

Polycondensation Reaction of *N,N'*-(4,4'-Oxydipthaloyl)-bis-L-methionine Diacid Chloride with Aromatic Diamines: Synthesis and Properties

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ABSTRACT: Several new optically active poly(amide-imide)s have been synthesized by solution polycondensation of readily available aromatic diamines with diacid chloride containing ether-imide and L-methionine moieties. Three polycondensation techniques were used: low temperature solution polycondensation, short period reflux conditions, and microwave-assisted polycondensation. In all cases, the reactions were carried out using equimolecular amounts of the two monomers, in polar aprotic solvents. The obtained

compounds were characterized by elemental analysis, solubility tests, FTIR, and ^1H NMR spectrometry. Thermal stability of the polymer was studied by thermogravimetric analysis. © 2006 Wiley Periodicals, Inc. *J Appl Polym Sci* 99: 1038–1044, 2006

Key words: poly(amide-imide)s; inherent viscosity; thermal stability; thermogravimetric analysis (TGA); methionine

INTRODUCTION

Optically active polymers^{1–3} have been used in several interesting applications: catalysis for asymmetric induction in organic synthesis,^{1,2,4} chiral separations,⁵ and ferroelectric and nonlinear optical applications.⁶ Achiral monomers can be induced to form polymers with a helical conformation by means of “helix-sense-selective” polymerization with either a chiral initiator or catalyst.³ These include poly(triarylmethylmethacrylates), poly(isocyanides), poly(isocyanates), and poly(chloral). Other polymers, obtained, for example, by cyclopolymerization of diolefins,⁷ diisocyanides,⁸ vinyl ethers,^{9–12} bis(styrenes),^{1,2,11,12} dimethacrylates,^{13–16} and diepoxides,¹⁷ derive their optical activity from chirality in the side chains or main chain. Development of methods for the preparation of chiral polymers remains a challenging field, as radical, anionic, and cationic methods are generally neither stereo- nor enantioselective.^{18,19}

The importance of adequate stores of the amino acid methionine cannot be underestimated. Methionine is particularly important because it supplies sulfur—a mineral—that helps to maintain healthy skin tone, well-conditioned hair, and strong nails. Because body cannot produce this essential amino acid on its own,

one must get it from methionine-rich foods, such as cheddar cheese, eggs, chicken, and beef. Supplements are also a source.

Methionine is thought to keep fat from building up in the liver, and it's often included in liver-detoxifying products called lipotropic combinations. These formulations are believed to accelerate the flow of bile and cell-damaging toxins away from the liver. They commonly contain the B vitamins choline and inositol in addition to methionine.²⁰

Much attention has been paid in recent years to fabrication of chiral separation materials. Those polymers possess high chiral recognition ability as a chiral stationary phase in chromatography technique to resolve a wide range of racemates or chiral media for asymmetric synthesis.²¹ Recently, we synthesized novel optically active polymers by a wide variety of methods.^{22–29} In these polymers, we also used amino acids as chiral inducing agents. These materials are naturally occurring compounds, therefore synthetic polymers based on amino acids are expected to be biodegradable and biocompatible.

This paper presents a polycondensation method for the preparation of novel optically active polymers, which integrates our efforts to obtain new products with optically active properties. Thus, polycondensation reaction of *N,N'*-(4,4'-oxydipthaloyl)-bis-L-methionine diacid chloride with aromatic diamines was performed by three different methods. This is the first report of synthesis and characterization of novel poly(amide-imide)s containing L-methionine amino acid moiety.

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EXPERIMENTAL

Materials

4,4'-Diaminodiphenylmethane (**5b**) and 4,4'-diaminobiphenyl (**5f**) was purified by recrystallization from water. 4,4'-Diaminodiphenylether (**5c**), 1,4-phenylenediamine (**5d**), and 1,3-phenylenediamine (**5e**) were purified by sublimation. 4,4'-oxydiphthalic anhydride was supplied from T.C.I Chemical Co (Japan). *N,N*-Dimethylacetamide (DMAc) was dried over BaO, then distilled in vacuum. The other chemicals were purchased from Fluka Chemical Co. (Buchs, Switzerland), Aldrich Chemical Co. (Milwaukee, WI), and Riedel-deHaen AG (Seelze, Germany), and were used as obtained without further purification.

Apparatus

The apparatus used for the polycondensation was a Samsung microwave oven (2450 MHz, 900 W). All of the polymerization reactions were performed in a hood with strong ventilation.

Measurements

¹H NMR (500 MHz) spectra were recorded on a Bruker, Avance 500 instrument (Rheinstetten, Germany). Proton resonances are designated as singlet (s), doublet (d), doublet of doublet (dd), and multiplet (m). FTIR spectra were recorded on (Jasco-680, Japan) spectrophotometer. Spectra of solids were carried out using KBr pellets. Vibrational transition frequencies are reported in wavenumber (cm⁻¹). Band intensities are assigned as weak (w), medium (m), strong (s), and broad (br). Inherent viscosities were measured by a standard procedure using a Cannon Fenske Routine Viscometer (Mianz, Germany). Specific rotations were measured by a Jasco Polarimeter (Japan). Thermogravimetric analysis (TGA) data for polymers were taken on TGA 7 Perkin-Elmer (Jugeshein, Germany) in nitrogen atmosphere at a rate of 40°C/min. Elemental analysis were performed by Malek-Ashtar University of Technology, Tehran, I.R. Iran.

MONOMER SYNTHESIS

N,N'-(4,4'-Oxydiphthaloyl)-bis-L-methionine diacid (**3**)

Into a 50-mL round-bottomed flask, 1.00 g (3.22×10^{-3} mol) of 4,4'-oxydiphthalic anhydride (**1**), 1.20 g (8.05×10^{-3} mol) of L-methionine (**2**), 30 mL of acetic acid, and a stirring bar were placed. The mixture was stirred at room temperature for 3 h and then was refluxed for 8 h. The solvent was removed under reduced pressure and 5 mL of cold concentrated HCl was added to the residue. A white precipitate was

formed, washed with cold water. It was removed under reduced pressure to give 1.72 g (93.5%) of compound (**3**). mp: 192–194°C, $[\alpha]_D^{25}$: -72.2° (0.050 g in 10 mL DMF); FTIR (KBr): 3344 (m, br), 3064 (m), 2921 (w), 1779 (m), 1757 (s), 1703 (m), 1608 (m), 1474 (w), 1383 (s), 1265 (s), 1233 (w), 1183 (w), 1093 (m), 952 (w), 793 (m), 621 (m) cm⁻¹. ¹H NMR (DMSO-*d*₆, TMS, 500 MHz): δ 2.00–2.10 (s, 6H), 2.40 (m, 4H), 2.50 (m, 4H), 4.90–5.00 (m, 2H), 7.54–7.60 (dd, 2H, $J_1 = 10.0$ Hz, $J_2 = 5.0$ Hz), 7.62–7.64 (d, distorted, 2H), 7.98–7.99 (d, 2H) ppm.

Elem. anal. calcd. for C₂₆H₂₄N₂O₉S₂: C, 54.54%; H, 4.22%; N, 4.89%. Found: C, 54.69%; H, 4.20%; N, 4.99%.

N,N'-(4,4'-Oxydiphthaloyl)-bis-L-methionine diacid chloride (**4**)

Into a 25-mL round-bottomed flask were placed 1.00 g (1.74×10^{-3} mol) of compound **3** and 2.0 mL of thionyl chloride. The mixture was stirred at room temperature for 0.5 h until the suspension mixture was converted to a clear solution. Unreacted thionyl chloride was removed under reduced pressure and was washed with fresh dry ether three times to give 1.04 g (98.0%) of pale yellow solid. mp: 102–104°C, $[\alpha]_D^{25}$: -96.3° (0.050 g in 10 mL DMF); FTIR (NaCl): 2950 (m), 2901 (m), 2850 (m), 1800 (s), 1709 (s), 1460 (m), 1390 (s), 1300 (w), 1170 (s), 1125 (m), 1040 (w), 1020 (w), 960 (w), 910 (w), 860 (w), 750 (m), 640 (w) cm⁻¹.

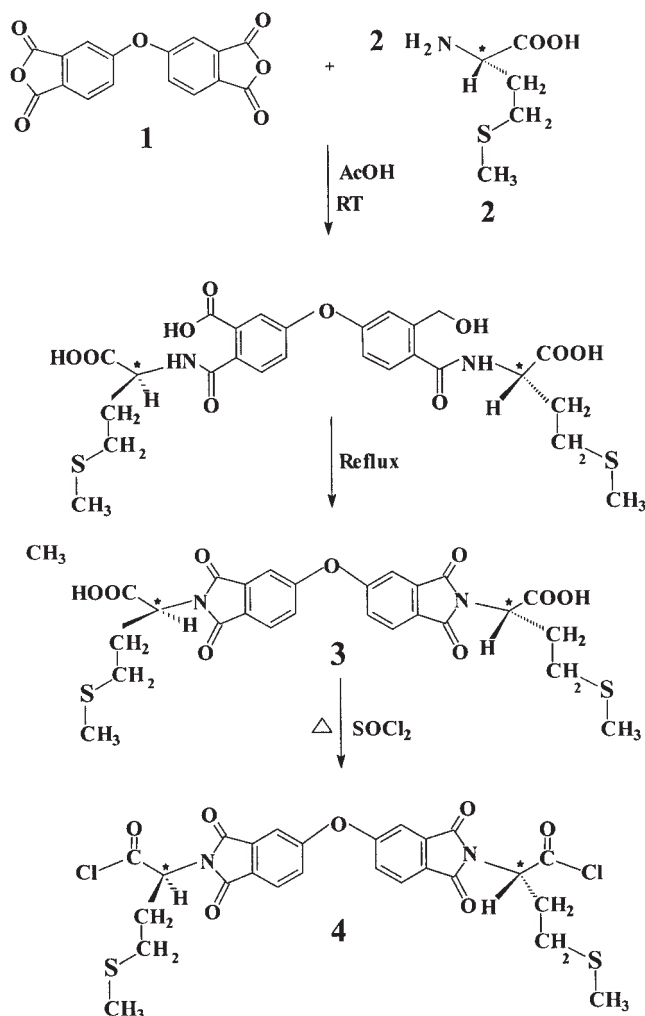
Elem. anal. calcd. for C₂₆H₂₂N₂O₇S₂Cl₂: C, 51.24%; H, 3.63%; N, 4.59%. Found: C, 51.63%; H, 3.64%; N, 4.73%.

POLYMER SYNTHESIS

All the polymers were synthesized by three different methods.

Method I: Low temperature solution polycondensation

Taking polymer **6aI** as an example, the general procedure consisted of adding 0.20 g (3.20×10^{-4} mol) of diacid chloride **4** to a cooled (-5.0°C) and stirred solution of 0.0813 g (3.20×10^{-4} mol) of diamine **5a** in 0.25 mL of 1-methyl-2-pyrrolidone (NMP). After the reagents dissolved completely, 0.05 mL of trimethylsilyl chloride (TMSC) was added and the reaction was allowed to proceed for 2 h under a blanket of nitrogen. Then the temperature was raised to room temperature. The reaction mixture was stirred for 5 h. The viscous solution was poured into 40 mL of methanol and the precipitated solid was filtered off and dried at 80°C for 10 h under vacuum to give 0.194 g (75.5%) of solid polymer **6aI**. FT-IR (KBr): 3345 (m, br), 2966 (m,



Scheme 1 Synthesis of monomer 4.

sh), 2876 (w), 1780 (s), 1720 (s), 1591 (s), 1528 (s), 1467 (m), 1421 (w), 1379 (s), 1252 (m), 1177 (s), 1105 (m), 921 (w), 840 (m), 741 (m), 635 (m) cm^{-1} .

The other poly(amide-imide)s (PAIs) (**6bI–6fI**) were prepared with similar procedures. All of spectra were recorded with KBr pellets.

Polymer 6bI

FTIR (KBr): 3487 (w, br), 3028 (w), 2922 (w), 1781 (m), 1718 (s), 1599 (m), 1511 (m), 1455 (w), 1411 (w), 1382 (s), 1317 (m), 1243 (w), 954 (w), 699 (w), 489 (w) cm^{-1} .

Polymer 6cI

FTIR (KBr): 3486 (w, br), 2965 (w), 1779 (m), 1721 (s), 1605 (m), 1498 (s), 1467 (w), 1377 (s), 1321 (s), 1056 (m), 834 (m), 745 (m), 514 (w) cm^{-1} .

Polymer 6dI

FTIR (KBr): 3359 (w, br), 2966 (w), 1719 (s), 1610 (w), 1515 (w), 1462 (w), 1380 (s), 1223 (w), 1187 (w), 1079

(w), 921 (m), 840 (m), 746 (m), 673 (s), 635 (m), 608 (w) cm^{-1} .

Polymer 6eI

FTIR (KBr): 3352 (w, br), 2965 (m), 1780 (w), 1723 (s), 1602 (m), 1530 (w), 1491 (w), 1378 (m), 1322 (m), 1220 (w), 745 (m), 564 (m) cm^{-1} .

Polymer 6fI

FTIR (KBr): 3487 (m, br), 2927 (m), 1780 (m), 1721 (s), 1601 (m), 1499 (m), 1381 (s), 1242 (w), 1177 (m), 1147 (w), 1104 (m), 741 (m), 671 (m) cm^{-1} .

Method II: High temperature solution polycondensation

Taking polymer **6aII** as an example, into a 5-mL round-bottomed flask, 0.20 g (3.20×10^{-4} mol) of diacid chloride **4**, 0.0813 g (3.20×10^{-4} mol) of diamine **5a**, 0.25 mL of DMAc, and 0.05 mL of TMSC was added. The mixture was refluxed for 1 min. The viscous solution was poured into 40 mL of methanol and the precipitated solid was filtered off and dried at 80°C for 10 h under vacuum to give 0.170 g (66.3%) of polymer **6aIII**.

The other PAIs (**6bII–6fII**) were prepared with similar procedures.

Method III: Polymerization under microwave irradiation

The PAIs were prepared by the following general procedure (using polymer **6aIII** as an example). Into a porcelain dish were placed 0.20 g (3.20×10^{-4} mol) of

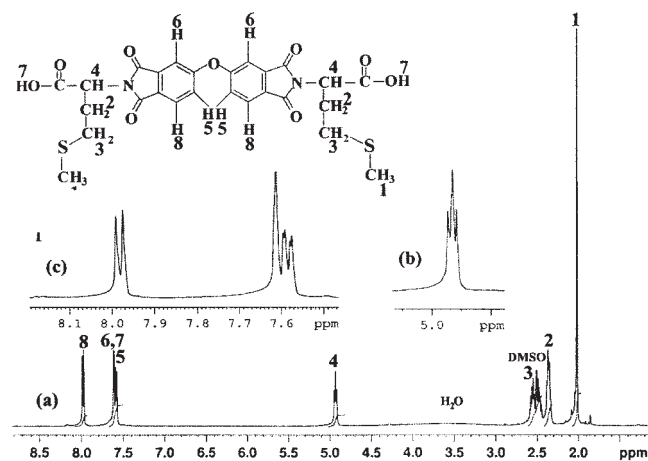
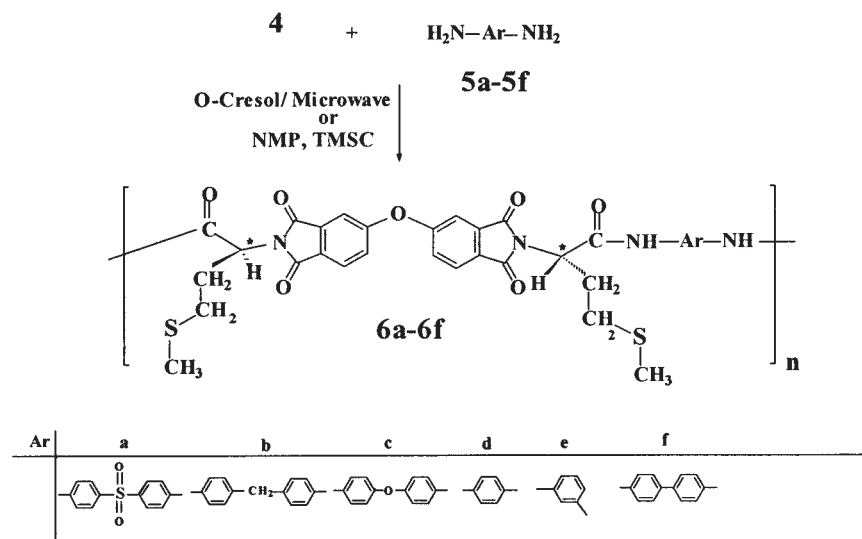


Figure 1 (a) ^1H NMR (500 MHz) spectrum of diacid **3** in $\text{DMSO-}d_6$ at rt. (b) Expanded region for the aliphatic protons ($\delta = 4.7\text{--}5.0$ ppm). (c) Expanded region for the aromatic protons ($\delta = 7.5\text{--}8.1$ ppm).



Scheme 2 Polycondensation reactions of monomer 4 with aromatic diamines.

diacid chloride 4 and 0.0813 g (3.20×10^{-4} mol) of diamine 5a. After the reagents were completely ground, 0.25 mL of *o*-cresol was added as a solvent. The mixture was ground for 5 min. The reaction mixture was irradiated in the microwave oven for 4 min with 100% of the power of microwave apparatus. The resulting product was isolated by adding methanol and triturating, followed by filtration, and was dried at 80°C for 10 h under vacuum to give 0.162 g (63.2%) of solid 6aIII.

RESULTS AND DISCUSSION

Monomer synthesis

The diacid 3 was synthesized by the condensation reaction of dianhydride 1 with 2 moles of L-methionine² in acetic acid. In this reaction, the intermediate amic acid was not isolated and ring closure for the formation of imide ring was performed under refluxing conditions (Scheme 1).

The diacid 3 was converted to diacid chloride derivative 4 by reaction with thionyl chloride. The chem-

ical structure and purity of the compounds 3 and 4 were proved using elemental analysis, FTIR, and ¹H NMR spectroscopic techniques. The FTIR spectrum of compound 3 showed a broad and strong peak at 3500–2500 cm^{-1} , which was assigned to the COOH groups, and two absorption bands at 1780 and 1718 cm^{-1} , which are characteristic peaks for imide rings. The disappearance of strong acidic hydroxyl peak in FTIR spectrum of compound 4 confirmed a complete conversion of diacid 3 to diacid chloride 4.

The ¹H NMR spectrum (500 MHz) of compound 3 is shown in Figure 1. The peaks at 4.80–5.10 ppm are assigned to the protons of the chiral center, which appeared as doublet of doublet by the two-diastrotropic protons. The ¹H NMR spectrum (500 MHz) of diacid chloride is similar to that of diacid derivative.

Polymer synthesis

The polymerization of diacid chloride 4 with aromatic diamines 5a–5f was performed under low temperature (method I), reflux condition (method II), and mi-

TABLE I
Some Physical Properties of PAIs 6aI–6fI Prepared by Method I

Diamine	Code	Yield (%)	Polymer			
			$\bar{\eta}_{\text{inh}}^a$ (dL/g)	$[\alpha]_{589}^{25}$	$[\alpha]_{577}^{25}$	$[\alpha]_{546}^{25}$
5a	6aI	75.5	0.42	–86.1	–37.2	–44.3
5b	6bI	69.3	0.27	–60.3	–44.1	–45.1
5c	6cI	70.1	0.35	–20.3	–25.1	–27.1
5d	6dI	76.1	0.36	–66.1	–71.2	–80.2
5e	6eI	80.1	0.33	–50.9	–59.2	–81.3
5f	6fI	83.2	0.37	–54.3	–71.3	–88.1

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

TABLE II
Some Physical Properties of PAIs 6aII–6fII Prepared by Method II

Diamine	Polymer					
	Code	Yield (%)	$\bar{\eta}_{inh}^a$ (dL/g)	$[\bar{\alpha}]_{589}^{25}$	$[\bar{\alpha}]_{577}^{25}$	$[\bar{\alpha}]_{546}^{25}$
5a	6aII	66.3	0.32	-24.2	-30.1	-37.1
5b	6bII	66.2	0.29	-10.3	-30.6	-49.3
5c	6cII	56.3	0.33	-11.9	-12.1	-20.3
5d	6dII	75.2	0.37	-12.1	-20.1	-44.3
5e	6eII	73.1	0.45	-33.8	-40.3	-48.1
5f	6fII	80.1	0.29	-48.2	-67.3	-70.1

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

crowave-assisted polycondensation reaction (method III) (Scheme 2). In method I, polycondensation reaction was performed in cold NMP solution (-5°C) in the presence of small amount of TMSC. In this method, the polymerization reactions occurred at lower temperature in a period of 2 h. In method II, polycondensation reactions were proceeded rapidly at the reflux temperature of solvent in 1 min. We obtained comparable yields and viscosity of PAIs 6a–6f from methods I and II. The reaction yields and some physical data of the solution polycondensations are listed in Tables I and II.

To compare conventional solution polycondensation reaction methods with microwave-assisted polycondensation, the reactions were also carried out under microwave conditions in the presence of small amount of *o*-cresol that acts as a primary microwave absorber. The reaction mixture was irradiated for 6 min with 100% of radiation power. At higher radiation times, dark products were obtained, and on the other hand, under low radiation times or power, reactions gave low yield and viscosity. The reaction yields and some physical data for PAIs 6aIII–6fIII are listed in Table III.

Polymers characterization

The structures of these polymers were confirmed as PAIs by means of elemental analysis, FTIR, and ¹H

NMR spectroscopy. Elemental analysis data of the resulting polymers are listed in Table IV.

FTIR spectra of all polymers show the characteristic absorption peaks for the imide ring at 1780 and 1718 cm⁻¹ because of the symmetrical and asymmetrical carbonyl stretching vibrations. Bands of amide N–H groups appeared around 3345 cm⁻¹ (hydrogen band) and 1455–1530 cm⁻¹ (amide II band). All of them exhibited strong absorptions at 1381 and 746 cm⁻¹ that show the presence of the imide heterocycle ring in these polymers.

The ¹H NMR spectra of PAI-6aI are shown in Figure 2. The pattern of spectra is similar to those of monomer diacid chloride and corresponding diamine.

Most of the polymers are soluble in aprotic dipolar solvents such as NMP, DMAc, DMF, and DMSO, and even in less polar solvents such as *o*-cresol, *m*-cresol, and acetone. But these polymers are insoluble in solvents such as chloroform, acetonitril, cyclohexane, and water.

Thermal properties

The thermal stability of the polymers 6aI and 6bI was characterized by TGA conducted in nitrogen at a heating rate of 40°C/min. In all cases, the thermal stability is very good. The temperature at which the decomposition begins is never under 300°C. The highest thermal stability was shown by PAIs 6aI. This behavior

TABLE III
Some Physical Properties of PAIs 6aIII–6fIII Prepared by Method III

Diamine	Polymer					
	Code	Yield (%)	$\bar{\eta}_{inh}^a$ (dL/g)	$[\bar{\alpha}]_{589}^{25}$	$[\bar{\alpha}]_{577}^{25}$	$[\bar{\alpha}]_{546}^{25}$
5a	6aIII	63.2	0.35	-39.1	-40.1	-49.3
5b	6bIII	67.1	0.28	-52.1	-67.1	-73.1
5c	6cIII	60.1	0.37	-40.2	-41.8	-44.8
5d	6dIII	70.2	0.28	-56.7	-59.1	-75.1
5e	6eIII	76.3	0.27	-76.1	-67.1	-78.3
5f	6fIII	77.3	0.44	-57.3	-59.1	-74.2

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

TABLE IV
Elemental Analysis of PAIs 6aI–6fI

Polymer	Formula		Elemental analysis (%)			Moisture content ^a (%)
			C	H	N	
6aI	$(C_{38}H_{32}N_4O_9S_3)_n$ (748.8) _n	Calcd	58.16	4.11	7.14	0.29
		Found	58.19	4.19	7.31	
		Corr ^b	58.35	4.17	7.33	
6bI	$(C_{39}H_{34}N_4O_7S_2)_n$ (734.8) _n	Calcd	63.75	4.66	7.62	0.40
		Found	63.81	4.65	7.69	
		Corr ^b	64.00	4.63	7.72	
6cI	$(C_{38}H_{32}N_4O_8S_2)_n$ (736.7) _n	Calcd	61.94	4.38	7.60	0.35
		Found	62.00	4.39	7.62	
		Corr ^b	62.20	4.37	7.64	
6dI	$(C_{32}H_{28}N_4O_7S_2)_n$ (644.6) _n	Calcd	59.62	4.37	8.69	0.25
		Found	59.53	4.38	8.79	
		Corr ^b	59.67	4.36	8.81	
6eI	$(C_{32}H_{28}N_4O_7S_2)_n$ (644.6) _n	Calcd	59.62	4.37	8.69	0.30
		Found	59.63	4.38	8.80	
		Corr ^b	59.80	4.36	8.82	
6fI	$(C_{38}H_{32}N_4O_7S_2)_n$ (720.8) _n	Calcd	63.32	4.47	7.77	0.20
		Found	63.32	4.49	7.96	
		Corr ^b	63.44	4.48	7.97	

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

^b Corrected value for C and N = Found value $\times (100 + \text{moisture content})/100$ and Corrected value for H = Found value $\times (100 - \text{moisture content})/100$.

could be a consequence of the sulfone moieties present in the main chain of polymer. Typical TGA curves of representative polymers are shown in Figure 3. The temperatures of 5 and 10% weight loss together with char yield at 600°C for PAIs 6aI and 6bI have been calculated from their thermograms. From these data it is clear that the resulting polymers are thermally sta-

ble. The thermo analyses data of PAIs 6aI and 6bI are summarized in Table V.

CONCLUSIONS

This is the first report of synthesis and properties of novel PAIs containing oxydiphthaloyl-bis-L-methio-

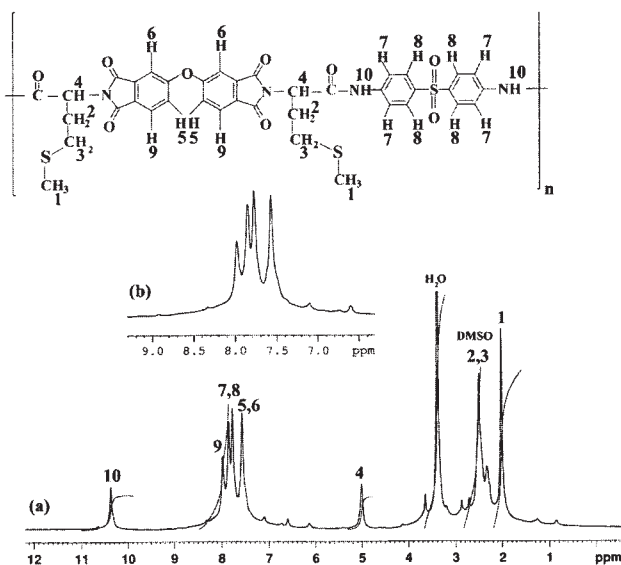


Figure 2 (a) ¹H NMR (500 MHz) Spectrum of PAI-6aI in DMSO-*d*₆ at rt. (b) Expanded region for the aromatic protons ($\delta = 6.5\text{--}9.0$ ppm).

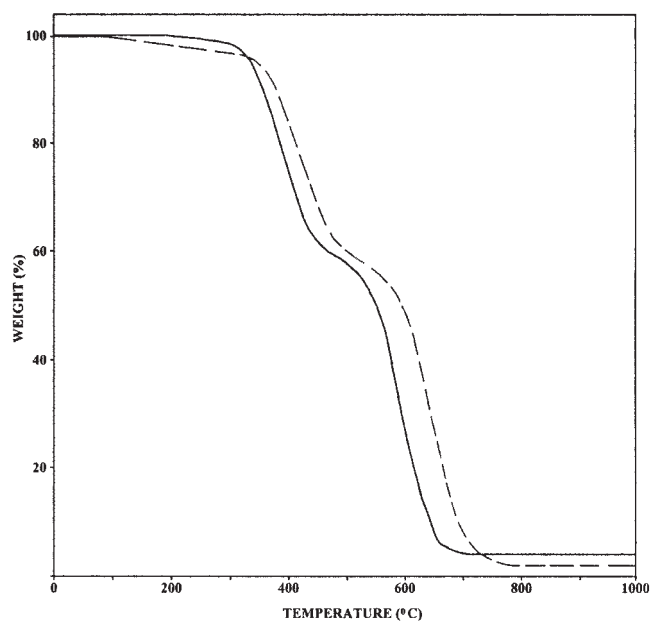


Figure 3 TGA of PAI-6aI (---) and PAI-6bI (—) with a heating rate of 40°C/min and chart speed 10 mm/min in nitrogen atmosphere.

TABLE V
Thermal properties of PAIs

Polymer	T_0^a (°C)	T_5^b (°C)	T_{10}^c (°C)	Char yield ^d (%)
6aI	353	384	411	52
6bI	321	358	398	32

^a The initial decomposition temperature by TGA at a heating rate of 40°C/min in N₂.

^b Temperature at which 5% weight loss was recorded by TGA at a heating rate of 40°C/min in N₂.

^c Temperature at which 10% weight loss was recorded by TGA at a heating rate of 40°C/min in N₂.

^d Percentage weight of material left undecomposed after TGA analysis at a maximum temperature 600°C in N₂.

nine moieties. Thus, solution polycondensation reaction of optically active diacid chloride **4** with several different aromatic diamines was performed by three different methods. The resulting PAIs are thermally stable and are readily soluble in common organic solvents. The presented syntheses must be considered as a proof that chiral polymers could be obtained from the synthesized chiral diacid chloride and diamine monomers. The synthetic polymers are expected to have potential as packing materials in chromatography technique. Furthermore, the resulting polymers due to the presence of amino acid moieties could have biodegradability and biocompatibility properties, so could be environmentally friendly.

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