

Synthesis and characterization of new optically active poly(amide-imide)s derived from N,N'-(pyromellitoyl) bis-L-tyrosine and various diamines

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Five new optically active poly(amide-imide)s (PAIs) **5a – e** were prepared by direct polycondensation reaction of N,N'-(pyromellitoyl) bis-L-tyrosine **3** as chiral dicarboxylic acid with various aromatic diamines **4a – e**. Triphenylphosphite (TPP)/pyridine (py) in the presence of calcium chloride (CaCl₂) and N-methyl-2-pyrrolidone (NMP) were successfully applied to direct polycondensation reaction. The resulting new polymers were obtained in good yields with inherent viscosities ranging between 0.48 dL/g and 0.6 dL/g. They were analyzed with a C.H.N. elemental analyzer, FTIR, ¹H-NMR, UV-VIS spectroscopy and polarimeter (specific rotation measurement, $[\alpha]_D^{25}$). Thermogravimetric analysis (TGA) indicated that the residual weight percentage of polymers at 600 °C were between 48.66 % and 64.21 %, which showed their thermal stability. These polymers are attractive to be used as packing materials in chromatography columns for separation of enantiomers.

Keywords: *polycondensation; poly(amide-imide)s; optical activity*

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1. Introduction

Aromatic polyamides and polyimides are an important class of high performance polymers providing many useful applications for high technology as advanced materials. These polymers constantly attract wide interest because of their excellent mechanical strength, high thermal stability, good chemical resistance, and low flammability as well as balanced mechanical and electrical properties. However their applications have been limited in some fields due to their insolubility in common organic solvents and infusibility [1–5]. This fact promoted investigations on modification of these compounds to overcome these problems. As a result, some copolymers have been proposed through synthetic modification by the incorporation of flexible linkages and bulky pendant groups into the backbone of the polymers [6–11]. One of the important copolymers that shows good balance between processability and thermal stability is the poly(amide-imide)s (PAIs). It brings together the superior mechanical properties associ-

ated with amide groups and the high thermal stability determined by imides rings. Poly(amide-imide)s show good solubility in highly polar solvents, lower glass transition temperatures and easier processability compared with corresponding polyimides. Moreover, flexible asymmetrical linkages, large pendant groups or polar substituent improve further solubility in organic solvents when introduced into the backbone of the polymer [12–22].

Recently, optically active poly(amide-imide)s have been synthesized by the reaction of optically active monomer with several diamines using the solution polymerization methods. The synthesis and application of the chiral poly(amide-imide)s are of particular interest from the viewpoint of materials science and newly considered topics. The synthesis and characterization of the condensation of optically active polymer utilizing different amino acids as chiral compounds have already been reported [23–27]. The polymers containing chiral center group have found successful uses as stationary phases in chromatographic separation of enantiomers in solutions by high performance liquid chromatography (HPLC) [28–30].

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In this work we synthesized and characterized a series of novel optically active poly(amide-imide)s PAIs (**5a–e**) containing L-tyrosine as a chiral center group as well as polar pendant group. Direct polycondensation reactions of N,N'-(pyromellitoyl) bis-L-tyrosine as dicarboxylic acid with five aromatic diamines (1,4-phenylene diamine, 4,4'-diphenylene diamine, o-tolidine, 2,5-diamino toluene, 1,3-phenylene diamine) were carried out. N-methyl-2-pyrrolidone (NMP) triphenyl phosphate (TPP), pyridine and calcium chloride were the media of reaction. We hope that these novel polymers can be utilized as stationary phases in chromatographic separation of enantiomers mixtures.

2. Experimental

2.1. Materials

All chemicals were purchased from Fluka, Merck, Aldrich and BDH chemical companies. Pyromellitic dianhydride (1,3,4,5-benzene-tetracarboxylic acid-1,2,4,5-dianhydride) was purified by recrystallization from a mixture of acetic acid and acetic anhydride (4:1). L-tyrosine amino acid (ultra pure) and all aromatic diamines (1,4-phenylene diamine, 4,4'-diphenylene diamine, o-tolidine, 2,5-diamino toluene, 1,3-phenylene diamine), were used without further purification. The solvent, N-methyl-2-pyrrolidone (NMP) was dried by distillation under reduced pressure over calcium hydride and stored on the molecular sieve 4 Å. Triphenyl phosphate (TPP) (above 97 %) was purified by vacuum distillation before use. Glacial acetic acid was used as received. Commercially available calcium chloride (CaCl₂) was dried under vacuum at 150 °C, for 6 h.

2.2. Apparatus and techniques

¹H-NMR spectra were recorded on a Bruker 300 MHz instrument, using DMSO as a solvent and chemical shifts were given relative to tetramethyl silane (TMS). The multiplicities of proton resonances were designated as singlet (s), doublet (d), triplet (t), quartet (q), multiplet (m), and broad (br). FT-IR spectra were recorded on an ABB Spectrolap MB 3000 England, transform infrared (FT-IR) spectrometer. Vibration transition frequencies were

recorded in the wave number region between 500 – 4000 cm⁻¹. The band intensity was classified as weak (w), medium (m), shoulder (sh), strong (s), and broad (br). UV/VIS absorption values were obtained in the 200 – 700 nm spectral regions on a Shimadzu multispect – 1501 spectrophotometer. DMF (99.99 %) solution at a concentration of 0.1 mg mL⁻¹ at room temperature and cell path length of 1cm were used for the measurement. Elemental analysis was run on a Heraeus Vario analyzer. Specific rotation was measured by a Jasco polarimeter (Japan). The inherent viscosities (η_{inh} , dL/g) of the polymers were determined with a Ubbelohde viscometer, by using polymer solution in NMP at 20 °C and concentration of 0.5 g/dL. Thermogravimetric analysis (TGA) data for polymers were obtained on a Mettler TA4000 system under a nitrogen atmosphere at a heating rate of 10 °C min⁻¹ and sample weight of about 10 mg.

2.3. Synthesis of monomer N,N'-(pyromellitoyl) bis-L-tyrosine 3

In a 50 mL round-bottomed flask, 1.1 g (5 mmol) of pyromellitic dianhydride **1**, 1.82 g (10 mmole) of L-tyrosine **2**, 35 mL of glacial acetic acid and a stirring magnetic bar were placed. The mixture was stirred at room temperature overnight. The round bottomed flask was then fitted with a condenser and the mixture refluxed for 5 h. The solvent was removed under reduced pressure and heating. After addition of about 10 mL concentrated hydrochloric acid and stirring, a yellow precipitate was formed which was filtered and washed thoroughly with plenty of cold distilled water. Finally, the yellow precipitate was dried under vacuum at 80 °C to give 2.887 g (89 % yield) of compound **3**, mp = 248 °C (decomposed), $[\alpha]_D^{25} = 86.7^\circ$ (0.05 g in 10 mL DMF at 25 °C). UV-VIS absorption spectrum $\lambda_{max} = 278\&310$ nm, FT-IR (KBr): 3500 – 2500 (br), 1769 (w), 1723 (s), 1614 (m), 1516 (m), 1442 (m), 1386 (s), 1229 (m), 1117(m), 944 cm⁻¹ (w), 891 cm⁻¹ (w), 839 cm⁻¹ (m), 735(w), 635(w) cm⁻¹. ¹H-NMR (300 MHz DMSO, TMS): δ , ppm; H₁ = 9.20 (s, br, 2H), H₂ = 8.22 (s, 2H), H₃ = 8.00 (d, 2H), H₄ = 7.00 (d, 4H), H₅ = 6.55 (d, 4H), H₆ = 5.00 (dd, 2H), H₇ = 3.35 (m, 4H). C.H.N Analysis:

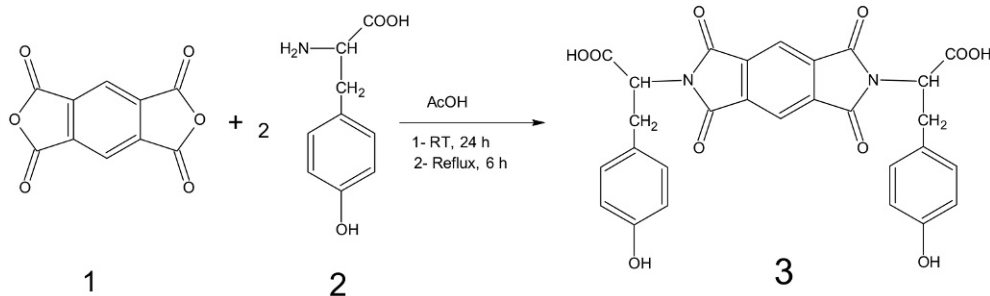


Fig. 1. Synthesis of *N,N'*-(pyromellitoyl) bis-L-tyrosine **3**.

calculated for $C_{28}H_{20}N_2O_{10}$; C, 61.76; H, 3.67; N, 5.14, found: C, 62.01; H, 3.60; N, 5.09.

2.4. Synthesis of poly(amide-imide)s (PAIs)

The synthesis of poly(amide-imide)s **5a** is used as an example to illustrate the general synthetic route; it was prepared as follows: A mixture of the prepared *N,N'*-(pyromellitoyl) bis-L-tyrosine **3** (1.071 g, 2.02 mmol), 4,4'-diphenylene diamine **4a** (0.375 g, 2.02 mmole), 1 g of calcium chloride, 10 mL of NMP, 8 mL of triphenyl phosphite and 3 mL of pyridine was placed in 50 mL round bottomed flask with a stirring bar and condenser. The mixture was left at room temperature for 2 h with continuous stirring. Then the temperature was elevated gradually to 120 – 130 °C and the reaction was continued for 6 h. The obtained polymer solution was poured slowly into stirred methanol (300 mL), giving rise to stirring precipitate. The precipitate was separated by filtration, washed with methanol (40 mL) then with hot water (150 mL) and dried at 120 °C under vacuum for 8 h to give 1.230 g (90 %) of solid polymer. Other poly(amide-imide)s were synthesized by a similar procedure.

3. Results and discussion

3.1. Synthesis of diimid-diacid monomer

The *N,N'*-(pyromellitoyl)bis-L-tyrosine **3** was synthesized by the condensation reaction of the equimolar quantity of pyromellitic dianhydride **1** with two equimolar quantities of L-tyrosine **2** in an glacial acetic acid solution according to the reported method [17], (Fig. 1).

The procedure consists of two steps: the first is the formation of amic acid at room temperature while the second is imide ring formation by refluxing. The resulting optically active aromatic diimide-diacid **3** containing chiral center and imide rings was obtained in high yield.

The chemical structure and purity of the monomer was confirmed by means of FT-IR, NMR spectroscopy and elemental analysis. Fig. 2 shows an exemplary FT-IR spectrum of diimide-diacid **3**. A broad peak at 3500 – 2500 cm^{-1} is due to the OH stretching vibration of carboxylic group in which the OH stretching vibration of phenolic ring appears at 3412 cm^{-1} . The peaks at 1769 cm^{-1} (C=O), asymmetric imide stretching vibration, 1723 cm^{-1} (C=O), acid and symmetric imide stretching vibrations, 1386 cm^{-1} , 735 cm^{-1} and 1117 cm^{-1} (C–N), stretching vibration and ring deformation, respectively confirm the presence of imide ring and carboxylic groups in this compound. The peak at 839 cm^{-1} indicates the para substitution of aromatic ring (phenol).

The 1H -NMR spectrum in Fig. 3 confirms the proposed structure for diimide-diacid monomer **3** by showing the carboxylic hydrogens (H_1 , 9.20 ppm, 2H), the aromatic hydrogens of imide ring (H_2 , 8.22 ppm, 2H), hydrogen of phenolic hydroxyl (H_3 , 8.00 ppm, 2H). Several peaks were assigned to protons of phenol ring (H_4 , H_5 , 7.0 – 6.5 ppm, 8H), hydrogen chiral carbon (H_6 , 5.00 ppm, 2H), and the diastereotropic protons (H_7 , 3.35 ppm, 4H). The elemental analysis of the monomer showed good agreement with the calculated values, demonstrating that high purity of the resulting compound was obtained. The UV-VIS absorption of the diimide-

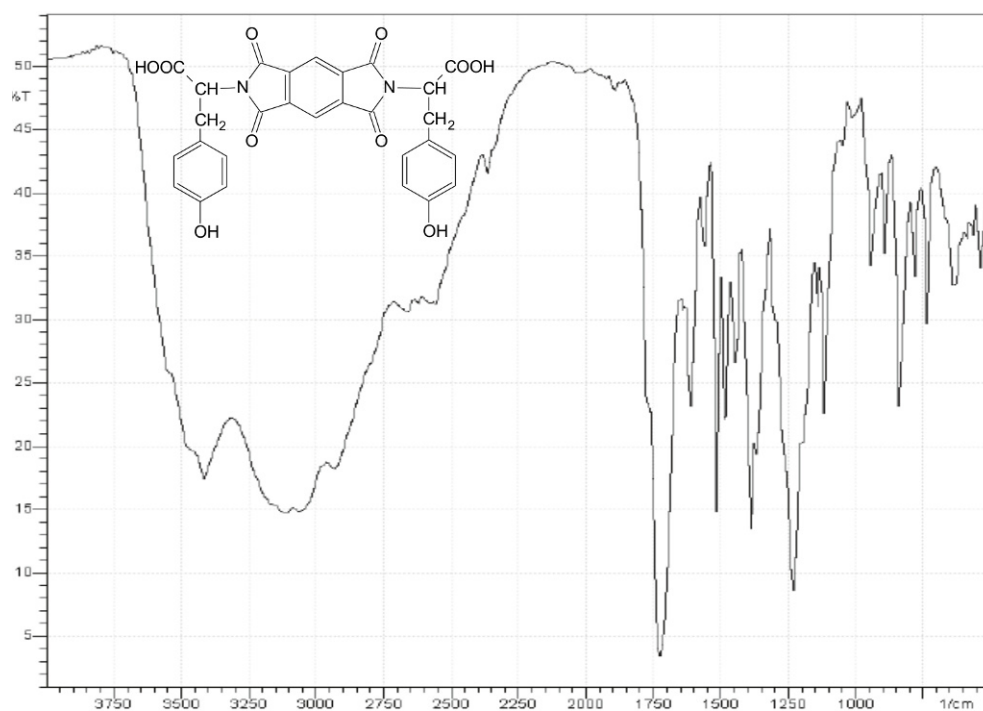


Fig. 2. FT-IR Spectrum of N,N'-(pyromellitoyl) bis-L-tyrosine **3**.

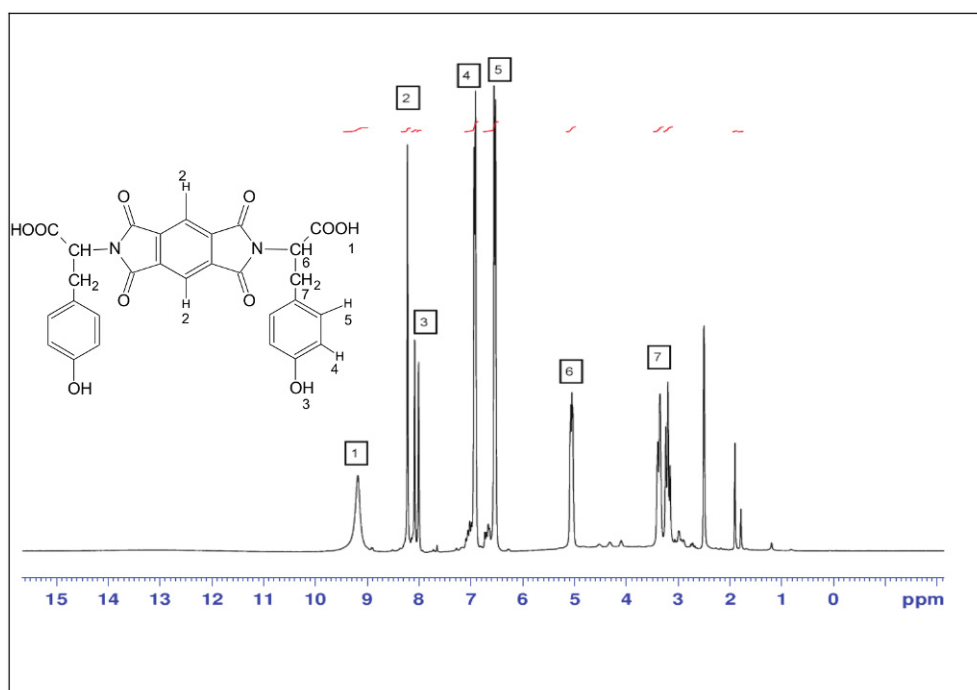
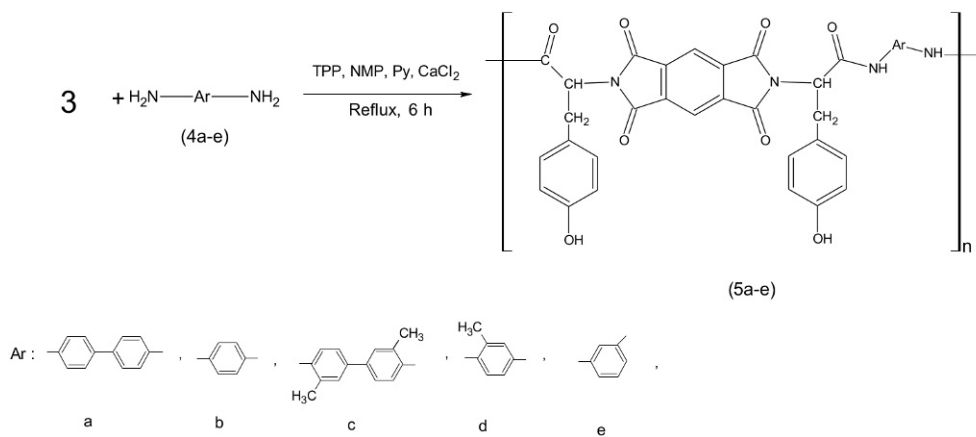


Fig. 3. ¹H-NMR Spectrum of N,N'-(pyromellitoyl) bis-L-tyrosine **3**.

Fig. 4. Synthesis of PAIs (**5a – e**).Table 1. The yield and physical properties of PAIs (**5a – e**).

Aromatic diamine	Polymer	Yield %	η_{inh} (dL/g) ^a	$[\alpha]_D^{25}$	Color
4a	5a	82	0.62	+73	Brown
4b	5b	85	0.47	+66	Beige
4c	5c	79	0.56	+94	Brown
4d	5d	80	0.51	+63	Deep brown
4e	5e	86	0.48	+69	Brown

^aMeasured at a concentration of 0.5 g dL⁻¹ in DMF at 25 °C.

diacid monomer in DMF solution displayed the maximum absorptions (λ_{max}) at 278 nm and 310 nm, which corresponds to $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ respectively.

3.2. Synthesis of polymers

PAIs (**5a – e**) were synthesized by direct polycondensation of an equimolar mixture of diimide-diacid monomer **3** with five different diamines (**4a – e**) according to the reported method [17] as shown in Fig. 4. The reaction was carried out in a medium consisting of N-methyl-2-pyrrolidone as a solvent and triphenyl phosphite, pyridine and calcium chloride as condensation agents. It is a well known convenient method to prepare aromatic polyimides because phosphorylation of the diacid and the phosphorylated acid is more reactive than the diacid itself.

The yields and some physical data of the new PAIs (**5a – e**) are summarized in Table 1. The re-

sulting polymers are optically active as shown by specific rotation ($[\alpha]_D^{25} = 63 - 94^\circ$) measurements. This is due to the incorporation of an optically active chiral center group of L-tyrosine. The values of specific rotation $[\alpha]_D^{25}$ of the synthesized PAIs (**5a – e**) could be not predicted, because they are highly dependent on the structure of the polymers. Due to chirality and optical activity of these polymers, they may be useful materials as column stationary phases applied in HPLC technique for the resolution of racemic mixtures. These polymers have inherent viscosity (η_{inh}), in the range of 0.47 – 0.62 dL/g.

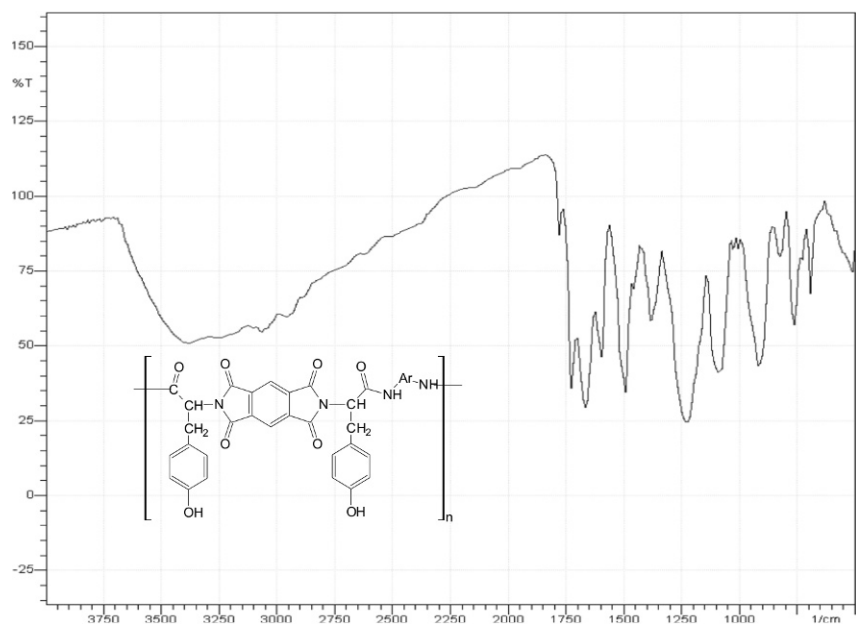
3.3. Polymer characterization

Table 2 shows the elemental analysis of the resulting PAIs (**5a – e**) which proves the proposed structure due to the agreement with the calculated values of the chemical formulas of these polymers.

The FT-IR and ¹H-NMR spectroscopy data of all the synthesized PAIs (**5a – e**) are listed in Table 3.

Table 2. Elemental analysis of PAIs (**5a – e**).

Polymer	Formula		C %	H %	N %
5a	C ₄₀ H ₂₈ N ₄ O ₈ (692)n	Calculated	69.36	4.04	8.09
		Found	70.13	4.10	8.22
5b	C ₃₄ H ₂₄ N ₄ O ₈ (616)n	Calculated	66.23	3.89	9.09
		Found	65.77	4.00	9.21
5c	C ₄₂ H ₃₂ N ₄ O ₈ (720)n	Calculated	70.00	4.44	7.77
		Found	68.98	4.38	7.48
5d	C ₃₅ H ₂₆ N ₄ O ₈ (630)n	Calculated	6.66	4.42	8.88
		Found	67.05	4.33	8.94
5e	C ₃₄ H ₂₄ N ₄ O ₈ (616)n	Calculated	66.23	3.89	9.09
		Found	66.94	3.92	9.11

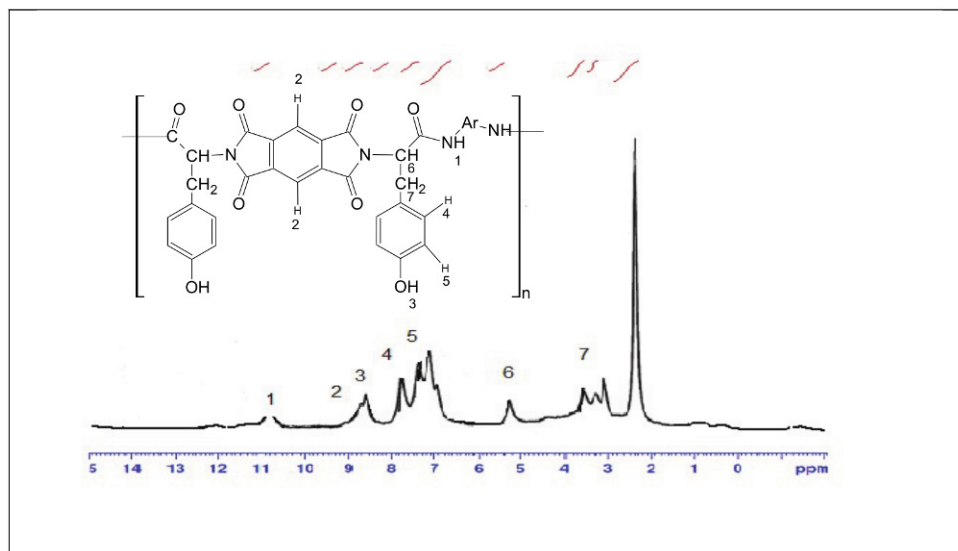
Fig. 5. FT-IR spectrum of PAI (**5a**).

Typical FT-IR and ¹H-NMR spectra of PAI (**5a**) are shown in Figs. 5 and 6, respectively.

All the PAIs (**5a – e**) show strong broad bands at 3500 – 2750 cm⁻¹ including the stretching vibrations at 3372 – 3360 cm⁻¹ and 3248 – 3250 cm⁻¹, related to phenolic hydroxyl and NH amide group formation, respectively. The bands at 1772 – 1780 cm⁻¹ and 1720 – 1724 cm⁻¹ are commonly attributed to the asymmetrical and symmetrical vibrations of carbonyl groups of imide. The stretching vibrations of C–N group of imide ring appear at 1371–1381 cm⁻¹ and imide ring deformation at 764 – 730 cm⁻¹. These absorptions confirm the imide ring, while the 1664 – 1670 cm⁻¹ (C=O,

amide I) and 1514 – 1496 cm⁻¹ (C=O, amide II, deformation) all prove the formation of amide group.

All polymers show a characteristic peak, H₁ = 10.92 – 10.85 ppm (br, s) related to the hydrogen of amide groups in the polymers chain. The absorption of H₂ aromatic protons from imide ring appears in the range of 8.77 – 8.66 ppm as a single peak. The protons –H₃ of phenolic ring appear in the range of 8.68 – 8.38 ppm while several peaks can be assigned to the protons for phenol and aromatic amino rings (H₄, H₅, HAr, 16H, 7.56 – 6.75 ppm). The protons of chiral center and diastereotopic hydrogen of lysine moiety appear in the ranges (H₆, 5.32 – 5.02 ppm, 2H) and (H₇, 3.57 – 3.17 ppm, 4H),

Fig. 6. ^1H -NMR spectrum of PAIs (5a).Table 3. FT-IR and ^1H -NMR Spectroscopy Data of PAIs (5a – e).

Polymer	IR (Cm^{-1})	^1H -NMR(δ , ppm)
5a	3372 (w, CON-H), 3248 (w, OH), 1772 (w, asymm. C=O imide), 1720 (s, sym. C=O), 1664 (s, C=O amide I), 1590 (w, C=O amide II), 1376 (m, C-N)	10.92 (br, 2H), 8.68 & 8.74 br, 4H, 7.52 – 6.75 (m, 16H) 5.19 (s, 2H), 3.57 – 3.17 (t, 4H)
5b	3369 (w, CON-H), 3244 (w, OH), 1775 (w, asymm. C=O imide), 1722 (s, sym. C=O), 1680 (s, C=O amide I), 1595 (w, C=O amide II), 1380 (m, C-N)	10.85 (br, 2H), 8.450 & 8.72 br, 4H, 7.52 – 6.75 (m, 12H) 5.30 (s, 2H), 3.55 – 3.22 (t, 4H)
5c	3365 (w, CON-H), 3249 (w, OH), 1770 (w, asymm. C=O imide), 1722 (s, sym. C=O), 1668 (s, C=O amide I), 1570 (w, C=O amide II), 1379 (m, C-N)	10.87 (br, 2H), 8.53 & 8.66 br, 4H, 7.49 – 6.78 (m, 14H) 5.22 (s, 2H), 3.52 – 3.15 (t, 4H), 2.25 (s, 6H)
5d	3368 (w, CON-H), 3240 (w, OH), 1772 (w, asymm. C=O imide), 1720 (s, sym. C=O), 1664 (s, C=O amide I), 1590 (w, C=O amide II), 1376 (m, C-N)	10.90 (br, 2H), 8.38 & 8.77 br, 4H, 7.52 – 6.89 (m, 11H) 5.15 (s, 2H), 3.39 – 3.12 (t, 4H), 2.31 (s, 3H)
5e	3377 (w, CON-H), 3260 (w, OH), 1769 (w, asymm. C=O imide), 1720 (s, sym. C=O), 1665 (s, C=O amide I), 1560 (w, C=O amide II), 1377 (m, C-N)	10.81 (br, 2H), 8.55 & 8.72 br, 4H, 7.55 – 6.75 (m, 12H) 5.30 (s, 2H), 3.50 – 3.20 (t, 4H)

respectively. The decaying peak related to the protons of carboxylic acid at 9.20 ppm and the peak related to amide group at 10.32 – 10.11 ppm in the polymers chain confirm the proposed structures of PAIs (5a – e).

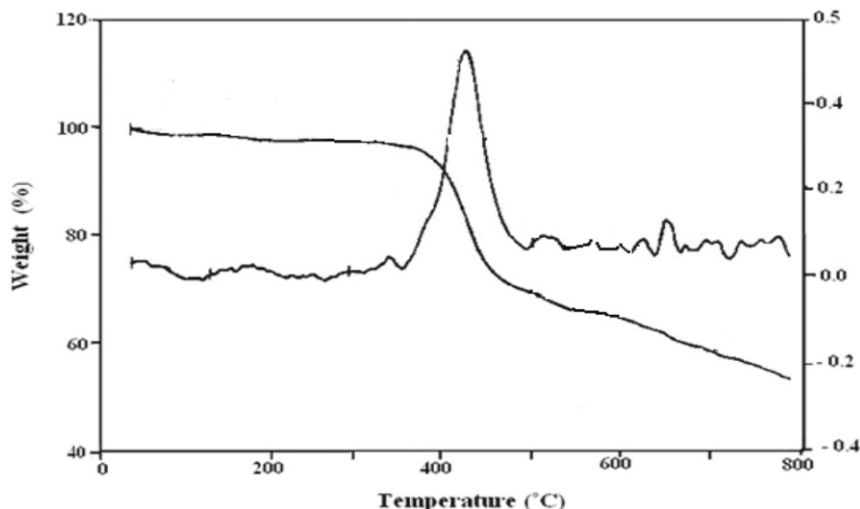
3.4. UV-VIS Absorption of the PAIs (5a-e)

All polymers solutions (0.5 g/dL in DMF at 25 °C) exhibited absorptions (λ_{max}) at around 274 – 272 nm and 306 – 304 nm, corresponding to $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ transitions, respectively. By compar-

ing these results with the diacid monomer (N,N'-(pyromellitoyl) bis-L-tyrosine 3), slight blue shift was shown.

3.5. The solubility properties of PAIs (5a – e)

The solubility of PAIs (5a – e) was investigated on 0.01 g of polymeric sample in 20 mL solvent. Table 4 illustrates the solubility of these polymers in various solvents.

Fig. 7. TGA and DTG thermogram of PAIs (**5a**).Table 4. Solubility of PAIs (**5a – e**).

Solvent	Polymer				
	5a	5b	5c	5d	5e
H ₂ SO ₄	+	+	+	+	+
DMSO	++	++	++	++	++
DMAc	++	++	++	++	++
DMF	++	++	++	++	++
NMP	++	++	++	++	++
THF	–	–	–	–	–
MeOH	–	–	–	–	–
EtOH	–	–	–	–	–
CHCl ₃	–	–	–	–	–
CH ₂ Cl ₂	–	–	–	–	–
H ₂ O	–	–	–	–	–

+ soluble at room temperature;

++ soluble after 60 °C heating,

– insoluble

Table 5. Thermal behavior of PAIs (**5a – e**).

Polymer	T ₅ (°C) ^a	T ₁₀ (°C) ^a	Char yield (%) ^b
5a	355	395	65.34
5b	370	410	62.56
5c	340	370	48.66
5d	365	415	55.25
5e	375	425	64.21

^aTemperatures at which 5 % and 10 % weight losses respectively, were recorded by TGA at a heating rate of 10 °C min^{–1} in nitrogen;^bPercentage weight of material left undecomposed after TGA analysis at 600 °C.

3.6. Thermal properties

Thermal gravimetric analysis (TGA) in N₂ atmosphere at a heating rate of 10 °C min^{–1} was performed in order to study the thermal properties of synthesized PAIs (**5a – e**). The thermal data are summarized in Table 5. All polymers show similar decomposition behavior for different samples. Initial decomposition temperatures (5 % and 10 % weight loss temperatures) together with the weight residue at 600 °C (char yield %) of PAIs **5a** is shown in Fig. 7. The temperatures of initial decomposition (T₅ and T₁₀) which range from 355 to 375 °C and

370 to 425 °C in nitrogen, respectively, and the high char yields (48.66 – 65.34 %) at 600 °C of PAIs (**5a – e**) indicate that these polymers have good thermal stability (Table 5). The presence of phenolic ring from tyrosine may assist to give the resistance to thermal decomposition.

4. Conclusions

A series of PAIs (**5a – e**) containing phenol as pendent groups was synthesized by direct polycondensation of various aromatic diamines (**4a – e**) with N,N'-pyromellitoyl bis-L-tyrosine **3** by using TPP, NMP, CaCl₂ and pyridine as condensing agents. The introduction of phenol group into the polymer backbone assisted the polymer to improve its thermal

stability. These polymers also showed good optical activity due to the presence of tyrosine amino acid. Optical activity, thermal stability and inertness to most of solvents can make these polymers attractive to be used as packing materials in chromatography columns for separation of enantiomers.

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