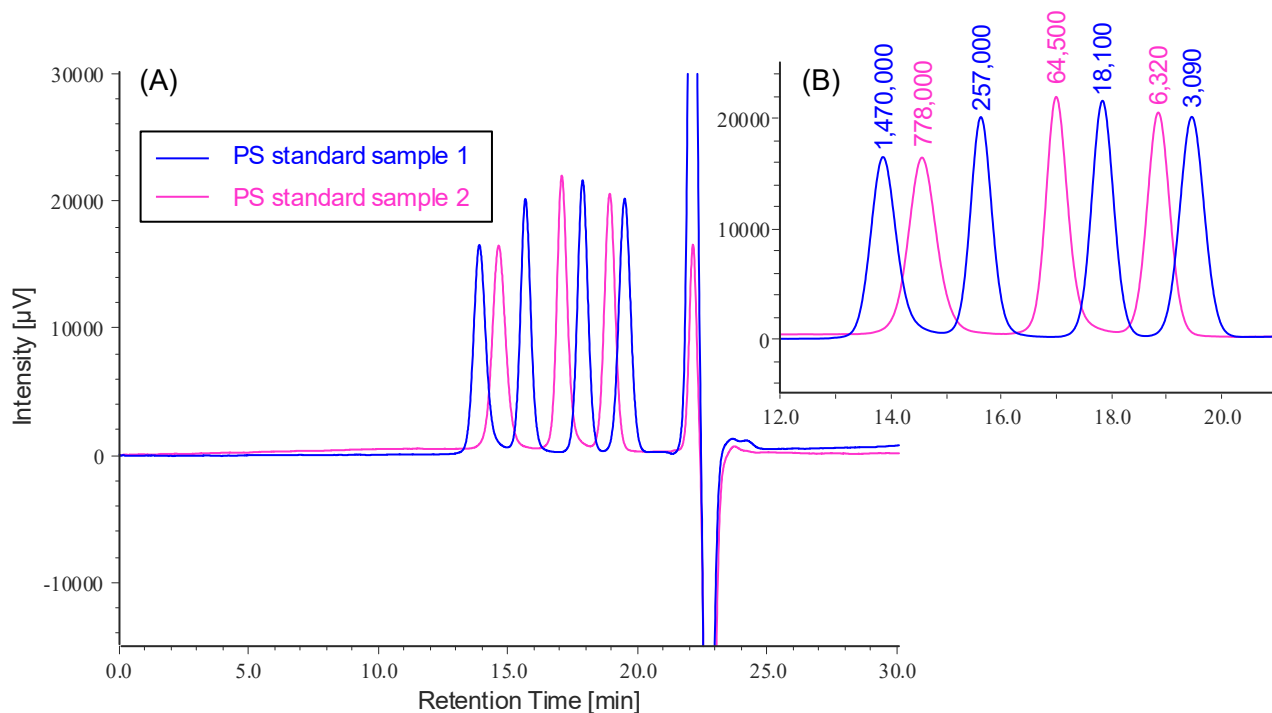


Fig. 3 Chromatograms of the two PS standard samples
(A) Full chromatograms (B) Enlarged view of the peaks
(The values above the peaks represent the M_p for the PS standard samples.)

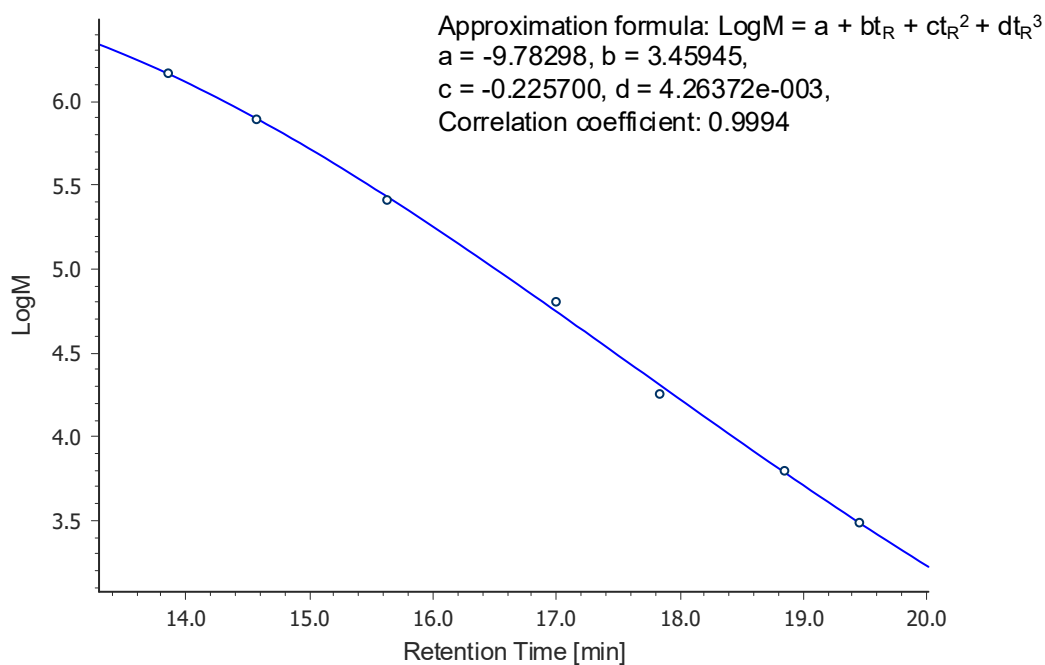


Fig. 4 Molecular weight calibration curve created using the two PS standard samples

Figure 5 compares the chromatograms of the three irradiated PC samples with that of an unirradiated sample. The corresponding differential molecular weight distribution curves are shown in Figure 6. Molecular weight is plotted on a logarithmic scale to facilitate comparison of the distributions. The curves show a slight shift toward higher molecular weights with increasing irradiation time.

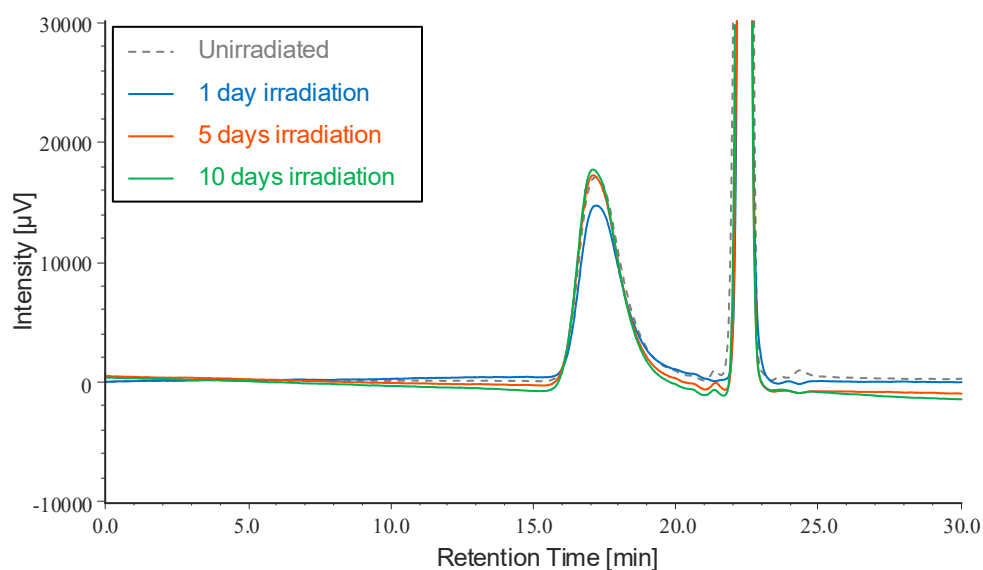


Fig. 5 Chromatograms of the PC test samples

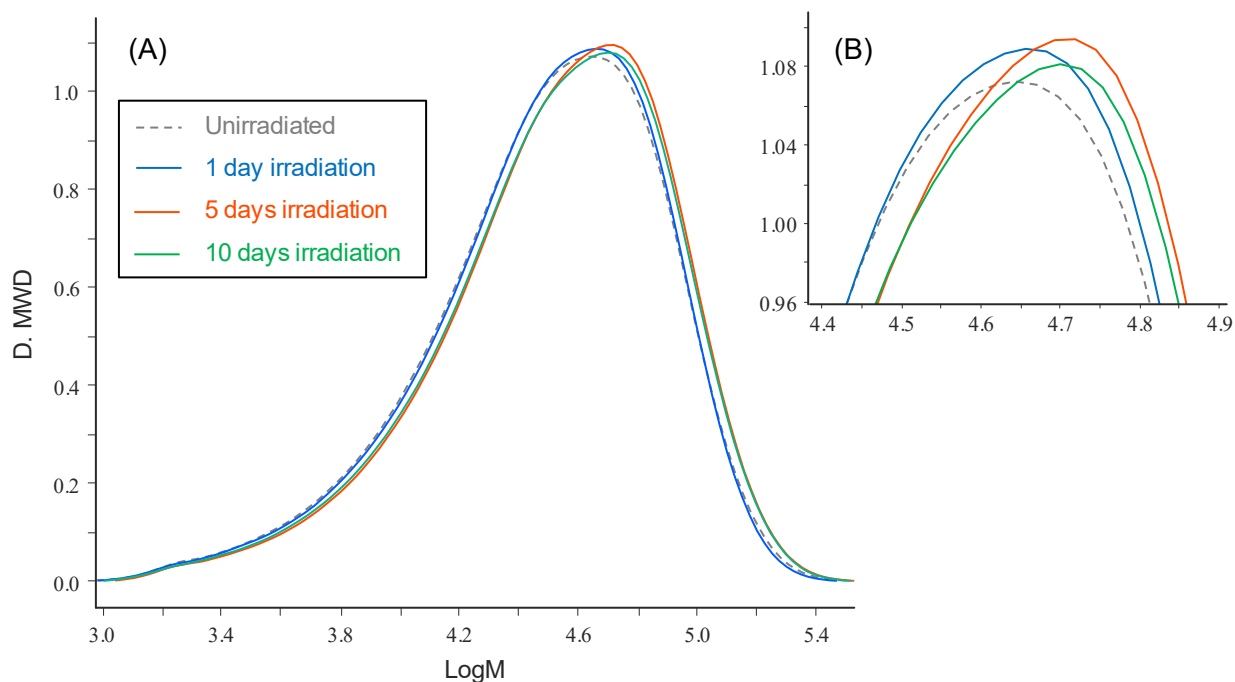


Fig. 6 Differential molecular weight distribution curves for PC test samples
(A) Full chromatograms (B) Enlarged view of peak top

Table 1 summarizes the PS equivalent average molecular weight calculation results. Figure 7 shows the changes in the number-average molecular weight (M_n) and weight-average molecular weight (M_w) as a function of irradiation time.

Table 1. Average molecular weight calculation results for PC test samples

Sample	M_p	M_n	M_w	M_w/M_n
Unirradiated	43,626	18,907	42,653	2.26
1 day irradiation	44,953	19,108	42,578	2.23
5 days irradiation	50,165	20,744	46,311	2.24
10 days irradiation	49,175	20,265	45,854	2.27

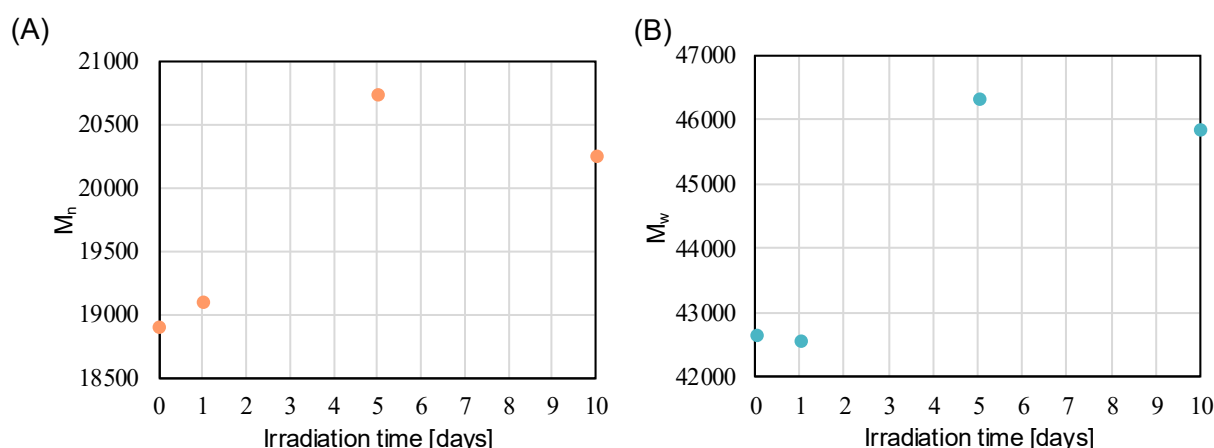


Fig. 7 Change in average molecular weight with irradiation time

(A) Number-average molecular weight (M_n) (B) Weight-average molecular weight (M_w)

Between 1 and 5 days of irradiation, the peak molecular weight (M_p), weight-average molecular weight (M_w), and number-average molecular weight (M_n) increased, suggesting that intermolecular condensation and cross-linking may occur during light irradiation.¹

Conclusion

In this application note, GPC analysis was used to evaluate changes in the molecular weight distribution of PC samples following irradiation in a xenon accelerated weathering tester. Between 1 and 5 days of irradiation, the PS-equivalent peak molecular weight (M_p), weight-average molecular weight (M_w), and number-average molecular weight (M_n) increased, suggesting that intermolecular condensation and cross-linking may occur during light exposure. These results demonstrate the utility of molecular weight distribution measurements for quantitatively assessing changes in PC associated with photodegradation.

References

1. SABIC Innovative Plastics IP B.V: J. F. Morizur, P. D. Sybert, US 20140178665A1, "Cross-linked polycarbonate resin with improved chemical and flame resistance" (2014.6.26).

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