

# DVD Substrate Application for Polycarbonate End-Capped with 4- $\alpha$ -Cumylphenol

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**ABSTRACT:** Using 4- $\alpha$ -cumylphenol as the end-capping agent, polycarbonates (PCs) with a viscosity-average molecular weight ( $M_v$ ) of 13,800 were prepared. DVD substrates were molded using these PCs. DVD substrates using PCs end-capped with 4- $\alpha$ -cumylphenol (CP-PCs) have lower retardation than do those using conventional PCs end-capped with 4-*tert*-butylphenol (TBP-PCs). Using CP-PC, it is possible

to bring the mold temperature down about 20°C from the standard conditions of molding DVD substrates using TBP-PC at the same radial birefringence. © 2003 Wiley Periodicals, Inc. *J Appl Polym Sci* 88: 404–406, 2003

**Key words:** polycarbonate; retardation; DVS substrate; end-cap structure; 4- $\alpha$ -cumylphenol

## INTRODUCTION

Polycarbonates (PCs) are excellent engineering thermoplastics because of their good impact strength, thermal resistance, and transparency.<sup>1,2</sup> Recently, the demand for optical disks where PCs are used as substrates for CDs and DVDs has drastically increased. To decrease radial birefringence, molecular weights of PCs are decreased and mold temperatures increased as much as possible. Accordingly, disks, especially DVDs 0.6 mm thick, are apt to break frequently in the molding process, and this requires developing such PCs with a better balance of fluidity and impact resistance.

In the latest studies on optical disks, there are several reports on the relationship between the method of molding and birefringence,<sup>3–9</sup> but there are no reports on decreasing radial birefringence using the specific end-cap structure of PC. In a previous report,<sup>10</sup> the author reported that PCs end-capped with 4- $\alpha$ -cumylphenol or 4-*tert*-octylphenol were superior in impact strength with a better balance of fluidity, thermal resistance, and impact resistance. This article describes the application of PCs end-capped with 4- $\alpha$ -cumylphenol (CP-PCs) for DVD substrates compared with conventional PCs end-capped with 4-*tert*-butylphenol (TBP-PCs).

## EXPERIMENTAL

### Materials

Commercial bisphenol A (BPA), TBP, CP, triethylamine (TEA), sodium hydroxide, dichloromethane, sodium dithionite, and tris(nonylphenyl) phosphite

were used without further purification. The TBP-PC used was Panlite AD5503 (optical disk grade,  $M_v$  15,000) manufactured by Teijin Chemicals Ltd. (Chiyoda, Tokyo, Japan).

### Preparation of the PC oligomer

Sodium dithionite (120 g, 0.69 mol) and BPA (60 kg, 263 mol) were dissolved in 5 wt % aqueous sodium hydroxide (400 L). This solution, dichloromethane, and phosgene were introduced through an orifice plate at 25°C with a flow rate of 138 L/h, 69 L/h, and 10.7 kg/h (108 mol/h), respectively, into a tubular reactor with an inner diameter of 10 mm and a length of 10 m. This procedure was continued for 3 h. Since phosgene is a toxic, irritating gas, it was dealt with only in an efficient fume hood. After allowing the reaction solution to stand for a certain time, the organic phase was separated to obtain a PC oligomer solution with an oligomer concentration of 311 g/L and a chloroformate group (CF) concentration of 0.72 mol/L.

### Preparation of CP-PC

A solution of CP (157 g, 0.74 mol) in dichloromethane (400 mL), aqueous 5.8 wt % sodium hydroxide (0.6 L), and TEA (3.76 g, 37.2 mmol) was added to the PC oligomer solution (9L, CF: 6.48 mol). The solution was stirred at 300 rpm at room temperature for 1 h. Then, a solution of sodium dithionite (1.2 g, 66 mmol), BPA (611 g, 2.67 mol) and sodium hydroxide (357 g, 8.93 mmol) in water (5 L), and dichloromethane (10 L) was added and stirred at 500 rpm at 23°C for 2 h. Dichloromethane (5 L) and water (5 L) were added, and the solution was kept standing for a certain time. The

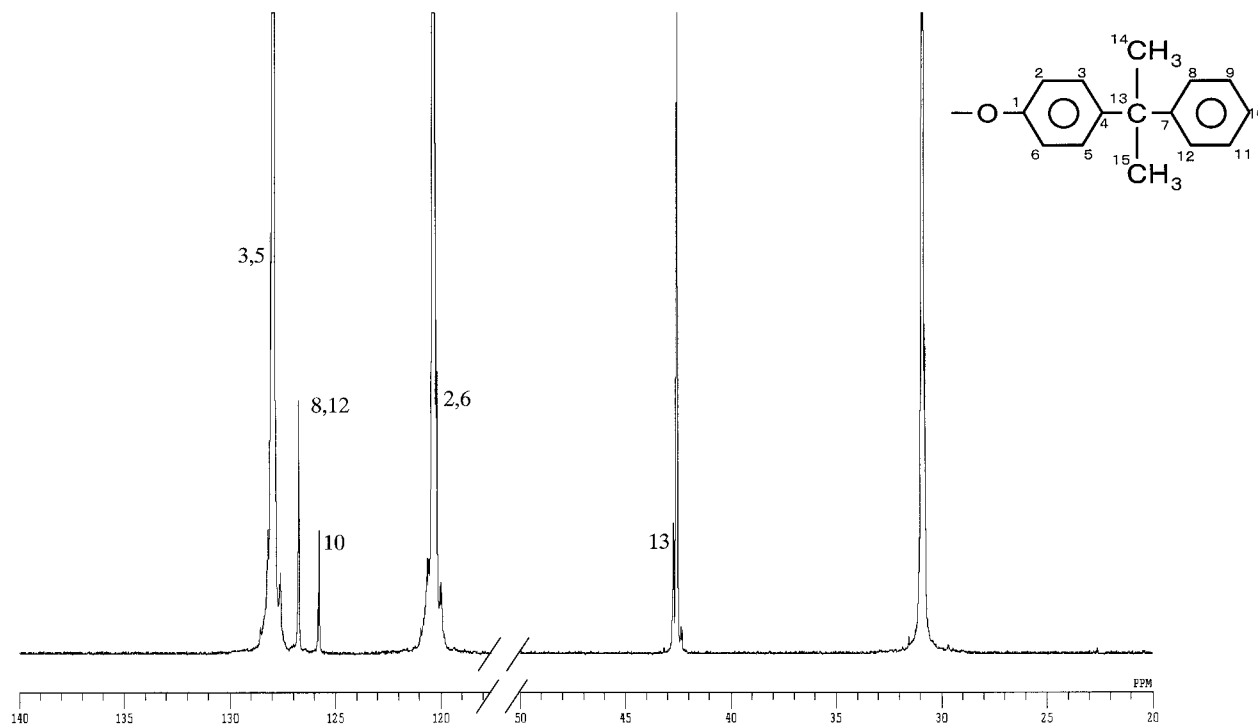


Figure 1  $^{13}\text{C}$ -NMR spectrum of CP-PC in  $\text{CDCl}_3$ .

organic phase that separated was washed successively with 0.01M aqueous sodium hydroxide (5 L), 0.1M aqueous hydrochloric acid (5 L), and water (5 L). Thereafter, the dichloromethane was evaporated to obtain PC in a flake form.

The resulting PC was dried for 12 h at  $120^\circ\text{C}$  and pelletized with tris(nonylphenyl) phosphite as an oxidation inhibitor (50 wt ppm) by an extruder at  $270^\circ\text{C}$ . The pellets obtained were dried for 24 h at  $120^\circ\text{C}$ . The average viscosity molecular weight ( $M_v$ ) of the CP-PC was 13,800.<sup>1,10</sup> Similarly, a TBP-PC with an  $M_v$  of 13,800 was prepared.

### Molding

The resulting pellets were molded to produce DVD substrates by injection-compression molding using an M35B-D-DM (VISTAC) manufactured by the Meiki Co., Ltd. The conditions were a cylinder temperature of  $380^\circ\text{C}$ , mold temperature of  $90$ – $110^\circ\text{C}$ , locking force of 17.5 tons, and molding cycle of 5.5 s. Each sample

was picked after 50 shots. The DVD substrates were subjected to measurement of the retardation and tilt.

## RESULTS AND DISCUSSION

### Preparation and characterization of CP-PC

A two-step method was used to prepare the PCs: The first step was precondensation between the phenols and part of the PC oligomers, which have the chloroformate group at their ends. The second step was polycondensation between BPA and the prepolymers.

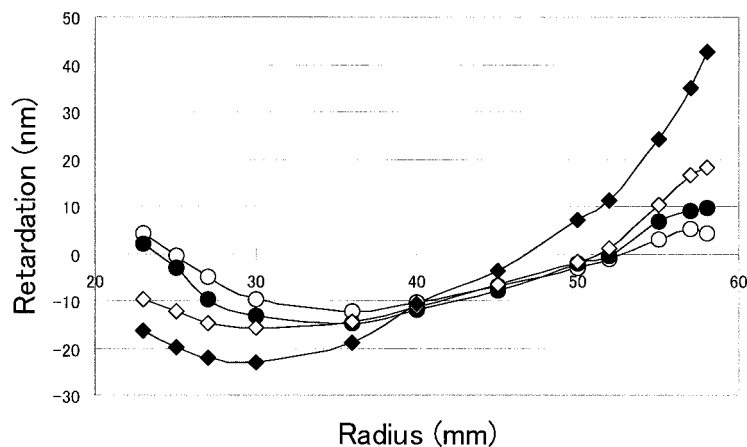
The structures of the end-capped PCs were characterized by IR and  $^{13}\text{C}$ -NMR spectroscopy.<sup>10</sup> The IR spectra of the PCs showed characteristic carbonate bonds at 1770 and  $1230$ – $1260\text{ cm}^{-1}$ . The  $^{13}\text{C}$ -NMR spectrum of CP-PC is shown in Figure 1. The numbers in the figure correspond to the terminal carbons. Signals for the carbon nuclei of C 13, C 2, C 10, C 8, and C 3 are observed at 42.3, 120.2, 125.8, 126.7, and 128.1 ppm, respectively. These findings clearly indicate the formation of CP-PC.

TABLE I  
Retardation and Tilt of DVD Substrate

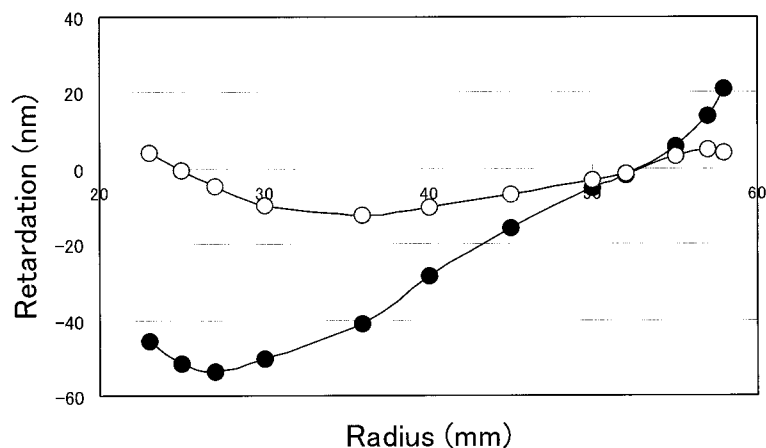
| Run | Resin               | Mold temperature ( $^\circ\text{C}$ ) | Retardation <sup>a</sup> (nm) | Radial tilt (max) | Tangential tilt (max) |
|-----|---------------------|---------------------------------------|-------------------------------|-------------------|-----------------------|
| 1   | CP-PC               | 110                                   | -14 to +13                    | -1.21 $^\circ$    | 0.32 $^\circ$         |
| 2   | CP-PC               | 105                                   | -17 to +18                    | -0.98 $^\circ$    | 0.14 $^\circ$         |
| 3   | CP-PC               | 100                                   | -18 to +25                    | -0.54 $^\circ$    | 0.15 $^\circ$         |
| 4   | CP-PC               | 90                                    | -28 to +53                    | -0.93 $^\circ$    | 0.12 $^\circ$         |
| 5   | TBP-PC <sup>b</sup> | 110                                   | -58 to +24                    | -1.09 $^\circ$    | 0.11 $^\circ$         |

<sup>a</sup> Double pass.

<sup>b</sup> Panlite AD5503 manufactured by Teijin Chemicals LTD.



**Figure 2** Relationships between radius of the DVD substrate and retardation (double pass) of the CP-PC. Mold temperature: (○) 110°C; (●) 105°C; (◇) 100°C; (◆) 90°C.



**Figure 3** Relationships between radius of the DVD substrate and retardation (double pass): (○) CP-PC; (●) TBP-PC. Mold temperature: 110°C.

### Radial birefringence of the DVD using CP-PC

In a previous report,<sup>10</sup> the author reported that CP-PC is superior in impact strength with a better balance of fluidity and impact resistance than those of the conventional TBP-PC, so a CP-PC with an  $M_v$  of 13,800 was prepared and evaluated for the DVD substrate. The optical grade  $M_v$  currently on the market is about 15,000. The TBP-PC obtained with an  $M_v$  of 13,800 was broken in the molding process. The results are summarized in Table I. The relationships between the radius of the DVD substrate and retardation (double pass) are shown in Figures 2 and 3. In Figure 2, retardation decreases with an increasing mold temperature. When the mold temperature is higher, the decrease of radial birefringence is considered to be due to the decrease of residual stress of the disk by relaxation of the molecular orientation. In Figure 3, CP-PC has lower a retardation than that of TBP-PC due to the differences in  $M_v$ , and CP-PC is superior in radial birefringence at the same mold temperature. Using CP-PC, it is possible that the mold temperature can be

decreased about 20°C from standard molded DVD conditions that use TBP-PC at same birefringence. As a result, it is considered that the molding cycle can be shortened still more.

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