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Syntheses and Optical Properties of Polyesters with Disperse Red 19 by the Polycondensation Reaction

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We synthesized the polyesters containing azobenzene units in the side chain for data storage materials. The structurally similar polyesters, DR-Est-1, DR-Est-2 and DR-Est-3 were synthesized to compare the optical properties of polyesters with different structure from dihalide in the main chain. The diffraction efficiencies of films were measured as a function of the reaction time. Typically, the diffraction efficiency of the DR-Est-3 film prepared from 4,4'-biphenyldicarbonyl chloride was observed up to a level of 0.4 %.

Keywords Polyester; Polycondensation reaction; Disperse red 19; Azobenzene; Diffraction efficiency

INTRODUCTION

Recently, a large number of azobenzene containing polymers have been

chemically synthesized with the main focus on polymers containing azobenzene groups in the side chain ^[1, 2-3]. The photochemically induced trans-cis-trans isomerization of the azobenzene groups within the polymer can generate a local orientation of the azobenzene groups when the polymer film is irradiated with a linearly polarized laser beam. Polymers containing azobenzene groups in the side chain are being studied as suitable materials for reversible optical storage ^[4].

In this study, we investigated the polyesters containing azobenzene units in the side chain for data storage materials. The structurally similar polyesters, DR-Est-1, DR-Est-2 and DR-Est-3 were synthesized to compare the optical properties of polyesters with different structure from dihalide in the main chain. In addition, the process of reversibly photoinduced birefringence was tested. The diffraction efficiency of the polyester film was measured as a function of the reaction time.

EXPERIMENTAL

Four structurally similar polyesters containing azobenzene group in the side chain were prepared by the polycondensation reaction. Typical polymerization of DR-Est-1 was carried out as follows;

Phthaloyl chloride (2 mmol) in tetrahydrofuran (6 ml) was added to a solution of DR 19 (2 mmol) and triethylamine as a catalyst (2 mmol) dissolved in tetrahydrofuran (7 ml) under nitrogen atmosphere. The reaction mixture was refluxed and vigorously stirred at 60°C for 3h. Then, the solution was poured into methanol. The precipitate was filtered, washed with water, and methanol. Finally, the polymer was dried under vacuum at 40°C for 24h. DR-Est-2 and DR-Est-3 were synthesized according to the previously described procedure for DR-Est-1, except *i*-phthaloyl chloride and 4,4'-biphenyldicarbonyl chloride, respectively. The reaction path to the formation of DR-Est-*n*'s is shown in FIGURE 1. DR-Est-*n*'s were dissolved in chloroform (3.5% w/w) and then polymer solutions were filtered with 0.45 μm syringe filters. The films were prepared by spin-coating from solution onto a clean glass slide and dried in vacuum at 50°C for 24h. The film thickness

was 1 μm measured by interferometry.

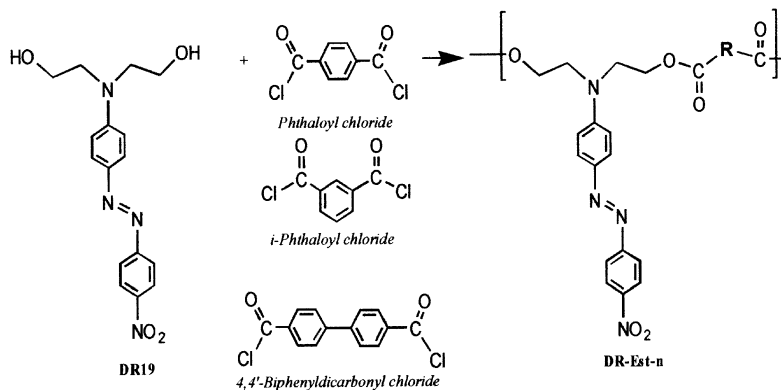


FIGURE 1. The reaction path to the formation of DR-Est-*n*'s

RESULTS and DISCUSSION

We synthesized three different polyesters (DR-Est-1, DR-Est-2, and DR-Est-3) by the polycondensation reaction in the presence of triethylamine. The structures of DR-Est-*n*'s were characterized by FT-IR and $^1\text{H-NMR}$ spectra. FT-IR spectra of DR-Est-*n*'s showed characteristic absorption peaks around 1725 cm^{-1} due to C=O stretching of the carbonyl group, and 1145 cm^{-1} due to -O- stretching of the carbonyl group. The $^1\text{H-NMR}$ spectrum of DR-Est-3 exhibited characteristic peaks at δ 8.36-8.08 ppm assignable to aromatic carbons of the azobenzene, δ 7.93-7.81 ppm assignable to aromatic carbons of the phthaloyl chloride. The UV absorption spectra of DR-Est-1, DR-Est-2, and DR-Est-3 showed the maximum at 416, 482, and 480 nm, respectively. The detailed structural data can be summarized as follows. Spectral data for DR-Est-1: FT-IR (KBr), 2923 cm^{-1} (CH aliphatic), 1725 cm^{-1} (C=O), 1145 cm^{-1} (-O-); Spectral data for DR-Est-2: FT-IR (KBr), 2924 cm^{-1} (CH aliphatic), 1723 cm^{-1} (C=O), 1143 cm^{-1} (-O-); Spectral data for DR-Est-3: FT-IR (KBr), 2925 cm^{-1} (CH aliphatic), 1721 cm^{-1} (C=O), 1144 cm^{-1} (-O-).

FIGURE 2 shows the diffraction efficiency as a function of time for DR-Est-3 during the recording process. An Ar^+ laser beam at 514 nm with an intensity of 23 mW/cm^2 was used as the pump beam. A laser diode at 650 nm used as the probe beam (writing beam) was inputted as an intensity of 26.5 mW/cm^2 . When the writing beam was turned on, the diffraction efficiency increased rapidly and reached 0.4 % in DR-Est-3. In the case of DR-Est-1 and DR-Est-2, the diffractions were appeared relatively low values (below 0.1 %). After the laser beams were turned off, the diffraction efficiency decayed to nearly zero, indicating that most of the orientation grating had been erased.

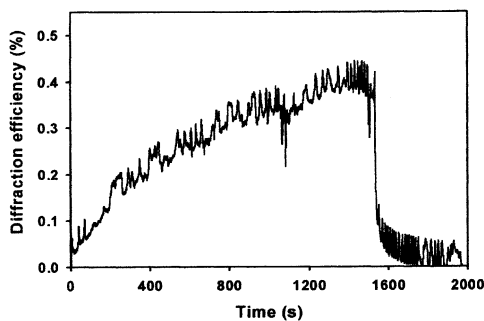


FIGURE 2. Diffraction efficiency as a function of time for DR-Est-3.

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