

Synthesis and Characterization of Novel Thermally Stable and Optically Active Poly(amide-imide)s Derived from *N,N'*-(4,4'-Diphthaloyl)-bis-L-leucine Diacid and Aromatic Diamines

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ABSTRACT: A new diacid containing optically active functional groups, *N,N'*-(4,4'-diphthaloyl)-bis-L-leucine diacid (3), was synthesized and used in a preparation of a series of poly(amide-imide)s (PAIs) by direct polycondensation with various aromatic diamines in *N*-methyl-2-pyrrolidone (NMP). All polymers derived from diacid (3) were highly organosoluble in the solvents like *N*-methyl-2-pyrrolidinone, *N,N*-dimethylacetamide, *N,N*-dimethylformamide, dimethyl sulfoxide, tetrahydrofuran, γ -butyrolactone, cyclohexanone, and chloroform at room temperature or upon heating. Inherent viscosities of the PAIs were found to range between 0.34 and 0.61 dL g⁻¹. All the PAIs

afforded flexible and tough films. The glass-transition temperatures of these PAIs were recorded between 212 and 237°C by differential scanning calorimetry, and the 10% weight loss temperatures were ranging from 372 to 393°C and 336–372°C under nitrogen and air, respectively. The polyimide films had a tensile strength in the range of 63–88 MPa and a tensile modulus in the range of 1.2–1.7 GPa. Optically active PAIs exhibited specific rotations in the range of –10.58° to –38.70°. © 2007 Wiley Periodicals, Inc. *J Appl Polym Sci* 104: 3096–3102, 2007

Key words: chiral; polyimide; leucine; optical; synthesis

INTRODUCTION

Poly(amide-imide)s (PAIs) are well known as a class of polymers with good compromise between thermal stability and processability.¹ There is a growing interest in PAIs for a variety of applications as they retain good mechanical properties at high temperature when compared with other aromatic thermostable polymers.^{2–4} Recently, the triphenyl phosphite (TPP) and pyridine-promoted polycondensation route to PAIs can offer us the option of introduction of specific functionality between amide or imide groups. Several attempts have been made to modify the PAIs' structure by introducing the functional groups or substituents capable of reducing the chain rigidity and further increasing their tractability and applicability.^{5–10}

The synthesis and application of chiral polymers is of particular interest from the viewpoint of material science and newly considered topics. Chiral polymers have found successful uses in chromatographic

separation of enantiomers, chiral liquid crystals, nonlinear optical devices, optical switches, biomedical devices, etc.^{11–21} A direct and effective way for synthesizing chiral polymers is to introduce chiral elements into the polymer backbone or side chains. The combination of PAIs with chiral elements is of synthetic interest and also may lead to chiral recognition membranes.

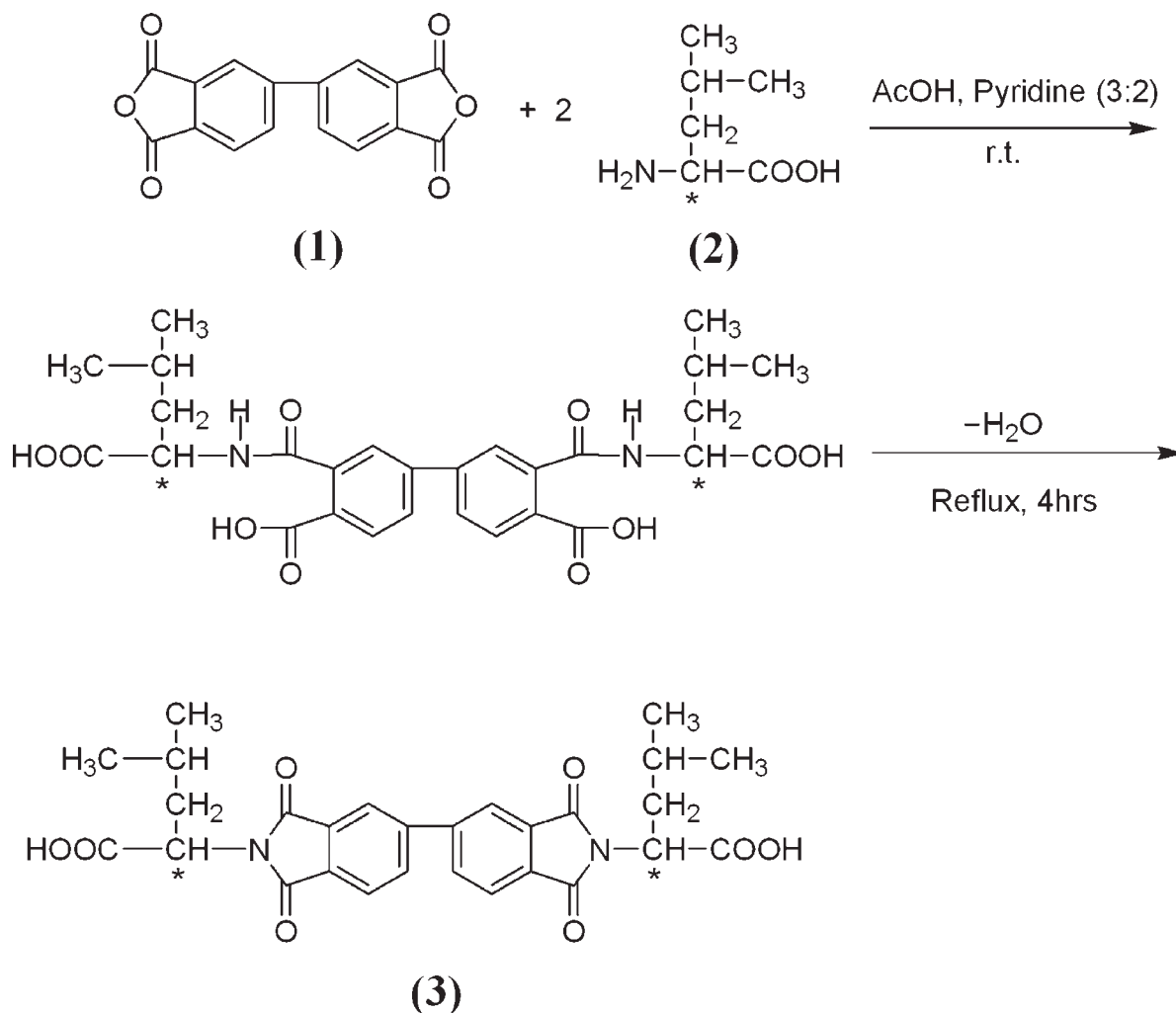
The present investigation deals with the synthesis and characterization of various PAIs derived from a new chiral monomer, *N,N'*-(4,4'-diphthaloyl)-bis-L-leucine diacid, containing biphenyltetracarboxydiimide and L-leucine moieties. Because of the presence of biphenyltetracarboxydiimide group on the PAI backbone, they may have good thermo-oxidative stability. The solubility, optical active properties, thermal properties, and tensile properties of the PAIs are investigated in this article.

EXPERIMENTAL

Materials

The dianhydride 3,3',4,4'-biphenyltetracarboxylic dianhydride [BPDA (1); from CHRISKEV, Leawood, KS] was purified by recrystallization with acetic anhydride. L-leucine [(2); from MP Biomedicals, Eschwege,

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Scheme 1 Synthesis of new monomer *N,N'*-(4,4'-diphthaloyl)-bis-L-leucine diacid (3).

Germany] was used as received. 4,4'-Diaminodiphenylether (**4a**), 2,2-bis[4-(4-aminophenoxy)phenyl]propane (**4b**), 4,4'-bis(4-aminophenoxy)biphenyl (**4c**), bis[4-(4-aminophenoxy)phenyl]sulfone (**4d**), 2,2-bis[4-(4-aminophenoxy)phenyl]hexafluoropropane (**4e**), 1,3-bis(4-amino-phenoxy)benzene (**4f**), and 1,4-bis(4-amino-phenoxy)benzene (**4g**) were purified by recrystallization from ethanol. Reagent-grade calcium chloride was dried under vacuum at 180°C before use. *N*-methyl-2-pyrrolidinone (NMP), *N,N*-dimethylacetamide (DMAc), *N,N*-dimethylformamide (DMF), pyridine, dimethyl sulfoxide (DMSO), and TPP were purified by distillation under reduced pressure over calcium hydride and stored over 4 Å molecular sieves.

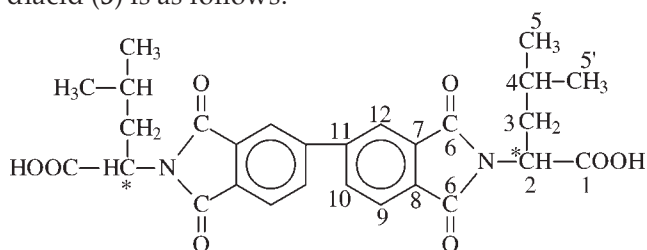
Synthesis of monomer (Scheme 1)

N,N'-(4,4'-diphthaloyl)-bis-L-leucine diacid (3)

The dianhydride 3,3',4,4'-biphenyltetracarboxylic dianhydride (**1**) (2.94 g; 10 mmol), L-leucine (**2**) (2.62 g; 20 mmol), a mixture of acetic acid and pyridine

(3:2 v/v; 50 mL), and a stirring bar were placed in a 250-mL round-bottomed flask. The mixture was stirred overnight at room temperature and was then refluxed for 4 h. The solvent was removed under reduced pressure and the residue was dissolved in 100 mL of cold water, and 5 mL of concentrated HCl were then added. The solution was stirred until a white precipitate was formed, and the precipitate was then filtered off and dried, to give 4.6 g (88%) of Compound **3**. mp 275°C (by DSC), $[\alpha]_D^{25} = -76.9^\circ$ (0.1 g in 10 mL of DMF). FTIR (ATR): 3300–2800 (m, br), 1773 (sh), 1743 (m), 1690 (s, br), 1620 (w), 1468 (w), 1430 (m), 1377 (s, br), 1242 (w), 1209 (m, br), 1159 (m), 1063 (w), 914 (m), 842 (m), 748 (s), 671 (m), 652 (m) cm^{-1} ; The ^1H NMR spectrum of (**3**) in DMSO- d_6 exhibited signals at δ (ppm) = 0.84–0.87 (q, 12H, H_5 , and H_5'), 1.41–1.49 (m, 2H, H_4), 1.83–1.88 and 2.16–2.22 (m, 4H, H_3), 4.79–4.82 (d, 2H, H_2), 7.99–8.01 (d, 2H, H_9), 8.26–8.29 (t, 4H, H_{10} , and H_{12}); The ^{13}C NMR spectrum of (**3**) exhibited signals at δ (ppm) = 20.88 and 23.09 (C_5 and C_5'), 24.72 (C_4), 36.82 (C_3), 50.37 (C_2), 122.59 (C_{12}), 124.20 (C_9),

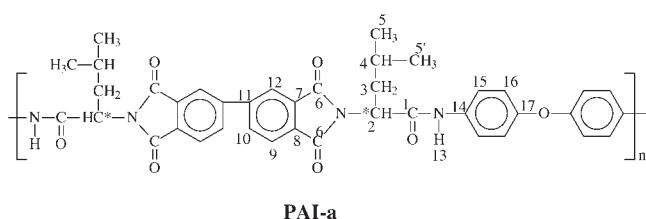
130.94 (C₈), 132.22 (C₇), 134.04 (C₁₀), 144.79 (C₁₁), 167.13 (C₆), 170.87 (C₁); Elemental analysis: Calculated for C₂₈H₂₈N₂O₈: C, 64.61%; H, 5.42%; N, 5.38% and Found: C, 64.58%; H, 5.44%; N, 5.31%. The structure of *N,N'*-(4,4'-diphthaloyl)-bis-L-leucine diacid (**3**) is as follows:



N,N'-(4,4'-diphthaloyl)-bis-L-leucine diacid (**3**)

Synthesis of poly(amide-imide)s (Scheme 2)

A mixture of 0.5205 g (1 mmol) of diacid (**3**), 0.2002 g (1 mmol) of diamine **4a**, 1 mL of triphenyl phosphite, 1 mL of pyridine, 0.6 g of calcium chloride, and 4 mL of NMP was heated with stirring at 120°C for 8 h. After cooling, the reaction mixture was poured into a large amount of methanol with constant stirring, producing a stringy precipitate that was washed thoroughly with methanol and hot water, collected on a filter, and dried at 100°C under vacuum for 12 h. The inherent viscosity of the poly(amide-imide) PAI-a in DMAc was 0.44 dL g⁻¹, measured at a concentration 0.5 g dL⁻¹ at 30°C. The IR spectrum of PAI-a exhibited absorption band at 3290 cm⁻¹ (ν_{N-H}), 1774 cm⁻¹ (imide ν_{C=O} asymmetric), 1709 cm⁻¹ (imide ν_{C=O} symmetric), 1656 cm⁻¹ (amide ν_{C=O}), 1606 cm⁻¹ (ν_{N-H} amide), 1366 cm⁻¹ (ν_{C-N} imide); The ¹H NMR spectrum of PAI-a in DMSO-*d*₆ exhibited signals at δ (ppm) = 0.87–0.91 (q, 12H, H₅, and H_{5'}), 1.45 (s, 2H, H₄), 1.98 and 2.25 (d, 4H, H₃), 4.93 (s, 2H, H₂), 6.90–6.92 (d, 4H, H₁₆), 7.50–7.52 (d, 4H, H₁₅), 8.01–8.02 (d, 2H, H₉), 8.27–8.32 (t, 4H, H₁₀, and H₁₂), 9.95 (s, 2H, H₁₃); The ¹³C NMR spectrum of (**3**) exhibited signals at δ (ppm) = 20.95 and 23.31 (C₅ and C_{5'}), 24.90 (C₄), 37.00 (C₃), 52.30 (C₂), 118.68 (C₁₆), 122.08 (C₁₅), 122.34 (C₁₂), 124.11 (C₉), 130.14 (C₈), 132.70 (C₇), 133.81 (C₁₀), 134.11 (C₁₄), 144.64 (C₁₁), 153.00 (C₁₇), 167.32 (C₁), 167.50 (C₆); Elemental analysis: Calculated for C₄₀H₃₆N₄O₇: C, 70.16%; H, 5.30%; N, 8.18% and Found: C, 68.00%; H, 5.42%; N, 7.83%. The structure of PAI-a is as follows:



PAI-a

All the other polymers were prepared using a similar procedure.

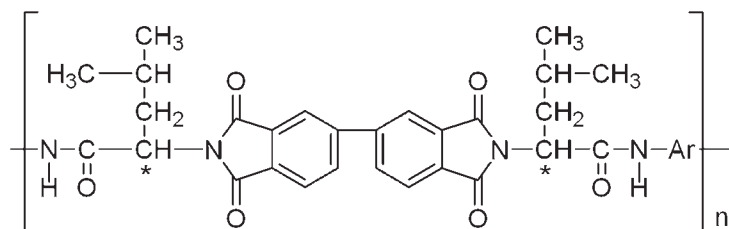
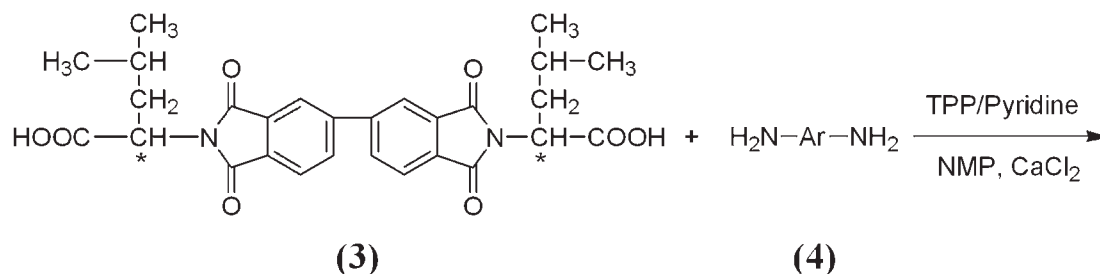
Measurements

IR spectra were recorded in the range 4000–500 cm⁻¹ for the synthesized monomer and polymers in an ATR (attenuated total reflectance) cell (Bio-Rad Digilab FTS-3500). Elemental analysis was made on a Perkin-Elmer 2400 instrument. The inherent viscosities of all PAIs were measured using Ubbelohde viscometer. NMR spectra were recorded using a BRUKER AVANCE 500 NMR (¹H at 500.13 MHz and ¹³C at 125.77 MHz). Optical rotations were measured at 25°C in dimethylformamide (DMF) using a Jasco DIP-360 automatic digital polarimeter with readings to ±0.001°. Weight-average (*M_w*) and number-average molecular weights (*M_n*) were determined by gel permeation chromatography (GPC). Four Waters (Ultrastayragel) columns (300 × 7.7 mm², guard, 10⁵, 10⁴, 10³, and 500 Å in a series) were used for GPC analysis with tetrahydrofuran (THF; 1 mL min⁻¹) as the eluent. The eluents were monitored with a UV detector (JMST Systems, USA, VUV-24) at 254 nm. Polystyrene was used as the standard. Thermogravimetric data were obtained on a Du Pont 2200 under nitrogen or air flowing condition at a rate of 50 cm³ min⁻¹ and a heating rate of 10°C min⁻¹. Differential scanning calorimetric analysis was performed on differential scanning calorimeter (Du Pont 910) at a heating rate of 10°C min⁻¹. Tensile properties were determined from stress-strain curves obtained with an Orientec Tensilon with a load cell of 10 kg. A gauge of 2 cm and a strain rate of 2 cm min⁻¹ were used for this study.

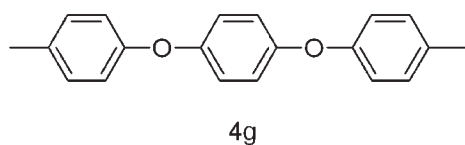
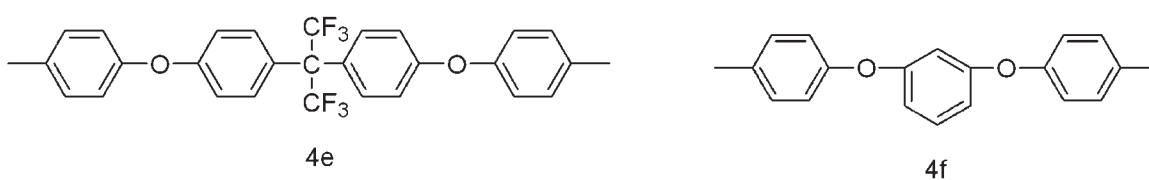
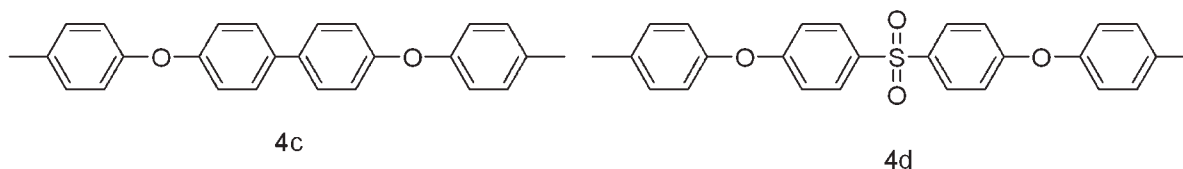
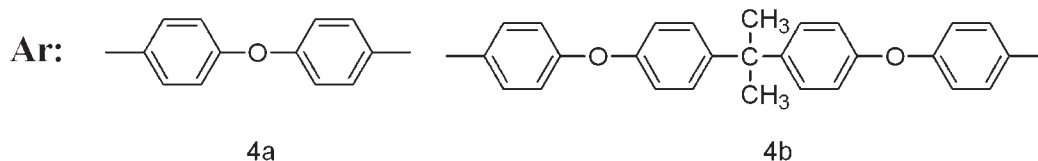
RESULTS AND DISCUSSION

Monomers synthesis

Scheme 1 shows the synthesis of *N,N'*-(4,4'-diphthaloyl)-bis-L-leucine diacid (**3**). The dicarboxylic acid compound containing optically active moieties was synthesized via imidization of two equivalents of optically active L-leucine with one equivalent BPDA in acetic acid and pyridine solvent mixture. Elemental analysis, IR, and NMR spectroscopies confirmed the structure of this compound. The IR spectrum showed absorption bands around between 2800 and 3300 cm⁻¹ (—OH, carboxylic acid), 1773 cm⁻¹ (imide C=O asymmetrical stretching), 1743 cm⁻¹ (imide C=O symmetrical stretching), 1690 cm⁻¹ (carboxylic acid C=O), and 1377 cm⁻¹ (C—N stretching), confirming the presence of imide ring and carboxylic acid groups in the structure. The ¹H NMR spectrum showed the peak at 0.84–4.82 ppm, which are attributable to the leucine protons and the peaks at 7.99–8.29 ppm, which are attributable to the aromatic protons. The ¹³C NMR spectrum was observed that the



PAI



Scheme 2 Preparation of various poly(amide-imide)s (PAI-a–PAI-g).

carbonyl carbons of carboxylic acid and imide groups resonate in the downfield at 170.87 and 167.13 ppm, respectively. Those results clearly confirm that the diacid (3) prepared herein is consistent with the proposed structure.

Polymer synthesis

The poly(amide-imide)s PAI-a–PAI-g were prepared by phosphorylation polycondensation of diacid (3)

with commercial diamines in *N*-methyl-2-pyrrolidone (NMP) containing dissolved CaCl_2 , triphenyl phosphite (TPP), and pyridine as condensing agents (Yamazaki reaction condition, Scheme 2). All the polycondensations proceeded in a homogeneous, transparent, and viscous solution throughout the reaction, and the resulting PAIs were isolated as fibers or powders with almost quantitative yield (91–96%). The optically active PAIs were obtained in quantitative yields with moderate to high inherent viscosities of

TABLE I
Yield, Inherent Viscosity, Specific Rotation, Molecular Weights, and Elemental Analysis of Poly(amide-imide)s

Polymer code	Yield (%)	η_{inh}^a (dL g ⁻¹)	$[\alpha]_D^{25}$ ^b	\overline{M}_n^c (10 ⁴)	\overline{M}_w^c (10 ⁴)	PDI	Elemental analysis (%)			
							C	H	N	
PAI-a	91	0.44	-22.44	- ^d	- ^d	- ^d	Calcd.	70.16	5.30	8.18
							Found	68.00	5.42	7.82
PAI-b	93	0.41	-17.58	2.7	7.4	2.8	Calcd.	73.81	5.63	6.26
							Found	71.75	5.70	5.94
PAI-c	96	0.61	-38.70	1.3	2.9	2.1	Calcd.	73.23	5.20	6.57
							Found	71.16	5.38	6.25
PAI-d	94	0.34	-19.40	0.6	0.9	1.3	Calcd.	68.11	4.84	6.11
							Found	65.78	5.00	5.75
PAI-e	94	0.35	-22.20	1.3	3.8	2.9	Calcd.	65.86	4.42	5.59
							Found	64.03	4.63	5.31
PAI-f	95	0.51	-10.58	- ^d	- ^d	- ^d	Calcd.	71.12	5.19	7.21
							Found	69.27	5.32	6.98
PAI-g	96	0.53	-14.92	1.0	1.7	1.6	Calcd.	71.12	5.19	7.21
							Found	68.84	5.36	6.78

^a Measured in DMAc at a concentration of 0.5 g dL⁻¹ at 30°C.

^b Measured at a concentration of 0.5 g/dL in DMF at 25°C.

^c Measured by GPC in THF; polystyrene was used as standard.

^d The polymer does not dissolve in THF.

0.34–0.61 dL g⁻¹ (Table I), which were measured in DMAc solutions. These polymers exhibited number-average molecular weights (\overline{M}_n) and weight-average molecular weights (\overline{M}_w) in the range of 0.6–2.7 × 10⁴ and 0.9–7.4 × 10⁴, respectively, as measured by GPC, relative to polystyrene standards. The specific rotations of these PAIs were measured in DMF at a concentration of 0.5 g dL⁻¹ at 25°C and summarized in Table I. The specific rotation of the chiral monomer (**3**) is -76.9°. The specific rotations of polymers are in the range of -10.58° to -38.70°. Because of the content ratio of the chiral units in the alternative copolymers, copolymerization of the chiral monomer with various achiral comonomers gives relatively lower values as compared with that of the chiral monomer. For every chiral center, introduction of the achiral comonomer changes its polarity and leading to the variation of the chirality of the chiral center. Conformation of the copolymers may also affect the final appearance of its

chirality. The specific rotation shown in Table I shows the final balance of the factors of polarity and conformation for each copolymer. All of the resulting PAIs show optical rotation and are optically active.

The chain structures were confirmed by elemental analysis, IR, and NMR spectra. The elemental analysis values of these PAIs listed in Table I were in good agreement with the calculated values for the proposed structures. IR spectra of the polymers exhibited characteristic absorption bands around 1780, 1710, and 1375 cm⁻¹ due to the C=O asymmetric, C=O symmetric, and C–N stretches of imide ring. The C–H stretching showed bands between 2900 and 3100 cm⁻¹ and bands of amide N–H appeared around 1600 and 3300 cm⁻¹. The ¹H NMR spectrum exhibits similar patterns to the diacid monomer (**3**) and a new signal at about 10.0 ppm in the ¹H NMR spectrum appeared, which is characteristic of amide N–H group.

TABLE II
Solubility of Poly(amide-imide)s^{a,b}

Polymer code	NMP	DMAc	DMF	DMSO	H ₂ SO ₄	Pyridine	THF	CHCl ₃	γ -Butyrolactone	Cyclohexanone
PAI-a	++	++	++	++	++	++	+-	+-	+	-
PAI-b	++	++	++	++	++	++	++	+	+	+
PAI-c	++	++	++	++	++	++	++	++	+	+
PAI-d	++	++	++	++	++	++	++	+-	+	+
PAI-e	++	++	++	++	++	++	++	+	+	+
PAI-f	++	++	++	++	++	++	+	+	+	+
PAI-g	++	++	++	++	++	++	+	+	+	+

^a Solubility: ++, soluble at room temperature; +, soluble on heating; +-, partially soluble on heating; -, insoluble on heating.

^b Abbreviations: NMP, *N*-methyl-2-pyrrolidinone; DMSO, dimethyl sulfoxide; DMAc, *N,N*-dimethylacetamide; DMF, *N,N*-dimethylformamide; CHCl₃, chloroform; THF, tetrahydrofuran.

Polymer properties

Table II summarizes the solubility of these PAIs. All of the polymers PAI-a–PAI-g exhibited excellent solubility in aprotic polar solvents such as NMP, DMAc, DMF, DMSO, and sulfuric acid at room temperature, as well as in less polar solvents such as γ -butyrolactone, THF, chloroform, and cyclohexanone at room temperature or upon heating. The excellent solubility makes these PAIs potential candidates for practical applications in spin- or dip-coating processes.

DSC and TGA were used to investigate the thermal properties of the PAIs, and some of the thermal behavior data are reported in Table III. In the DSC experiments, there is no melting endotherm in the DSC thermograms. Glass transition temperatures of the PAIs were found to be in the range of 212–237°C. Polyamide PAI-e containing hexafluoropropane group exhibited a lower T_g value than PAI-b, this might be because of its bulky trifluoromethyl groups increased the free volume, and reduced in the density of hydrogen bonding between chains.^{22,23} As shown in Table III, the T_g value of PAI-g containing *p*-phenyl units is higher than PAI-f containing *m*-phenyl units. This might be resulted from higher packing density rigidity of PAI-g the polymer chains.²⁴ The thermal and thermo-oxidative stabilities of these polyimides were evaluated by TGA under both nitrogen and air atmospheres with 10 wt % loss temperature. The decomposition temperatures at a 10% weight loss (T_d^{10}) as well as the anaerobic char yield at 800°C in nitrogen are also summarized in Table III. They reach 372–393°C and 336–372°C in nitrogen and air, respectively. The polymers had char yields above 45% at 800°C under atmosphere of nitrogen. These polymers exhibited a two-step weight loss behavior in air. The first major weight loss occurs at about 350°C, which could be attributed to the decomposition of the leucine segment. The second major weight corresponds to the decomposition of biphenyltetracarboxydiimide groups around

TABLE III
Thermal Properties of Poly(amide-imide)s

Polymer code	T_g (°C) ^a	Decomposition temperature T_d^{10} (°C) ^b		
		In N ₂	In air	Char yield (%) ^c
PAI-a	237	372	336	47
PAI-b	214	385	364	48
PAI-c	229	381	357	48
PAI-d	226	393	372	46
PAI-e	212	382	353	45
PAI-f	212	374	349	45
PAI-g	225	380	342	50

^a Glass transition temperature (T_g) measured by DSC at a heating rate of 10°C min⁻¹.

^b Temperature at which 10% weight loss occurred, as recorded on TGA at a heating rate of 10°C min⁻¹.

^c Residual weight (%) at 800°C in nitrogen.

TABLE IV
Mechanical Properties of Poly(amide-imide)s

Polymer code	Tensile strength (MPa)	Elongation at break (%)	Tensile modulus (GPa)
PAI-a	70	15	1.6
PAI-b	86	11.5	1.5
PAI-c	63	15	1.6
PAI-d	88	8	1.2
PAI-e	72	9.5	1.3
PAI-f	78	16	1.7
PAI-g	73	13	1.7

500°C. All the polymers show similar decomposition behavior. Comparing these results with some literatures, the good thermal stability of PAIs are due to the presence of biphenyltetracarboxydiimide groups.^{20,25,26} The promotion thermal stability also renders advantages in various applications.

All the PAIs possessed outstanding film-forming properties. Highly transparent flexible films were obtained via the casting of polymer solutions in DMAc. The mechanical properties of the polyamide films obtained by solution casting are summarized in Table IV. The polymer films had a tensile strength in the range of 63–88 MPa, elongation at break in the range of 8.0–16.0%, and tensile modulus in the range of 1.2–1.7 GPa. Most of the polymer films exhibited high tensile strength; thus they could be considered as strong materials.

CONCLUSIONS

In the present study, the new diacid *N,N'*-(4,4'-diphthaloyl)-bis-L-leucine diacid (**3**) containing L-leucine and biphtalic moieties was successfully prepared. A series of novel optically active PAIs containing L-leucine and biphenyltetracarboxydiimide substituents have been obtained from diacid (**3**) and different aromatic diamines. These PAIs exhibited excellent solubility in the organic solvents at room temperature. The glass transition temperatures (T_g) of all polymers were more than that of 212°C as measured by DSC, and they also showed good thermal stability and mechanical properties. The specific rotations of these PAIs were in the range of -10.58° to -38.70°. These characteristics indicated that optically active PAIs are promising materials for optical applications.

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