Optically Transparent and Thermal-Stable Polyimide Films Derived from a Semi-Aliphatic Diamine: Synthesis and Properties

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A novel semi-aliphatic diamine with bulky cyclohexyl and ortho-substituted tertiary butyl groups (4,4'-(cyclohexylmethylene)bis[2-(tert-butyl)aniline], CHMBTBA) is synthesized via Mannich and rearrangement reactions of cyclohexanecarboxaldehyde and 2-tert-butylaniline. Afterward, several semi-aliphatic polyimides are prepared by the high-temperature one-pot polymerization of CHMBTBA with various aromatic dianhydrides respectively. The resulting PIs exhibit outstanding solubility in common organic solvents (i.e., trichloromethane, etc.) and are easily processed into light color or colorless transparent films (thickness: 25 ± 1 µm) by the blade coating of polymer solution. The transmittance of all resulting films is above 86% in the visible light region (400–760 nm). Their glass transition temperatures exceed 310 °C and reach even to 374 °C (by differential scanning calorimetry). They also display low water absorption (0.29–0.87%) and good mechanical properties (tensile strength: 50.9–87.7 MPa, Young’s modulus: 2.0-2.5 GPa, elongation at break: 3.8–7.6%). Thus, they are promising potential candidates for flexible display substrates.

1. Introduction

Over the past few decades, polyimides (PIs) were widely used in various high-tech fields on account of their excellent comprehensive performance (i.e., thermal stability, low coefficient of thermal expansion (CTE < 50 ppm °C−1), and good processability). But so far, it is still a great challenge to balance the mutual restriction of colorless transparency/processability and heat-resistance/low CTE of PIs. In order to lessen or remove the deep color of PIs, the formation of CTC must be depressed by incorporating aliphatic/alicyclic moieties or asymmetric noncoplanar structures, and fluorine elements in polyimide main chains. Simultaneously these approaches could also improve the processability of PIs, but inevitably accompanying with the thermostability of PIs decreasing and the CTE or synthetic cost increasing.

Fortunately, the introduction of bulky pendant substituents can improve the processability and optical transparency without seriously sacrificing the thermal stability of PIs. In other words, PIs containing alicyclic moieties as bulky pendant substituents were more stable than those as structural units in main chains. In addition, our earlier works indicated tert-butyl as ortho-substituent of amino-groups in diamine was greatly enhanced the solubility of corresponding PIs while restrained the formation of CTC to improve the transparency of PI films. Therefore, we speculate that introducing bulky cyclohexyl and ortho-substituted tertiary butyl groups into the backbone of PIs can improve the colorless transparency as well as retain the high thermally stability. Herein, a novel diamine...
CHMBTBA containing bulky cyclohexyl and ortho-substituted tertiary butyl groups was synthesized via Mannich and rearrangement reactions from cyclohexanecarboxaldehyde and 2-tert-butylaniline in the presence of hydrochloric acid (HCl). The corresponding PIs were prepared by the one-pot polycondensation of CHMBTBA with various aromatic dianhydrides and their properties were investigated in detail.

2. Results and Discussion

The synthesis processes of novel semi-aliphatic diamine CHMBTBA (c) and corresponding PIs are shown in Scheme 1. In the presence of HCl as the catalyst, cyclohexanecarboxaldehyde (b) was used as a linker to bridge 2-tert-butylaniline (TBA; a) to produce CHMBTBA. No other organic solvent was required in the reaction process because excessive TBA itself could be used as the reaction solvent.

The chemical structure of CHMBTBA was confirmed by means of $^1$H NMR and $^{13}$C NMR measurements in dimethyl sulfoxide (DMSO)-d$_6$ and the resulting spectra are shown in Figure 1. According to the $^1$H NMR spectrum, the peaks ranging from 0.67 to 1.18 ppm and from 1.44 to 1.65 ppm were ascribed to the CH$_2$ protons of cyclohexane moiety, while the peak at around 1.31 ppm was assigned to the tert-butyl groups. The signal of amino groups appeared at about 4.48 ppm and three signals of benzene rings were found in the range from 6.48 to 7.00 ppm. Furthermore, the signals of $^{13}$C NMR spectrum were also ascribed to the corresponding carbon atoms of CHMBTBA. The melt point of CHMBTBA was observed at 210 °C by the differential scanning calorimetry (DSC) analysis.

Furthermore, the preparation process of corresponding PIs is shown in Scheme 2. According to the previous studies, the reactivity of diamines was reduced by introducing tert-butyl groups into the ortho-position of amino group.[25–28] Thus the high-temperature one-step method should be used to prepare PIs. In this study, all the PIs were synthesized in m-cresol with isoquinoline as the catalyst at the solid content of 10 wt% by the high-temperature one-step method.

![Scheme 1](image1.png)

**Scheme 1.** Synthesis of semi-aliphatic diamine CHMBTBA.

![Scheme 2](image2.png)

**Scheme 2.** Synthesis of PIs from diamine CHMBTBA with various dianhydrides.

![Figure 1](image3.png)

**Figure 1.** $^1$H and $^{13}$C NMR spectra of diamine CHMBTBA.
The resulting PIs were characterized by $^1$H NMR, fourier transform infrared spectroscopy (FTIR), elemental analysis, and gel permeation chromatography (GPC) measurements. The $^1$H NMR spectra are shown in Figure 2, and all the signals have been labeled and assigned to the corresponding protons. The FTIR spectra of the PIs are shown in Figure 3. As shown in the FTIR spectra, the characteristic bonds of imide units of all PIs appear at 1367–1374 cm$^{-1}$ (C–N asymmetric stretching), 1725–1730 cm$^{-1}$ (C=O symmetrical stretching), and 1778–1787 cm$^{-1}$ (C=O asymmetrical stretching). In addition, the carbon–hydrogen bond in tert-butyl could be observed at 2927–2932 cm$^{-1}$. No obvious signals of amide units were observed in the region between 3220 and 3440 cm$^{-1}$ (N-H stretching), which indicated the PIs were imidized completely.

The elemental analysis results in Table 1 were in accordance with the theoretically calculated values of them and further confirmed their chemical structures. The number–average molecular weights ($M_n$) and the polydispersity indices (PDIs) of these PIs were in the range from 2.28 to 15.43 $\times$ 10$^4$ and 2.38 to 5.78, respectively, in Table 2. The water absorption rates of the resulting PIs were measured at the room temperature and the data are listed in Table 2. Obviously, the water absorption rates were in the range from 0.28% to 0.87%, which were far lower than that of the commercial Kapton film (3.1%).

The solubility of the resulting PIs was determined by dissolving 10 mg polymer in 1 mL solvent at 25 °C and the results are displayed in Table 3. All the PIs exhibited excellent solubility in various organic solvents not only polar solvents DMSO, NMP but also low boiling solvents THF, CHCl$_3$, etc. Obviously the introduction of tert-butyl and cyclohexyl groups into PIs significantly inhibited the formation of CTC while the steric hindrance of bulky pendant groups increased interchain spacing. The synergistic effect of both enhanced the solubility of the resulting PIs. These highly soluble PIs were easily processed into light color or colorless transparent films by the blade coating of polymer solution. The morphology of the resulting PI films was studied by X-ray diffraction (XRD) and the corresponding diffractograms were shown in Figure 4. Evidently, all the PI films were in amorphous patterns, which suggested the tert-butyl and cyclohexyl groups disturbed the packing of PI chains and decreased their packing density.

The thermal properties of the PIs were evaluated by DSC, dynamic mechanical analyzer (DMA), and thermogravimetric analysis (TGA) measurements under N$_2$ atmosphere. The DSC
and DMA curves of PIs were shown in Figure 5a,b. Both of the corresponding $T_g$ were collected in Table 2. Accordingly, the $T_g$ of the resulting PIs were from 310 to 374 °C by DSC measurement as well as from 314 to 394 °C by DMA measurement. The variation trends of both agreed well with each other. Among them, PI-4 possessed the lowest $T_g$ due to the flexible ether linkage of ODPA dianhydride whereas PI-1 displayed the highest $T_g$ because of the rigid structure of PMDA. Therefore, the introduction of bulky pendant groups (tert-butyl and cyclohexyl groups) did not decrease the $T_g$s of the resulting PIs because they increased the steric hindrance and hindered the motion of PI chains.

The TGA curves of the resulting PIs are displayed in Figure 5c and the values of $T_{5\%}$ were also summarized in Table 2. Evidently, all the PIs exhibited high thermal stability and maintained their weight within 5% up to 480 °C. In the same way, the introduction of tert-butyl and cyclohexyl groups did not sacrifice the inherent thermal stability of PIs.

The UV–vis spectra of the resulting PI films (Thickness: 25 ± 1 μm) are displayed in Figure 5d and the corresponding optical properties are listed in Table 4. The transparency of all PI films was above 86% in the visible light range (400–760 nm) and the cut-off wavelengths were in the range of 306–350 nm. In particular, the PI-5 film prepared from CHMBTBA and 6FDA had the highest average transparency (90%) and the lowest cut-off wavelength (306 nm).

The optical photographs of the PI films are shown in Figure 6 and the thickness of all the PI films was 25 ± 1 μm. Obviously PI-4 to PI-5 presented better transmittance and colorlessness than PI-1 to PI-3 in visible light range, which was agreed with the results of UV–vis spectra. In a word, two bulky pendant groups (tert-butyl and cyclohexyl groups) significantly restrained the formation of CTC and resulted in the excellent optical properties of PI films.

The dimensional stability of the PI films was evaluated by thermomechanical analysis (TMA) measurements and the values of CTE were outlined in Table 5. Generally, PIs with linear/stiff backbones usually had low CTE values due to their large packing density. What’s more, the level of in-plane orientation of PI films played a more important part in dominating the low CTE than that of the backbone. However, the high molecular mobility usually caused orientational relaxation in annealing. Here, the introduction of tert-butyl and cyclohexyl groups was able to increase the free volume and improve the molecular mobility in the blade-coating process. Thus these PI films exhibited relatively high CTEs from 47 to 71 ppm °C⁻¹. On the other hand, the structure of dianhydrides also had effects on the CTEs of PI films. For example, PI-1 (CHMBTBA-PMDA) displayed the lowest CTE (47 ppm °C⁻¹) on the base of the rigid structure of PMDA. On the contrary, PI-4 (CHMBTBA-ODPA) showed the largest CTE (71 ppm °C⁻¹) due to the flexible ether-linkage of ODPA with the high segmental motion. The mechanical properties of the PI films were measured by tensile tests at 25 °C and

### Table 1. Elemental analysis data of PIs.

| PIs   | Formula               | C         | H         | N         |
|-------|-----------------------|-----------|-----------|-----------|
| PI-1  | C_{37}H_{38}N_{2}O_{4} | 77.33     | 6.50      | 2.28      |
| PI-2  | C_{46}H_{42}F_{6}N_{2}O_{4} | 76.90     | 6.32      | 4.97      |
| PI-3  | C_{43}H_{42}N_{2}O_{4} | 79.36     | 6.50      | 2.85      |
| PI-4  | C_{42}H_{42}N_{2}O_{4} | 77.45     | 6.35      | 4.28      |
| PI-5  | C_{46}H_{42}F_{6}N_{2}O_{4} | 78.57     | 6.61      | 4.25      |

### Table 2. Characterization data of PIs.

| PIs   | $M_n \times 10^{4}\text{[g mol}^{-1}\text{]}$ | PDI$^{(\text{h})}$ | $T_g^{(\text{i})}$ [°C] | $T_m^{(\text{i})}$ [°C] | $T_{5\%}^{(\text{j})}$ [°C] | Water absorption [%] |
|-------|------------------------------------------|-----------------|----------------------|----------------------|----------------------|---------------------|
| PI-1  | 15.43                                    | 2.38            | 374                  | 394                  | 517                  | 0.84                |
| PI-2  | 2.28                                     | 3.78            | 325                  | 340                  | 487                  | 0.28                |
| PI-3  | 7.87                                     | 3.20            | 352                  | 378                  | 500                  | 0.87                |
| PI-4  | 2.28                                     | 3.31            | 310                  | 314                  | 525                  | 0.29                |
| PI-5  | 4.97                                     | 3.72            | 317                  | 322                  | 526                  | 0.34                |

* $M_n$, number-averaged molecular weight; PDI, polydispersity index; $T_g$, glass transition temperature, evaluated by DSC; $T_m$, glass transition temperature, evaluated by DMA; $T_{5\%}$, temperature at 5% weight loss.

### Table 3. Solubility of PIs.

| PIs   | DMSO$^{(\text{k})}$ | NMP$^{(\text{l})}$ | CHCl$_3$ | THF$^{(\text{m})}$ | acetone | Methylbenzene | n-Hexane |
|-------|---------------------|-------------------|---------|-------------------|---------|---------------|----------|
| PI-1  | ++                  | ++                | ++      | ++                | ++      | ++            | +        |
| PI-2  | ++                  | ++                | ++      | ++                | --      | --            | --       |
| PI-3  | ++                  | ++                | ++      | ++                | --      | --            | --       |
| PI-4  | ++                  | ++                | ++      | ++                | ++      | ++            | +        |
| PI-5  | ++                  | ++                | ++      | ++                | ++      | ++            | ++       |

$^{(\text{a})}$DMSO, dimethyl sulfoxide; $^{(\text{b})}$NMP, N-methyl-2-pyrrolidone; $^{(\text{c})}$CHCl$_3$, trichloromethane; $^{(\text{d})}$THF, tetrahydrofuran; $^{(\text{e})}$acetone at 25 °C; $^{(\text{f})}$soluble at 25 °C; $^{(\text{g})}$+−, soluble slightly or partial soluble; $^{(\text{h})}$−, insoluble.

Figure 4. X-ray diffraction curves of PI films.

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the results were listed in Table 3. The tensile strength values, tensile modulus, and elongation at break were in the range of 50.9–87.7 MPa, 2.0–2.5 GPa, and 3.8–7.6%, respectively.

3. Conclusions

A series of semi-aliphatic polyimides with bulky pendent tert-butyl and cyclohexyl groups were synthesized and characterized in detail. All of them exhibited excellent solubility and good film-forming property. The transparent and colorless tough PI films with the thickness of 25 ± 1 µm were obtained by the blade coating. These films exhibited good mechanical properties, outstanding thermal stability, and excellent optical properties. The average transmittance of them was from 86% to 90% in the visible light range (400–760 nm) and the cut-off wavelength ranged from 306 to 350 nm. It is feasible approach to improve the transparency and reduce the color of PI films without sacrificing their thermal property by introducing tert-butyl and cyclohexyl groups into PI. These transparent and colorless PI films were potential candidates used as transparent flexible substrates in optical fields.

Table 4. Optical properties of PI films.

| Pls  | Film thickness [µm] | Tvis, [%] | T400b, [%] | λ0c, [nm] |
|------|---------------------|-----------|------------|-----------|
| PI-1 | 24                  | 87        | 50         | 338       |
| PI-2 | 26                  | 86        | 62         | 326       |
| PI-3 | 24                  | 86        | 76         | 350       |
| PI-4 | 25                  | 88        | 79         | 343       |
| PI-5 | 24                  | 90        | 86         | 306       |

a) The average transmittance in the visible light range (400–760 nm); b) Transmittance at 400 nm; c) UV cut-off wavelength.

Figure 5. a) DSC curves of PIs; b) DMA curves of PIs; c) TGA curves of PIs; d) UV–vis spectra of PI films (Thickness: 25 ± 1 µm).

Figure 6. Optical photographs of the PI films (thickness: 25 ± 1 µm).
Table 5. Mechanical behaviors and CTEs of PIs.

| PIs  | Film thickness [µm] | Tensile strength [MPa] | Tensile modulus [GPa] | Elongation at break [%] | CTE [ppm °C⁻¹] |
|------|---------------------|------------------------|----------------------|-------------------------|----------------|
| PI-1 | 77                  | 87.7                   | 2.1                  | 7.6                     | 47             |
| PI-2 | 42                  | 50.9                   | 2.0                  | 5.3                     | 60             |
| PI-3 | 44                  | 68.6                   | 2.5                  | 3.8                     | 57             |
| PI-4 | 56                  | 71.9                   | 2.3                  | 6.5                     | 71             |
| PI-5 | 41                  | 63.5                   | 2.0                  | 5.5                     | 61             |

4. Experimental Section

Materials: 2-tert-Butylaniline (Changzhou Yonghe Fine Chemical Co. Ltd.) and m-cresol (Shanghai Macklin Biochemical Co. Ltd.) were purified by the vacuum distillation before use. Pyromellitic dianhydride (PMDA, Sinopharm Chemical Reagent Co. Ltd.), 3,3',4,4'-diphenylenetetraacryloyl dianhydride (ODPA, Adamas Reagent Co. Ltd.), 3,3',4,4'-benzophenonetetraacryloyl dianhydride (BTDA, J&K Chemical Co. Ltd.), 3,3',4,4'-biphenyltetracarboxylic dianhydride (BPDA, Adamas Reagent Co. Ltd.), and 2,2-bis(3,4-dicarboxyphenyl)-1,1,1,3,3,3-hexafluoropropane dianhydride (6FDA, Shanghai Titan Scientific Co. Ltd.) were purified by recrystallization from acetic anhydride before use. Cyclohexanecarboxaldehyde (Adamas Reagent Co. Ltd), isoquinoline (Shanghai Titan Scientific Co. Ltd.), hydrochloric acid (Sinopharm Chemical Reagent Co. Ltd.), and other materials were used as received.

Measurements: The 1H and 13C NMR measurements were carried out on a 400 MHz Varian NMR spectrometer with CDCl3 or DMSO-d6 as solvents. The spectra of liquid chromatographymass spectrometer and high resolution mass spectrometer (HRMS) were measured by ACQUITYTM UPLC & Q-TOF MS Premier. The FTIR spectra were obtained on a Perkin-Elmer Fourier transform infrared spectrometer. The molecular weights were measured with a Perkin-Elmer series 200 GPC analyzer (polystyrene calibration), using N6, N-dimethylformamid as eluent containing 0.02 M LiBr (0.6 mL min⁻¹). Elemental analysis was performed on a vario EL cube elemental analyzer. The mechanical property of the polyimide films was performed using an INSTRON 4465 tensile tester at a drawing speed of 1 mm min⁻¹. The transparency of films (25 µm thick) was obtained on a Perkin-Elmer Lambda 20 UV–vis spectrometer. The XRD measurement was carried out on a Bruker D8 advance X-ray diffractometer using Cu Kα radiation with 2θ in the range from 5° to 50°. The rates of water absorption were determined by immersing the vacuum-dried films in 25 °C water for 24 h and calculated from the differences of the weights. DSC curves were performed on a TA Discovery DSC Q2000 thermal analyzer under nitrogen atmosphere. The CTE values of PI specimens (15 mm long, 15.03 g (0.1340 mol) cyclohexanecarboxaldehyde was added dropwise over 1 h. Then it was heated to 150 °C and kept at this temperature for 10 h. When the mixture was cooled to 25 °C, 13.4 mL (0.201 mol) aqueous ammonia (28 – 30 wt%) and 50 mL CH2Cl2 were added into it. After washing with water (3 x 50 mL) and concentration under reduce pressure, some brown oil was obtained. The resulting oil was distilled and collected through fraction boiling at 240–250 °C (25 Pa) to give some yellow solid. The solid was further purified by recrystallization in anhydrous alcohol to afford colorless CHMBTBA needles. Yield: 39%; melting point: 210 °C; 1H NMR (400 MHz, DMSO-d6) δ 6.97 (2H, 6.78 (d, J = 8.0 Hz, 2H), 6.51 (d, J = 8.0 Hz, 2H). 4.48 (s, 4H), 3.11 (d, J = 10.8 Hz, 1H), 1.88 (d, J = 10.5 Hz, 1H), 1.59 (s, 3H), 1.50 (d, J = 12.1 Hz, 2H), 1.31 (s, 18H), 1.13 (s, 3H), 0.74 (d, J = 11.1 Hz, 2H). 13C NMR (101 MHz, DMSO-d6) δ 143.40, 133.20, 131.80, 125.34, 117.01, 33.88, 31.74, 26.02. HRMS (ESI, m/z): [M+H]+ calculated for C27H41N2O4, 393.3270; found, 393.3264.

Synthesis of PIs: All the PIs were synthesized by a high-temperature one-pot method. As a typical example, the polyimide of CHMBTBA-6FDA was synthesized according to the following procedure. 5 g (0.0127 mol) CHMBTBA, 5.642 g (0.0127 mol) 6FDA, 60 mL m-cresol, and ten drops isoquinoline were added into a 250 mL three-necked flask and the mixture was stirred at 50 °C for 1 h under N2 atmosphere. Then the mixture was raised successively to 80 °C for 4 h, 150 °C for 12 h, and 180 °C for 12 h. After cooled to 25 °C, 30 mL chloroform was added into the viscous mixture. Then the mixture was poured slowly into 500 mL methanol under the vigorous stirring to afford some white fiber-like precipitate. The polyimide was collected by filtration and dried in vacuum at 100 °C for 24 h. The polyimide was purified by precipitation twice.

Table 5. (Continued)
Acknowledgements

This work was supported by Science and Technology Commission of Shanghai Municipality Basic Research Project (No. 16JC1403600), Equipment Research and Development Sharing Technology Project (No. 41421060301).

Conflict of Interest

The authors declare no conflict of interest.

Keywords

colorlessness, films, polyimides, semi-aliphatic diamine, transparency

Received: November 14, 2019
Revised: December 23, 2019
Published online:

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