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ORIGINAL PAPER

New optically active poly(amide-imide)s from *N,N′*-(bicyclo[2,2,2]oct-7-ene-2,3,5,6-tetracarboxylic) bis-L-phenyl alanine and aromatic diamines: synthesis and characterization

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Abstract In this work, new optically active poly(amide-imide)s (PAIs) having bicyclo[2,2,2]oct-7-ene-2,3,5,6-tetracarboxylic diimide groups were prepared by the reaction of *N,N′*-(bicyclo[2,2,2]oct-7-ene-2,3,5,6-tetracarboxylic)bis-L-phenyl alanine (**4**) as a diacid monomer with various readily available aromatic diamines. Triphenyl phosphite (TPP)/pyridine (Py) in the presence of calcium chloride ($CaCl_2$) and *N*-methyl-2-pyrrolidone (NMP) were successfully applied for direct polycondensation. The diacid (**4**) was synthesized by the condensation reaction of bicyclo[2,2,2]oct-7-ene-2,3,5,6-tetracarboxylic dianhydride (**1**) with L-phenyl alanine (**2**) in acetic acid solution. The resulting new polymers were obtained in good yields, inherent viscosities ranging between 0.29 and 0.48 dLg^{-1} and were characterized with elemental analysis, FT-IR, ^{1}H-NMR spectroscopy, specific rotation, and thermal gravimetric analysis (TGA, DTG) techniques. Thermogravimetric analysis indicated that the residual weight percent of polymers at 600 °C was between 53.80 and 56.21%, which show these are moderately thermally stable. In addition because of existence of chiral center and optical activity of these polymers, they have potential to be used as chiral stationary phase in chromatography technique for the separation of racemic mixtures.

Keywords Poly(amide-imide)s · Thermal gravimetric analysis (TGA) · Optically active polymers

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Introduction

The development of heat resistant high performance polymers in the past decades has been quite dramatic and has drawn the attention of many polymer scientists all over the world. Wholly aromatic polymers such as polyimides and polyamides have already been noted for their high temperature resistance and excellent physico mechanical properties. They are difficult to process due to their insolubility in organic solvents and infusibility [1–6]. Considerable effort has been made to improve their processing properties by structural modification. One such method is the synthesis of copolymers. Aromatic poly(amide-imide)s are high performance materials with good compromise between stability and processability when compared with polyamides and polyimides of analogous structure [7–14].

Polymers with optically active properties have found interesting applications such as chiral phase for enantiomeric separation in chromatography methods or chiral media for asymmetric synthesis [15–20]. In polycondensation reactions, we use amino acids as chiral inducting agents. In addition, the synthesis of polymers containing amino acid fragments is a subject of much interest [21–23]. Since amino acids are naturally occurring compounds, synthetic poly(α-amino acid)s and their copolymers are expected to be biodegradable, biocompatible, and nontoxic [24]. Moreover, amino acid-based chiral polymers are likely to possess crystallinity with the ability to form higher ordered structures that exhibit enhanced solubility characteristics [25, 26]. Recently, optically active polymers by the reaction of an optically active monomer with several diamines via solution polymerization have been synthesized [27–33].

In this article, we describe synthesis and characterization a series of novel poly(amide-imide)s (**6a**–**e**) containing rigid segments bicyclo[2,2,2]oct-7-ene-2,3,5,6-tetracarboxylic diimide in the main chain from the direct polycondensation reaction of *N,N*′-(bicyclo[2,2,2]oct-7-ene-2,3,5,6-tetracarboxylic)bis-L-phenyl alanine (**4**) with five aromatic diamines such as 4,4′-diamino diphenyl ether (**5a**), 4,4′-diamino diphenyl sulfone (**5b**), 3,3′-diamino diphenyl sulfone (**5c**), 1,4-diamino benzene (**5d**), 1,5-diamino naphthalene (**5e**), in a medium consisting of *N*-methyl-2-pyrrolidone (NMP), triphenyl phosphite (TPP), calcium chloride ($CaCl_2$) and pyridine. Dianhydride **1** was used for synthesis of polyimides previously [34], but in this article, we used from dianhydride **1** for synthesis of diacid monomer **4** and were synthesized optically active poly(amide-imide)s containing bicyclo[2,2,2]oct-7-ene-2,3,5,6-tetracarboxylic diimide in the main chain. Also the direct polycondensation of aromatic diamines with aromatic dicarboxylic acids in NMP solution containing dissolved $CaCl_2$ using triphenyl phosphite (TPP) and pyridine as condensing agents is known to be a convenient method for the preparation of aromatic polyamides on the laboratory scale [35]. Results showed these new polymers with amide and imide groups in the main chain have better solubility in organic solvents when compared to aromatic polyamides and polyimide.

Experimental

Materials

All chemicals were purchased from Fluka Chemical Co. (Switzerland), Aldrich Chemical Co. (Milwaukee), Merck Chemical Co. (Germany) and Acros Organics N.V/S.A (Belgium).

Instruments

^{1}H-NMR and ^{13}C-NMR spectra were recorded on a Bruker 300 MHz instrument (Germany). Fourier transform infrared (FT-IR) spectra were recorded on Galaxy series FT-IR 5000 spectrophotometer (England). Spectra of solid were performed by using KBr pellets. Vibration transition frequencies were reported in wave number (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 by using a Technico Regd Trad Merk Viscometer. Specific rotations were measured by an A-Kruss polarimeter. Thermal gravimetric analysis (TGA and DTG) data for polymers were taken on a Mettler TA4000 System under N_2 atmosphere at rate of 10 °C/min. Elemental analyses were performed were performed by Vario EL equipment by Arak University.

Synthesis of monomer

*N,N′-(bicyclo[2,2,2]oct-7-ene-2,3,5,6-tetracarboxylic)bis-L-phenyl alanine **4***

Into a 250 mL round-bottomed flask were placed 1.25 g (0.005 mol) of bicyclo[2.2.2]oct-7-ene-2,3,5,6-tetracarboxylic dianhydride **1**, 1.65 g (0.01 mol) L-phenyl alanine **2** and 100 mL concentrate acetic acid. The mixture was stirred at room temperature for 8 h and then refluxed for 5 h. The solvent was removed under reduced pressure, and 5 mL cold concentrated HCl was added to the residue. A white precipitate formed was washed with cold water, and dried under reduced pressure to give 2.48 g (91%) of compound **4**. M.p. = 246–248 °C, $[\alpha]_D^{25} = 140^{\circ}$ (0.05 g in 10 mL DMF). FT-IR (KBr): 2,600–3,500 (m, br), 1,776 (w), 1,703 (s, br), 1,498 (w), 1,398 (w), 1,394 (s), 1,234 (m, br), 1,174 (s), 933 (w), 698 (m). ^{1}H NMR (DMSO-d$_6$, δ ppm): 13.15 (s, br, 2H), 7.21–7.23 (q, 6H), 7.02–7.03 (t, 4H), 4.85–4.91 (dd, br, 2H, $J = 6, 6$ Hz), 3.25–3.31 (dd, 2H, $J = 12, 3$ Hz), 3.10–3.12 (d, 2H, $J = 6$ Hz), 3.01–3.05 (d, 4H, $J = 12$ Hz), 2.92–2.94 (d, 2H, $J = 6$ Hz). ^{13}C NMR (DMSO-d$_6$, δ ppm): 176.69, 169.94, 132.02, 129.32, 128.53, 126.98, 53.07, 42.24, 42.11, 33.45, 33.24. Elemental analysis: calcd for $C_{30}H_{26}N_2O_8$: C, 66.41; H, 4.83; N, 5.16; found: C, 66.41; H, 4.82; N, 5.10.

Polymerization

As a typical example, PAI **6b** was prepared as follows: into a 50 mL round-bottom flask, 0.3536 g (0.652 mmol) diacid **4**, 0.1618 g (0.652 mmol) of 4,4′-diamino diphenyl sulfone **5b**, 0.1 g of calcium chloride, 1 mL of NMP, 0.8 mL of triphenyl phophite, and 0.3 mL of pyridine, and a stirring bar were placed. The mixture was stirred at room temperature for 2 h and then was heated with stirring at 120–130 °C for 8 h. At the end of the reaction, for quench growth polymer chain, the polymer solution was slowly trickled in to stirred methanol, giving rise to a stringy precipitate, which was washed thoroughly with hot methanol, collectedly filtration and dried at 80 °C for 12 h under vacuum to leave 0.42 g (85%) of solid polymer **6b**.

Result and discussion

Monomer synthesis

N,N′-(bicyclo[2,2,2]oct-7-ene-2,3,5,6-tetracarboxylic)bis-L-phenyl alanine **4** was prepared in one step as shown in Scheme 1. The asymmetric diacid **4** was synthesized by the condensation reaction of two equimolars of L-phenyl alanine **2** with one equimolar of dianhydride **1** in acetic acid solution. Dissolving the residue in cold water gives a gummy like solid that breaks by adding concentrated HCl and gave a white color solid. The chemical structure and purities of diacid **4** were also analyzed by elemental analysis, ^{1}H-NMR, ^{13}C-NMR, HMQC, and FTIR spectroscopy. The measured results in elemental analyses closely corresponded to the calculated ones, demonstrating that the expected compound was obtained.

Figure 1 displays FTIR spectrum of diacid **4**. Peaks appearing at 2,400–3,400 cm^{-1} (acid O–H stretching), 1,776 and 1,703 cm^{-1} (asymmetric and symmetric imide stretching), 1,394 and 752 cm^{-1} (imide characteristic ring vibration) confirmed the presence of imide ring and carboxylic groups in this compound.

The ^{1}H-NMR spectrum of diacid **4** is showed in Figs. 2 and 3. The protons related to O–H carboxylic acid groups appeared in region of 13.15 ppm. Peak in 4.89–4.91 ppm as a doublet which were assigned to the CH(b) protons, which is a chiral center and peaks between 7.02–7.23 ppm were assigned to aromatic protons H(d, e, f).

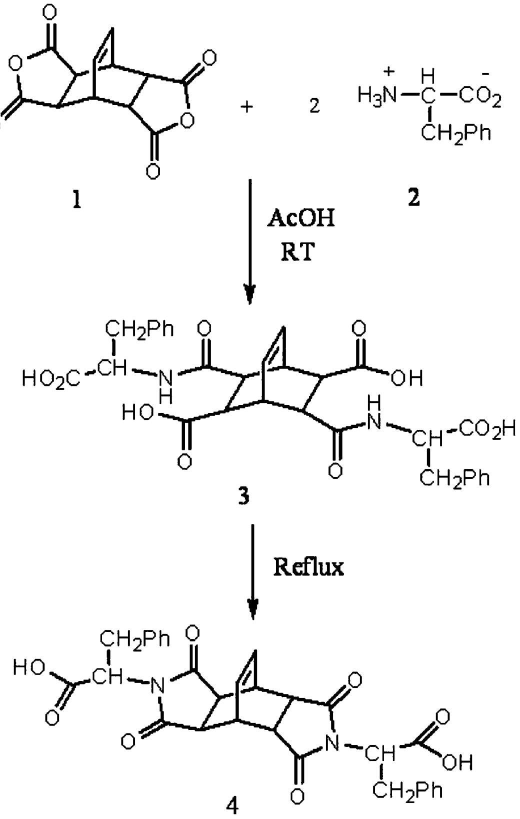
The ^{13}C-NMR spectrum of diacid **4** showed 11 signals, including C(a) and C(b) in imide rings and carboxylic acid carbon atoms, respectively, C(c) related to carbon atoms olefin and C(e) relevant to chiral carbon atoms (Fig. 4). Also Fig. 5 displays HMQC spectrum of diacid **4** that these peaks in the HMQC and in ^{13}C-NMR spectra along with elemental analyses data confirmed the proposed structure of compound **4**.

Polymer synthesis

Poly(amide-imide)s **6a**–**e** were synthesized by solution polycondensation reaction of an equimolar mixture of monomer **4** with five different derivatives of aromatic diamines **5a**–**e**, as shown in Scheme 2.

Synthesis and some physical properties of PAIs **6a**–**e** are summarized in Table 1. These polymers have inherent viscosities rang between 0.29 and 0.48 dL g^{-1}. Also

Scheme 1 Synthesis of diacid **4**



PAIs derived from monomer **4** have rang of color between cream to white. All of the resulting polymers show optical rotation and are optically active.

Structural characterization of polymer

The structures of these polymers were confirmed as PAIs by mean of FT-IR spectroscopy and elemental analyses. The representative FT-IR spectrum of PAI **6e** was shown in Fig. 6. The polymer exhibited characteristic absorption bands at 1,707–1,774 cm^{-1} for the imide ring (asymmetric and symmetric C=O stretching vibration), 1,377 cm^{-1} (C–N stretching vibration). The absorption bands of amide groups appeared at 3,306 cm^{-1} (N–H stretching) (Table 2).

The ^{1}H-NMR spectrums of polymers **6b** showed some peaks that confirm the chemical structure (Fig. 7). The aromatic protons related to diphenyl sulfone appeared in the region of 7.58–7.83 ppm and the peak in the region of 10.30 ppm is assigned for NH of amide groups in polymer backbone.

The elemental analyses of the resulting PAIs **6a**–**e** were in good agreement with the calculated values for the proposed structure (Table 3).

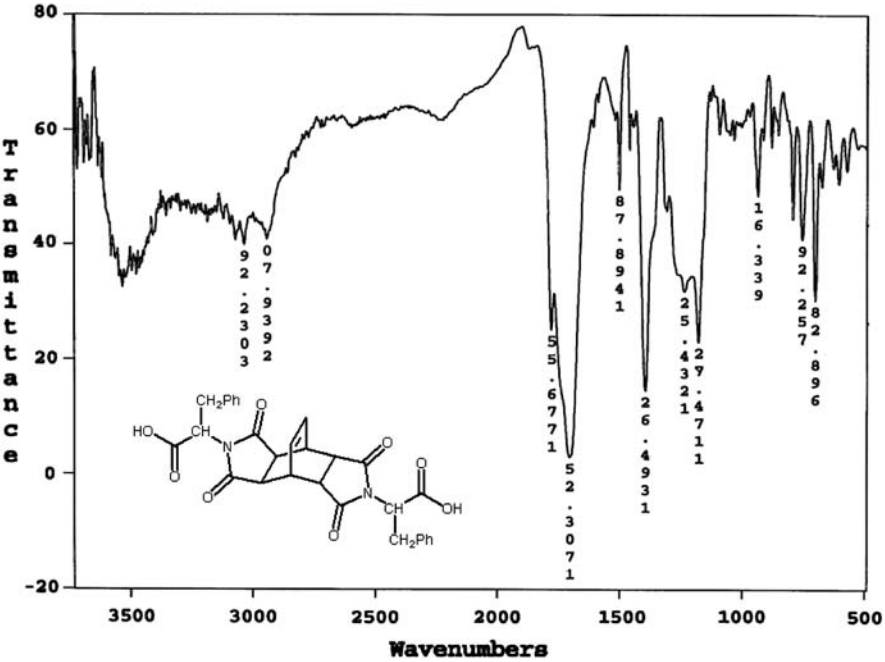


Fig. 1 FT-IR (KBr) spectrum of diacid **4**

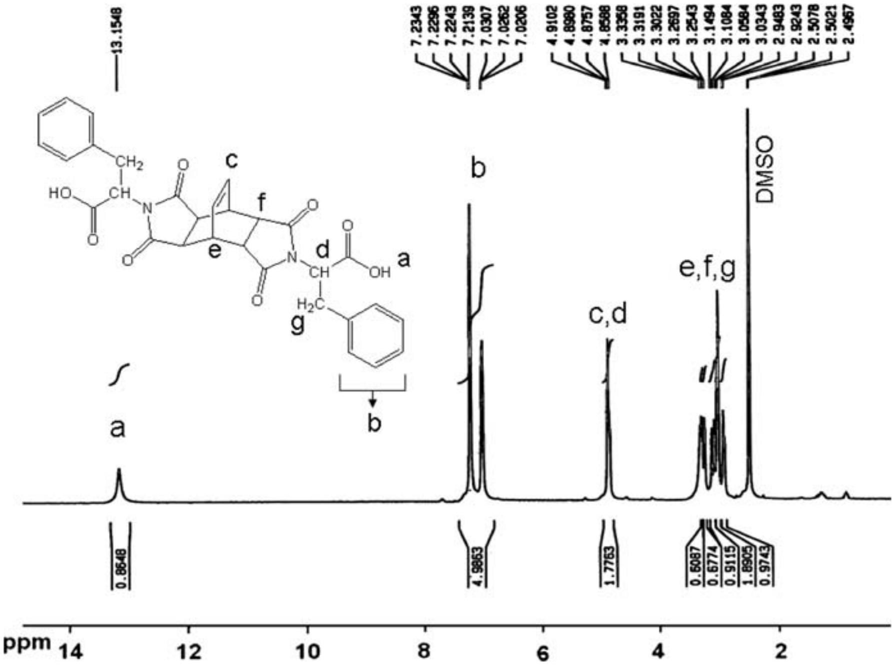


Fig. 2 ^{1}H-NMR spectrum of diacid **4**

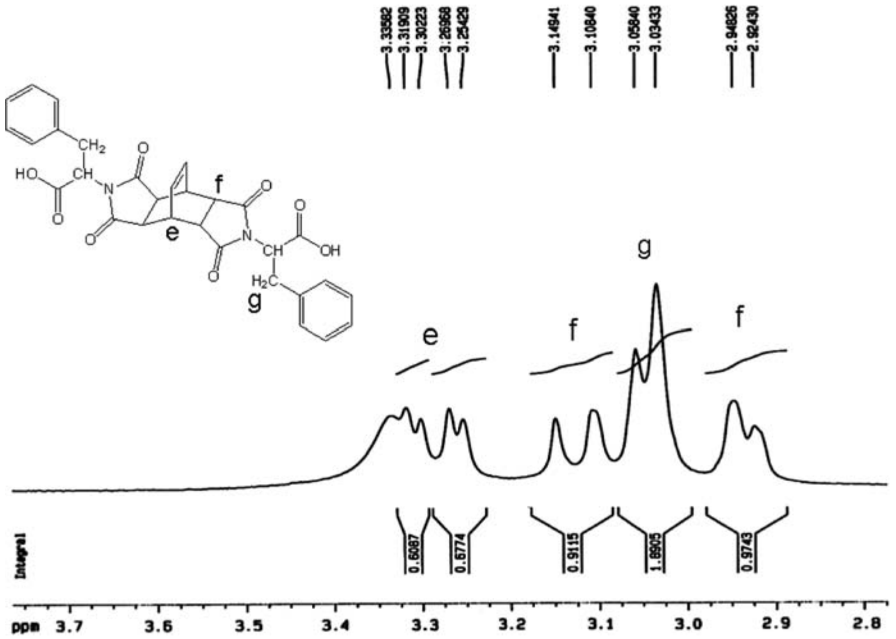


Fig. 3 ^{1}H-NMR spectrum of diacid **4** (expanded)

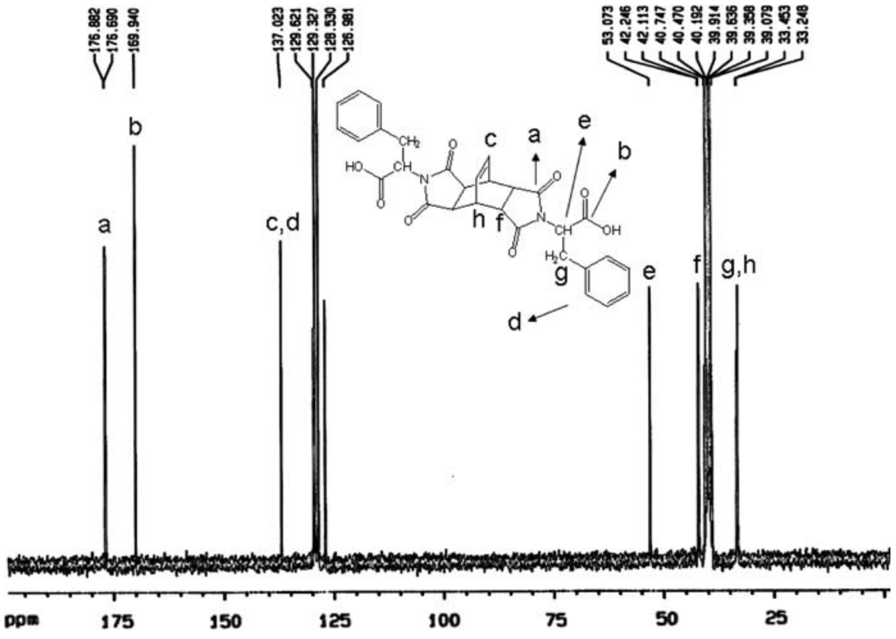


Fig. 4 ^{13}C-NMR spectrum of diacid **4**

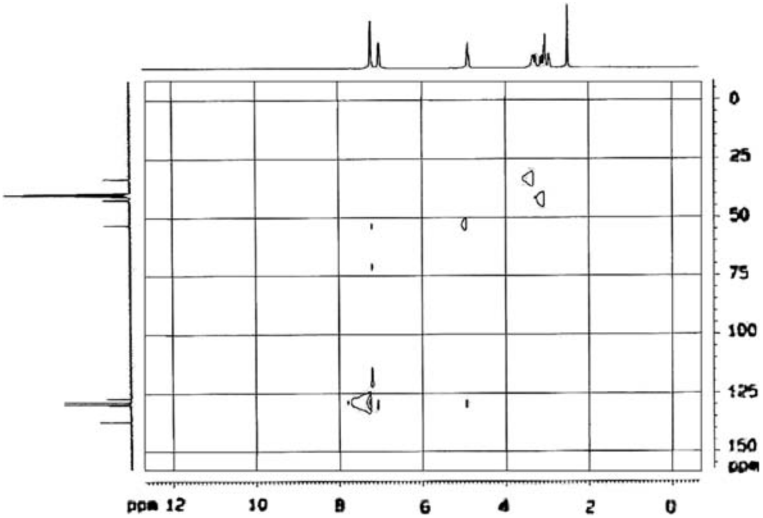
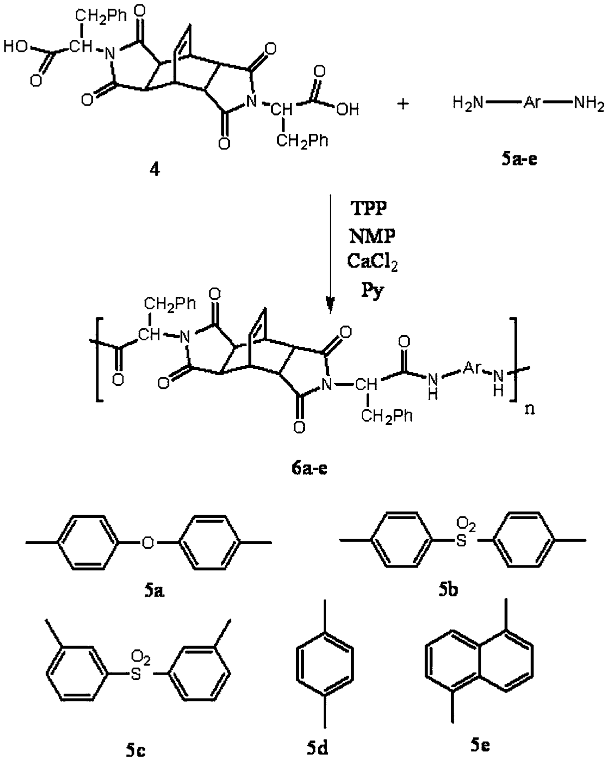


Fig. 5 HMQC spectrum of diacid **4**



Scheme 2 Synthesis of PAIs **6a–e**

Table 1 Some physical properties of PAIs **6a–e**

Diamine	Polymer			
	Polymer code	Yield (%)	η (dL g^{-1})[a]	$[\alpha]_D^{25}$[a]
5a	**6a**	95	0.42	−180
5b	**6b**	85	0.35	−110
5c	**6c**	83	0.29	−170
5d	**6d**	90	0.40	+130
5e	**6e**	93	0.48	−180

[a] Measured at a concentration of 0.5 g/dL in DMF at 25 °C

Table 2 FT-IR characterization of PAIs **6a–e**

Polymer	Spectral data
5a	FT-IR peaks (cm^{-1}): 3,348 (w), 1,774 (w), 1,710 (s, br), 1,604 (w), 1,498 (m, sh), 1,383 (m), 1309 (w), 1,222 (m), 1,170 (m), 1,012 (w), 937 (w), 833 (w), 744 (w), 698 (w).
5b	FT-IR peaks (cm^{-1}): 3,061 (w), 3,034 (w), 2,933 (w), 1,774 (w), 1,709 (s, sh), 1,591 (m), 1,525 (m), 1,496 (m), 1,381 (m), 1,315 (m), 1,249 (m), 1,174 (m), 1,149 (m), 1,105 (m), 1,072 (w), 837 (w), 790 (w), 744 (w), 696 (w), 578 (w).
5c	FT-IR peaks (cm^{-1}): 3,315 (w), 2,901 (w), 1,776 (w), 1,710 (s), 1,664 (m), 1,595 (m), 1,537 (m), 1,479 (m), 1,383 (m), 1,303 (m), 1,244 (m), 1,151 (m), 1,097 (w), 997 (w), 935 (w), 790 (w), 746 (w), 696 (w), 611 (w).
5d	FT-IR peaks (cm^{-1}): 3,306 (w), 2,930 (w), 1,774 (w), 1,707 (s, sh), 1,533 (m), 1,494 (m), 1377 (m), 1,234 (m), 1,170 (m), 1,030 (w), 931 (w), 844 (w), 785 (w), 696 (m), 596 (w).
5e	FT-IR peaks (cm^{-1}): 3,306 (w), 1,774 (w), 1,707 (s, sh), 1,610 (w), 1,533 (m), 1,494 (w), 1,377 (m), 1,234 (w), 1,170 (m), 1,030 (w), 931 (w), 785 (m), 696 (m).

The solubility of PAIs **6a–e** was investigated as 0.01 g of polymeric sample in 2 mL solvent. All of the polymer are soluble in organic solvents such as DMF, DMSO and are insoluble in solvents such as chloroform, methylene chloride, methanol, ethanol, and water (Table 4).

Thermal properties

The thermal properties of PAIs **6a** and **6e** were investigated by TGA and DTG in a nitrogen atmosphere at a heating rate of 10 °C/min.

All of these polymers showed similar decomposition behavior. Initial decomposition temperature, 5 and 10% weight loss temperature (T_5, T_{10}), and char yields are summarized in Table 5.

These polymers exhibited good resistance to thermal decomposition up to 243 °C in nitrogen and began to decompose gradually above that temperature. Them temperature of 5% weight loss for all the polymers ranged from 203 to 243 °C and the residual weight at 600 °C ranged from 33.62 to 56.21% in nitrogen. Figure 8 displays TGA and DTG thermogram of PAI **6e**. TGA and DTG results of these

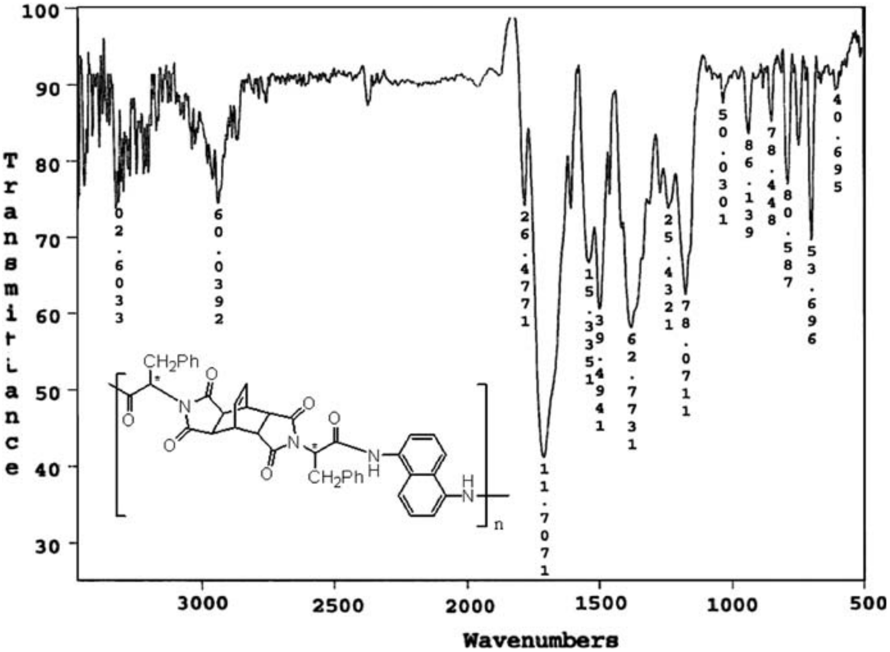


Fig. 6 FT-IR spectrum of PAI **6e**

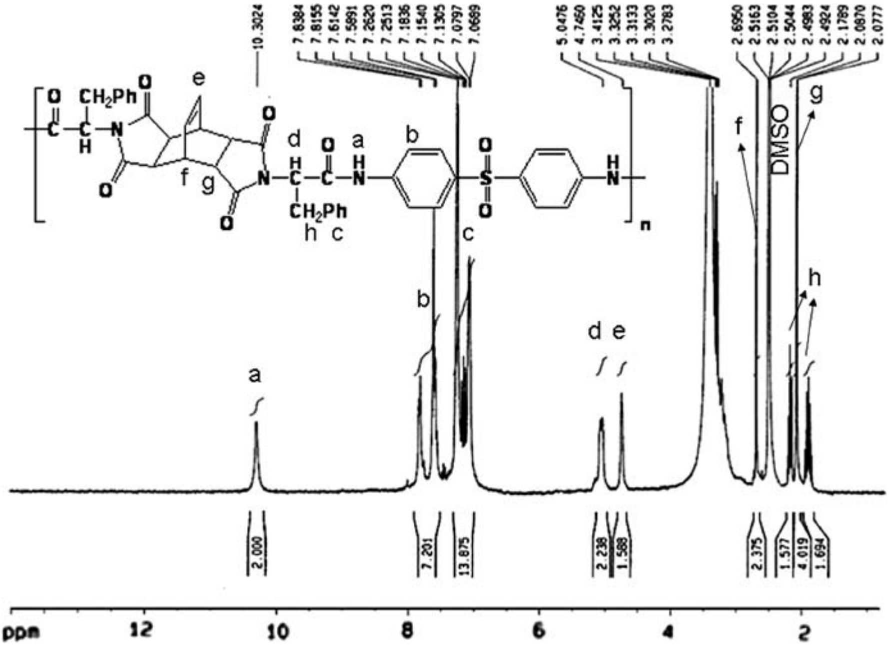


Fig. 7 ^{1}H-NMR spectrum of PAI **6b**

Table 3 Elemental analysis of PAIs **6a–e**

Polymer	Formula		Elemental analysis (%)		
			C	H	N
6a	($C_{42}H_{34}N_4O_7$)	Calcd.	71.37	4.81	7.92
	$(706.74)_n$	Found	70.60	4.10	7.20
6b	($C_{42}H_{34}N_4O_8S$)	Calcd.	66.82	4.50	7.42
	$(754.81)_n$	Found	66.49	4.27	7.37
6c	($C_{42}H_{34}N_4O_8S$)	Calcd.	66.82	4.50	7.42
	$(754.81)_n$	Found	66.35	4.26	7.18
6d	($C_{40}H_{32}N_4O_6$)	Calcd.	72.27	4.81	8.42
	$(664.70)_n$	Found	71.8	4.2	7.90
6e	($C_{36}H_{31}N_4O_6$)	Calcd.	70.34	5.04	9.11
	$(614.64)_n$	Found	69.92	4.86	8.78

Table 4 Solubility of PAIs **6a–e**

Solvent	**6a**	**6b**	**6c**	**6d**	**6e**
DMAc	+	+	+	+	+
DMF	+	+	+	+	+
DMSO	+	+	+	+	+
NMP	+	+	+	+	+
CH_3OH	−	−	−	−	−
C_2H_5OH	−	−	−	−	−
$CHCl_3$	−	−	−	−	−
CH_2Cl_2	−	−	−	−	−
H_2O	−	−	−	−	−

(+) Soluble in room temperature, (−) Insoluble in room temperature

Table 5 Thermal properties of PAIs **6a** and **6e**

Polymer	T_5 (°C)[a]	T_{10} (°C)[a]	Char yield (%)[b]
6a	203–213	348–358	53.80
6e	243–253	256–266	56.21

[a,b] Temperature at which 5 or 10% weight loss was recorded TGA at a heating rate of 10 °C/min in N_2

[b] Weight percentage of material left after TGA analysis at maximum temperature 600 °C in N_2

polymers show that PAI (**6e**) has a better thermal stability than PAI (**6a**). It can be due to presence of naphthalene structure in this polymer.

Conclusions

This work has shown that *N,N′*-(bicyclo[2,2,2]oct-7-ene-2,3,5,6-tetracarboxylic)bis-L-phenyl alanine **4** is an interesting monomer which contains both imide groups as

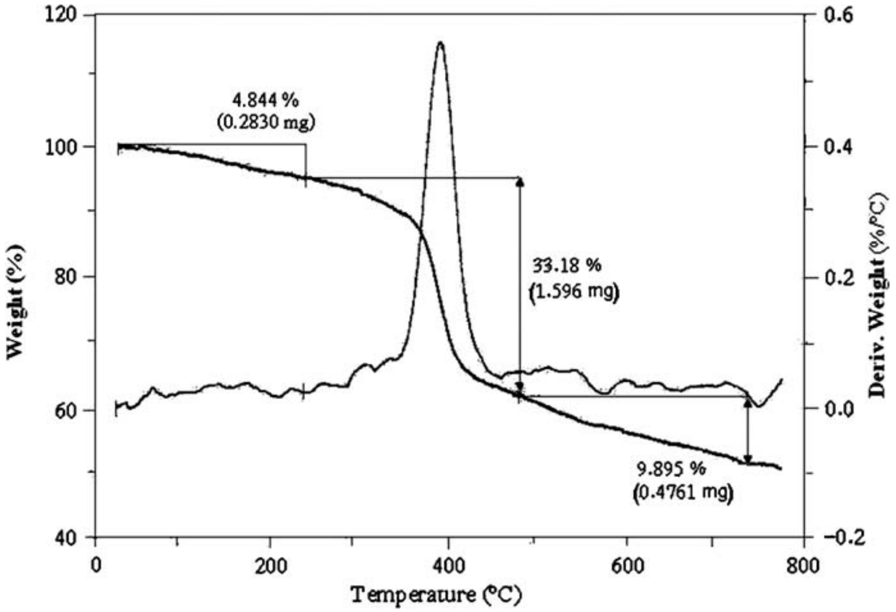


Fig. 8 TGA and DTG thermogram of PAI **6e**

well as a chiral L-phenyl alanine groups. Thus, a series of new optically active PAIs **6a**–**e** were able to be synthesized by polycondensation reaction using the optically active monomer **4** with five aromatic diamines **5a**–**e**. Due to the presence of chiral segment in the polymer backbone, these polymers are optically active. Also, all of these polymers have good solubility in polar aprotic solvents such as DMF, NMP, and DMSO.

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