Synthesis and Characterization of Novel Poly(amide imide)s Based on Bis(*p*-amidobenzoic acid)-*N*-trimellitylimido-Lleucine

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ABSTRACT: *N*-Trimellitylimido-L-leucine was reacted with thionyl chloride, and *N*-trimellitylimido-L-leucine diacid chloride was obtained in a quantitative yield. The reaction of this diacid chloride with *p*-aminobenzoic acid was performed in dry tetra-hydrofuran, and bis(*p*-amidobenzoic acid)-*N*-trimellitylimido-L-leucine (**5**) was obtained as a novel optically active aromatic imide-amide diacid monomer in a high yield. The direct polycondensation reaction of the monomer imide-amide diacid **5** with 4,4'-diaminodiphenylsulfone, 4,4'-diaminodiphenylether, 1,4-phenylenediamine, 1,3-phenylenediamine, 2,4-diaminotoluene, and benzidine (4,4'-diaminobiphenyl) was carried out in a medium consisting of triphenyl phosphite, *N*-methyl-2-pyrolidone, pyridine, and calcium chloride. The resulting novel poly(amide imide)s (PAIs), with inherent viscosities of 0.22–0.52 dL g⁻¹, were obtained in high yields, were optically active, and had moderate thermal stability. All of the compounds were fully characterized with IR spectroscopy, elemental analyses, and specific rotation. Some structural characterization and physical properties of these new optically active PAIs are reported. © 2002 John Wiley & Sons, Inc. J Appl Polym Sci 84: 35–43, 2002; DOI 10.1002/app.10181

Key words: bis(*p*-amidobenzoic acid)-*N*-trimellitylimido-L-leucine; poly(amide imide)s; direct polycondensation; optically active polymers; inherent viscosity; thermally stable polymers; thermogravimetric analysis (TGA)

INTRODUCTION

Aromatic polyimides and their copolymers are well known as high-performance polymer materials for their excellent mechanical and electrical properties, high thermal and thermo-oxidative stability, and outstanding solvent resistance.^{1,2}

Journal of Applied Polymer Science, Vol. 84, 35–43 (2002) © 2002 John Wiley & Sons, Inc. However, most aromatic polyamides are intractable materials that do not melt before thermally decomposing and, therefore, would be difficult for processing. Aromatic poly(amide imide)s (PAIs), however, are very useful high-performance materials that exhibit better processing characteristics than polyimides of analogues structures.³ A notable example is Torlon[®] (Amoco Co.), a commercial PAI obtained from trimellitic anhydride chloride and 4,4'-diaminodiphenylmethane.⁴

The synthesis and application of optically active polymers have been given more attention recently because polymers with chiral structures are biologically very important. Most natural

Dedicated to Professor Ahmad Banihashemi on the occasion of his 70th birthday.

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polymers are optically active and have special chemical activities, such as catalytic properties that exist in genes, proteins, and enzymes. Some other applications include (1) chiral media for asymmetric synthesis, (2) chiral stationary phases for the resolution of enantiomers in chromatographic techniques, and (3) chiral liquid crystals in ferroelectrics and nonlinear optical devices.^{5–8} These applications have led to more research for improving different synthetic procedures for optically active polymers. Optically active homopolymers, as well as copolymers, have been prepared and reported in the literature.^{9–19} Recently, we synthesized optically active polymers by different methods.^{20–22}

A triphenyl phosphite (TPP)-activated polycondensation (phosphorylation reaction) technique for the synthesis of polyamides was reported by Yamazaki et al.²³ Recently, this technique has been used for the preparation of high molecular weight polyamides as well as PAIs and is reported in the literature.^{24–26} In a previous study,²⁷ we synthesized a series of novel optically active PAIs containing one group of hexafluoropropylidene and two groups of chiral L-leucines (**2**). The resulting polymers were optically active and had inherent viscosities of 0.09-0.29 dL g⁻¹.

In this article, we report the synthesis and characterization of new optically active PAIs from aromatic diamines and bis(p-amidobenzoic acid)-*N*-trimellitylimido-L-leucine (**5**), a monomer containing preformed imide groups, by the direct polycondensation method.

EXPERIMENTAL

Materials

All chemicals were purchased from Fluka Chemical Co. (Buehs, Switzerland), Aldrich Chemical Co. (Milwaukee, WI), and Riedel-deHaen AG (Seelze, Germany). Trimellitic anhydride (1) was synthesized by the dehydration of 1,2,4-benzene tricarboxylic acid with acetic anhydride in boiling acetic acid. 4,4'-Diaminodiphenylsulfone (**6a**) was purified by recrystallization from water/ethanol (EtOH). 4,4'-Diaminodiphenylether (**6b**), 1,4phenylenediamine (**6c**), 1,3-phenylenediamine (**6d**), 2,4-diamonotoluene (**6e**), and benzidine (4,4'-diaminobiphenyl; **6f**) were purified by sublimation.

Techniques

¹H-NMR (90 MHz) spectra was recorded on a Varian (Varian Associates, Palo Alto, CA) EM-

390 instrument. Tetramethylsilane (TMS) was used as an internal reference. IR spectra were recorded on a Shimadzu 435 IR spectrophotometer (Japan). Spectra of solids were carried out with KBr pellets. Vibrational transition frequencies are reported as wave numbers (cm^{-1}) . Band intensities were assigned as weak (w), medium (m), shoulder (sh), strong (s), and broad (br). Inherent viscosities were measured by a standard procedure with a Cannon-Fenske routine viscometer. Specific rotations were measured with a PerkinElmer 241 polarimeter (Germany). Thermogravimetric analysis (TGA) data for polymers were taken on a Mettler TGA-50 (England) in an air atmosphere at a rate of 10°C/min. Elemental analyses were performed by the Research Institute of the Petroleum Industry (Tehran, Iran).

Monomer Synthesis

N-Trimellitylimido-1-leucine Diacid Chloride (4)

Into a 50-mL, round-bottom flask, 1.00 g (3.27 $\times 10^{-2}$ mol) of *N*-trimellitylimido-L-leucine (3), 10 mL (an excess amount) of thionyl chloride, and a stirring bar were placed. The stirrer was started, and the mixture was refluxed for 1 h. The reaction mixture was stirred at room temperature for 2 h. The thionyl chloride was removed via distillation, 20 mL of *n*-hexane was added, the mixture was heated, *n*-hexane was distilled off, and the solid was collected and dried *in vacuo* to give 1.12 g (100%) of a white solid.

mp: 91–92°C. $[\alpha]_D^{25}$: -48.2° [0.050 g in 10 mL of dimethylformamide (DMF)]. IR (KBr): 2950 (s), 2910 (m), 2860 (m), 1800 (s), 1780 (s), 1750 (s), 1720 (s), 1620 (w), 1470 (m), 1420 (w), 1380 (s), 1340 (m, sh), 1270 (w), 1210 (m), 1180 (m), 1170 (m), 1130(m), 1100 (s), 1075 (m), 1050 (m), 990 (s), 940 (m), 920 (w), 860 (m), 840 (s), 820 (m), 770 (s), 720 (m), 710 (m), 680 (m), 620 (w), 600 (w), 540 (w), 500 (w), 470 cm⁻¹ (w). ELEM. ANAL. Calcd. for C₁₅H₁₃Cl₂NO₄: C, 52.65%; H, 3.83%; N, 4.09%. Found: C, 53.20%; H, 4.10%; N, 4.20%.

Bis(p-amidobenzoic acid)-N-trimellitylimido-Lleucine (5)

Into a 20-mL, round-bottom flask, 0.30 g (8.77 $\times 10^{-4}$ mol) of 4, 5 mL of dry tetrahydrofuran (THF), and a stirring bar were placed. The stirrer was started, the mixture was cooled in an ice bath, and a solution of 0.24 g (1.75×10^{-4} mol) of *p*-aminobenzoic acid in 4 mL of dry THF was added dropwise over a period of 5 min. A yellow





precipitate was formed and became light pink. The reaction mixture was stirred at room temperature overnight, and a white precipitate was

formed. The reaction mixture was concentrated up to 2 mL, and the solid was filtered off and dried *in vacuo* to give 0.453 g (95%) of a white solid.







Scheme 3

Recrystallization from hot methanol (MeOH) gave white crystals.

mp: > 275°C (decompose). $[\alpha]_D^{25}$: +17.6° (0.050 g in 10 mL DMF). IR (KBr): 3450 (m, sh), 3250 (s, br), 3100 (s, br), 2950 (s), 2850 (s, sh), 2650 (m), 2550 (m), 1770 (m), 1720 (s), 1680 (s), 1600 (s), 1520 (s), 1410 (s), 1370 (s), 1310 (s), 1290 (s), 1250 (s), 1170 (s), 1100 (m), 1080 (m), 1010 (w), 990 (w), 920 (w), 850 (m), 770 (m), 720 (m), 690 (w), 650 (w), 600 (w), 540 cm⁻¹ (w). ¹H-NMR [dimethyl sulfoxide- d_6 (DMSO- d_6), TMS, 90 MHz, δ]: 1.00 (m, br, 6H), 1.76 (s, br, 3H), 4.80 (m, br, 1H),

7.40-9.20 (m, 11H), 10.50 (s, 1H), 10.90 (s, 1H), 12.70 ppm (s, br, 2H). ELEM. ANAL. Calcd. for $C_{29}H_{25}N_3O_8$: C, 64.08%; H, 4.64%; N, 7.73%. Found: C, 63.80%; H, 5.10%; N, 7.30%.

Polymerization: Synthesis of Polymer 7c

The PAIs were prepared by direct polycondensation with the following general procedure. As an example, we consider polymer 7c. Into a 5-mL, round-bottom flask fitted with a water-cooled condenser and a magnetic stirrer were placed 0.10 g

Table I Synthesis and Some Physical Properties of PAIs 7a-7f

	Polymer					
Diamine	Polymer	Yield (%)	$\eta_{\rm inh}~(dL/g)^{\rm a}$	$[lpha]_D^{25\mathrm{b}}$	Color ^c	
6a	7a	96	0.23	-0.6	W	
6b	7 b	91	0.51	0.4	OFF-W	
6c	7 c	92	0.52	-0.2	Y	
6d	7d	95	0.45	$-16^{\rm d}$	OFF-W	
6e	7 e	93	0.35	-0.6	OFF-W	
6f	7f	94	0.22	-0.4	Y	

^a Measured at a concentration of 0.5 g/dL in H₂SO₄ at 20°C.

^b Measured at a concentration of 0.5 g/dL in H_2SO_4 at 25 °C. ^c W = white, OFF-W = off-white, PY = pale-yellow, Y = yellow.

 $^{\rm d}$ Measured at a concentration of 1.0 g/dL in $\rm H_2SO_4$ at 25 °C.



Figure 1 IR (KBr) spectrum of PAI 7b.

 $(1.84 \times 10^{-4} \text{ mol})$ of diacid **5**, 0.02 g of calcium chloride, 0.103 mL $(3.93 \times 10^{-4} \text{ mol})$ of TPP, 0.092 mL of pyridine, 0.020 g $(1.84 \times 10^{-4} \text{ mol})$ of diamine **6c**, and 0.3 mL of *N*-methyl-2-pyrolidone (NMP). The stirrer was started, and the mixture was heated under a nitrogen atmosphere with a heating mantel at the refluxing temperature for 1 min. The viscous reaction mixture was cooled and diluted with 2 mL of DMF and was poured into 50 mL of MeOH. The precipitated polymer was collected by filtration and was dried at 100°C for 1 h *in vacuo* to leave 0.1073 g (92.0%) of solid **7c**.

 $\begin{array}{l} {\rm mp:} >& 290\,^{\circ}{\rm C} \ ({\rm dec}). \ IR \ ({\rm KBr}): \ 3300 \ ({\rm s, \ br}), \ 3100 \\ ({\rm m}), \ 3050 \ ({\rm m}), \ 2980 \ ({\rm m}), \ 2950 \ ({\rm m, \ sh}), \ 2880 \ ({\rm m}), \\ 1780 \ ({\rm m}), \ 1718 \ ({\rm s}), \ 1660 \ ({\rm s}), \ 1600 \ ({\rm s}), \ 1510 \ ({\rm s}), \\ 1405 \ ({\rm s}), \ 1375 \ ({\rm s}), \ 1310 \ ({\rm s}), \ 1245 \ ({\rm s}), \ 1180 \ ({\rm s}), \ 1090 \\ ({\rm m}), \ 1015 \ ({\rm w}), \ 990 \ ({\rm w, \ sh}), \ 940 \ ({\rm w}), \ 890 \ ({\rm w}), \ 840 \\ ({\rm m}), \ 760 \ ({\rm m}), \ 725 \ ({\rm m}), \ 690 \ ({\rm w}), \ 520 \ {\rm cm}^{-1} \ ({\rm w}). \end{array}$

The other PAIs, **7a**, **7b**, and **7d–7f**, were prepared with a similar procedure.

Polymer 7a

mp: >305°C (dec). IR (KBr): 3300 (m, br), 3100 (m), 3050 (m), 2950 (m), 2900 (m, br), 2850 (m),

1770 (m), 1715 (s), 1660 (s), 1590 (s), 1500 (s), 1400 (m), 1380 (m), 1310 (s), 1240 (s), 1180 (m), 1150 (s), 1100 (s), 1070 (m, sh), 1010 (w), 940 (w), 890 (w), 830 (m), 755 (m), 720 (m), 690 (m), 560 cm⁻¹ (m).

Polymer 7b

mp: >290°C (dec). IR (KBr): 3300 (m), 3100 (m), 3050 (m), 2950 (m), 2900 (m, sh), 2850 (m), 1770 (m), 1715 (s), 1660 (s), 1600 (s), 1495 (s), 1400 (m), 1370 (m), 1310 (s), 1250 (s), 1210 (s), 1190 (m, br), 1170 (m, br), 1090 (m), 1010 (m), 940 (w), 890 (w), 870 (m), 850 (m), 760 (m), 720 (m), 690 (w), 670 (w), 600 (w), 550 (w), 500 cm⁻¹ (w).

Polymer 7d

 $\begin{array}{l} {\rm mp:} >& 290^{\circ}{\rm C} \ ({\rm dec}). \ {\rm IR} \ ({\rm KBr}): \ 3300 \ ({\rm m, \ br}), \ 3100 \\ ({\rm m}), \ 3050 \ ({\rm m}), \ 2950 \ ({\rm m}), \ 2920 \ ({\rm m}), \ 2880 \ ({\rm m}), \ 1775 \\ ({\rm m}), \ 1720 \ ({\rm s}), \ 1660 \ ({\rm s}), \ 1600 \ ({\rm s}), \ 1520 \ ({\rm s}), \ 1505 \ ({\rm m}), \\ 1480 \ ({\rm m}), \ 1405 \ ({\rm m}), \ 1380 \ ({\rm m}), \ 1320 \ ({\rm s}), \ 1250 \ ({\rm s}), \\ 1180 \ ({\rm m}), \ 1100 \ ({\rm m}), \ 1015 \ ({\rm w}), \ 985 \ ({\rm w}), \ 920 \ ({\rm w}), \ 850 \\ ({\rm m}), \ 760 \ ({\rm m}), \ 720 \ ({\rm m}), \ 690 \ ({\rm w}), \ 660 \ ({\rm w, \ sh}), \ 610 \ ({\rm w}), \\ 570 \ {\rm cm}^{-1} \ ({\rm w}). \end{array}$

			Elemental Analysis (%)				
Polymer	Formula		С	Н	N	Moisture Intake (%) ^a	
7a	$(C_{41}H_{33}N_5O_8S)_n$	Calculated	65.15	4.40	9.27	3.90	
	$(756)_n$	Found	62.70	4.70	8.70		
		Corrected ^b	65.14	4.52	9.04		
7b	$(C_{41}H_{33}N_5O_7)_n$	Calculated	69.58	4.69	9.89	3.30	
	$(708)_n$	Found	66.80	5.00	9.00		
		Corrected ^b	69.00	4.83	9.30		
7c	$(C_{35}H_{29}N_5O_6)_n$	Calculated	68.28	4.75	11.38	4.50	
	$(616)_n$	Found	67.50	4.70	10.30		
		Corrected ^b	70.54	4.49	10.76		
7d	$(C_{35}H_{29}N_5O_6)_n$	Calculated	68.28	4.75	11.38	4.80	
	$(616)_n$	Found	66.00	5.20	10.80		
		Corrected ^b	69.17	4.95	11.32		
7e	$(C_{36}H_{31}N_5O_6)_n$	Calculated	68.67	4.96	11.12	3.90	
	$(630)_n$	Found	66.50	5.30	10.80		
		Corrected ^b	69.09	5.09	11.22		
7f	$(C_{41}H_{33}N_5O_6)_n$	Calculated	71.19	4.81	10.12	3.50	
	$(692)_n$	Found	67.90	4.60	8.10		
		$Corrected^{b}$	70.28	4.44	8.36		

Table II Elemental Analysis of PAIs 7a-7f

^a Moisture intake (%) = $(W - W_0)/W_0 \times 100$, where W is the weight of the polymer sample after standing at room temperature and W_0 is the weight of the polymer sample after drying *in vacuo* at 100°C for 10 h.

^b Corrected value for C and N = found value \times (100 + moisture intake)/100, and Corrected value for H = found value \times (100 - moisture intake)/100.

Polymer 7e

mp: >285°C (dec). IR (KBr): 3300 (m, br), 3100 (m, sh), 2950 (m), 2920 (m), 2850 (m), 1775 (m), 1720 (s), 1660 (s), 1600 (s), 1510 (s), 1495 (s, sh), 1410 (m), 1380 (m), 1320 (m), 1250 (m), 1180 (w), 1100 (w), 1090 (m), 1010 (w), 850 (m), 760 (m), 720 (m), 690 (w), 670 (w), 600 cm⁻¹ (w).

Polymer 7f

mp: >270°C (dec). IR (KBr): 3300 (m), 3070 (m), 3010 (m), 2920 (m), 2900 (m), 2850 (m), 2400 (m), 1770 (m), 1710 (s), 1650 (s), 1590 (s), 1500 (s), 1400 (m), 1375 (m), 1310 (s), 1240 (s), 1200 (s), 1180 (m), 1130 (m), 1090 (m), 1060 (m, sh), 1010 (w), 1000 (w, sh), 920 (m), 850 (m), 815 (m), 760 (m), 720 (w), 690 (w), 660 (w), 575 (w), 510 cm⁻¹ (w).

RESULTS AND DISCUSSION

Monomer Synthesis

3 has been reported in the literature;²⁸ we used a modified procedure²⁹ for its preparation. Thus,

the unsymmetrical diacid compound 3 was synthesized by the condensation reaction of equimolar amounts of 1 and 2 in toluene and in the presence of triethylamine as a base (Scheme 1).

3 was reacted with thionyl chloride, and the diacid chloride **4** was obtained in a high yield (Scheme 2). The chemical structure and purity of optically active **4** were proven with elemental

Table III Solubility of PAIs 7a-7f

Solvent	7a	7b	7c	7d	7 e	7 f
DMAc	+	+	_	+	+	_
DMF	+	+	_	+	+	_
NMP	+	+	_	+	+	_
DMSO	+	+	_	+	+	_
$H_{2}SO_{4}$	+	+	+	+	+	+
MeOH	_	_	_	_	_	_
EtOH	_	_	_	_	_	_
CHCl ₂	_	_	_	_	_	_
CH _a Cl _a	_	_	_	_	_	_
H_2O	—	—	_	—	—	_

Concentration = 5 mg/mL; + = soluble at room temperature; - = insoluble.





Figure 2 TGA/DTG thermograms of PAI 7b in a $\rm N_2$ atmosphere.

analysis and IR spectroscopy techniques. The reaction of 4 with *p*-aminobenzoic acid was performed in dry THF at 0°C. The resulting novel optically active aromatic imide–amide diacid **5** was obtained in a high yield (Scheme 2), and its chemical structure and purity were proven with elemental analysis and IR and ¹H-NMR spectroscopy techniques.

Polymer Synthesis

PAIs **7a**–**7f** were synthesized by the direct solution polycondensation reactions of an equimolar mixture of monomer **5** with six different aromatic diamines (**6a**–**6f**) with TPP and pyridine as condensing agents (Scheme 3).

The synthesis and some physical properties of these novel optically active PAIs are listed in Table I. All the polycondensations readily proceeded in a solution at the refluxing temperature over a period of 1 min. The reaction mixture was diluted with an additional amount of DMF to maintain a degree of agitation because the polymer solutions were too viscous to stir. Tough and stringy precipitates formed when the viscous PAIs solutions were trickled into stirred MeOH. All of the polymers were obtained in quantitative yields with moderate inherent viscosities of 0.22–0.50 dL g⁻¹; they showed optical rotation and so were optically active.

Polymer Characterization

The formation of PAIs was confirmed by IR spectroscopy analysis. As an example, the IR spectrum of PAI **7b** (Fig. 1) showed the characteristic absorptions of amide and imide groups around 3300, 1770, 1715, and 1660 cm⁻¹, which are peculiar to N—H stretching and carbonyl stretching of imide and amide, respectively. All of these PAIs exhibited strong absorption at 1370 and 710–720 cm⁻¹, which showed the presence of the imide



Figure 3 TGA/DTG thermograms of PAI 7c in a N_2 atmosphere.

41

heterocycle in these polymers. Polymer **7a** showed characteristic absorptions at 1310 and 1150 cm^{-1} due to the sulfone moiety (SO₂ stretching). Elemental analysis values of the resulting polymers are listed in Table II. PAIs derived from monomer **5** ranged in color from white to yellow.

The solubilities of the PAIs were tested quantitatively in various solvents and are listed in Table III. All of the PAIs were soluble in organic solvents such as DMF, dimethylacetamide (DMAc), DMSO, NMP, and H_2SO_4 at room temperature and were insoluble in solvents such as chloroform, methylene chloride, MeOH, EtOH, and water.

Thermal Properties

The thermal properties of PAIs (**7b–7d**) were evaluated by means of thermal gravimetric analysis (TGA)/differential thermal analysis (DTG) in a nitrogen atmosphere. All of these polymers



Figure 4 TGA/DTG thermograms of PAI 7d in a $\rm N_2$ atmosphere.

Table IV	Thermal	Behavior	of Aromatic	PAIs
7b–7d				

Polymer	Decomposition Temperature (°C) ^a	Char Yield (%) ^b
7b	217	53.91
7c	271	59.69
7d	258	52.79

 $^{\rm a}$ Temperature at which 5% weight loss was recorded by TGA at heating rate of 10°C/min in $\rm N_2.$

 $^{\rm b}$ Percentage weight of material left undecomposed after TGA analysis at maximum temperature 600°C in $\rm N_2.$

showed similar decomposition behavior (Figs. 2–4). Table IV summarizes the thermal properties of some of the PAIs. The PAIs exhibited good resistance to thermal decomposition between 220and 250°C in nitrogen and began to decompose gradually above that temperature. The temperature of 5% weight loss for all the polymers ranged from 217 to 270°C, and the residual weight for these polymers at 600°C ranged from 52.79 to 59.69% in nitrogen.

CONCLUSIONS

A series of optically active PAIs with inherent viscosities of 0.22–0.52 dL/g were synthesized for the first time by direct polycondensation of the optically active diacid **5** with a preformed imide ring as an enlarged monomer containing one chiral **2** group with some aromatic diamines. These aromatic PAIs showed optical rotation, were readily soluble in various organic solvents, and had moderate thermal stability.

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