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WHOLLY AROMATIC CHIRAL POLYAMIDES BEARING PENDANT PHTHALIMIDO AND L-ISOLEUCINE MOITIES*

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Abstract A potential biodegradable and optically active bulky chiral aromatic amide-imidic diacid monomer, (2*S*,3*S*)-5-(3-methyl-2-phthalimidylpentanoylamino)isophthalic acid (**7**), containing a rigid phthalimide and flexible L-isoleucine pendant group was synthesized in three steps. New aromatic polyamides including pendant phthalimido groups and flexible side spacers have been synthesized by direct polycondensation reaction of equimolar amounts of different aromatic diamines with an optically active diacid **7**, using *N*-methyl-2-pyrrolidone (NMP) as a solvent and triphenyl phosphite/$CaCl_2$/pyridine as a condensing agent. These polyamides were characterized by FTIR, ^{1}H-NMR spectroscopy, specific rotation, thermogravimetric and elemental analysis. The resulting polymers have inherent viscosities in the range of 0.21–0.45 dL/g. Amino acid existence in this backbone results in optically active polymers. Due to introduction of bulky and flexible groups in these polyamides, they show improved solubility in polar aprotic solvents such as NMP and dimethylacetamide and also good thermal stability (10% weight loss temperatures in excess of 330°C, and char yields at 600°C in nitrogen higher than 62%).

Keywords: Polyamides; Biodegradable; Thermally stable polymers; Thermogravimetric analysis; Polycondensation reaction.

INTRODUCTION

Aromatic polyamides (PAs) became breakthrough materials in commercial relevances as early as the 1960s, which opened up new horizons in the field of thermal and electrical insulation. In the main, they are characterized by having aromatic repeat units bonded together by amide-CONH-groups. All members of this group are typified by having thermal stabilities in excess of 300°C for short-term exposures and high levels of inherent flame resistance[1]. Entirely aromatic PAs have received exceptional interest because of their high thermal stability, chemical resistance and their potential as high-performance materials for numerous applications, for example high-strength, high-modules fibers, high temperature coating and high effectiveness semipermeable membranes[2–8]. Because of the rigidity of the aforementioned polymer chains and strong molecular interactions via hydrogen bonding, their application is limited by their deprived processability caused by their poor solubility in common organic solvents, the high melting or softening temperature and high glass transition[9–12]. Consequently, much attempt has been made to develop structurally modified macromolecules having improved solubility, in order to improve their processabillity whereas maintaining the good thermal stability[13–16]. The prologue of pendant bulky groups along the polymer backbone results in a less ordered polymer matrix increasing the solubility characteristics with no affecting thermal and mechanical properties to

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any large extent[12, 17–20].

Investigation on chiral macromolecules represents an interesting field of polymer chemistry[21, 22]. Some applications of these materials could be listed as the following: assembling chiral media for asymmetric synthesis, chiral stationary phases for resolution of enantiomers in chromatographic techniques, chiral liquid crystals in ferroelectrics and nonlinear optical devices. In all the abovementioned cases, the optical activity is induced preferentially using polymerization of a chiral monomer[23–27].

Lately, there is a growing demand for biodegradable polymers as a solution to problems concerning the energy resources, global environment and the solid waste management. Many bioanalogous polymers derived from natural amino acids or peptide linkages, including PAs, poly(ester-amides), poly(ester-urethane) and poly(ester-urea) have been prepared and found to be biodegradable[24]. The interest for developing novel biodegradable or biocompatible polymers, particularly PAs and polyesters, has basically encouraged the use of monomers based on naturally occurring products. Both carbohydrate and α-amino acid derived monomers are being presently utilized as building blocks to produce novel PA structures with improved biodegradability[28–33].

Due to the importance of optically active materials and polymers with amino acid moieties, in continuation of our study[28–33] in developing easily processable high performance polymers, the current work describes a successful use of 5-(3-methyl-2-phthalimidylpentanoylamino)isophthalic acid (**7**) to prepare aromatic PAs by the condensation of this chiral diacid **7** with dissimilar aromatic diamines. We report here the preparation and basic characterization of novel PAs based on this potential biodegradable and optically active monomer. The introduction of thermally stable side chain ought to disrupt interchain hydrogen bonding, and consequently diminish packing effectiveness and crystallinity, so all of the PAs are expected to show good solubility in many organic solvents.

EXPERIMENTAL

Materials

All chemicals were purchased from Fluka Chemical Co. (Buchs, Switzerland), Aldrich Chemical Co. (Milwaukee, WI) and Riedel-deHaen AG (Seelze, Germany). Pyridine (Py) **(**Merck) and *N*-methyl-2-pyrrolidone (NMP) (Merck) were dried over BaO and were distilled under reduced pressure. *N*,*N*-dimethylacetamide (DMAc) (Merck), dimethyl sulfoxide (DMSO) (Merck) and *N*,*N*-dimethylformamide (DMF) (Riedel-deHaen AG) were used as received. 1,5-Diaminonaphthalene (**8A**) and 4,4′-diaminodiphenylmethane (**8B**) were purified by recrystallization from water. 4,4′-Diaminodiphenylsulfone (**8E**) was purified by recrystallization from water/ethanol (EtOH) 50/50. 4,4′-Diaminobiphenyl (**8C**), 4,4′-diaminodiphenylether (**8D**), 2,4-diaminotoluene (**8F**), 1,3-phenylenediamine (**8G**) and 1,4-phenylenediamine (**8H**) were purified *via* sublimation.

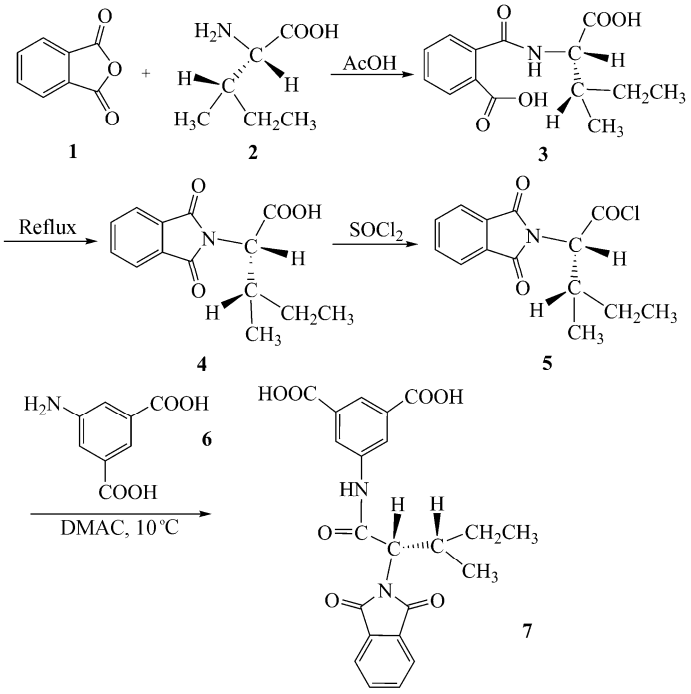
Techniques

Proton nuclear magnetic resonance (^{1}H-NMR, 500 MHz) spectra were recorded in DMSO-d_6 solution using a Bruker (Ettlingen, Germany) Avance 500 instrument. Multiplicities of proton resonance were designated as singlet (s), doublet (d), doublet of doublet (dd), triplet (t) and multiplet (m). FTIR spectra were recorded on a Jasco-680 spectrophotometer (Japan). 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) and strong (s). All melting points were taken with a (Gallenkamp, England) melting point apparatus. Inherent viscosities were measured by standard procedure using a Cannon Fenske Routine viscometer (Cannon, Mainz, Germany). Specific rotations [α] were measured by a JASCO (P-1030) Polarimeter (Toyonaka, Osaka, Japan). Thermogravimetric analysis (TGA) data for PAs were taken on a TGA Perkin Elmer (Pyris 1) at a heating rate of 10 K/min under nitrogen atmosphere by the Research Institute of Polymer and Petrochemical of Iran (IPPI).

Monomer Synthesis

Phthalic anhydride (**1**) and L-isolucine (**2**) underwent condensation reaction in acetic acid to produce the optically active 3-methyl-2-phthalimidyl pentanoic acid (**4**). In this reaction the intermediate amic-acid **3** was not isolated and ring closure for the formation of imide ring occurred under refluxing conditions. The imide acid **4** was converted to acid chloride **5** by the reaction with thionyl chloride. Finally, the reaction of acid chloride **5**

with 5-aminoisophthalic acid (**6**) gave novel optically active monomer **7** to yield 85% of diacid **7** according to our previous work[33] (Scheme 1). The chemical structure and purity of the compounds **7** were proved using thin layer chromatography, elemental analysis, FTIR, ^{13}C-NMR (Fig. 1), ^{1}H-NMR (Fig. 2) spectroscopic techniques and specific rotation. m.p. > 315°C (dec.), $[\alpha]_D^{25}$ = –27.3 (0.05 g in 10 mL DMF). FTIR (KBr): 3323 (m, br), 2972 (m, br), 2930 (m), 1773 (w), 1700 (s, br), 1609 (m), 1569 (m), 1466 (w), 1442 (w), 1396 (s), 1333 (m), 1289 (m), 1263 (m), 1199 (m), 1097 (w), 1069 (m), 901 (w), 753 (w), 726 (m), 529 (w) cm^{-1}. ^{1}H-NMR (500 MHz, DMSO-d_6): δ = 0.84 (t, 3H, J = 7.52 Hz), 0.97 (m, 1H), 1.00 (d, 3H, J = 6.72 Hz), 1.50 (m, 1H), 2.60 (m, 1H), 4.67 (d, 1H, J = 8.05 Hz), 7.88 (dd, 2H, J = 3.20 Hz), 7.93 (dd, 2H, J = 4.10 Hz), 8.16 (s, 1H), 8.42 (s, 2H), 10.34 (s, 1H), 13.28 (s, 2H). ^{13}C-NMR (125 MHz, DMSO-d_6): δ = 11.90, 17.41, 25.95, 34.63, 59.09, 124.20, 125.04, 125.68, 132.16, 132.48, 135.58, 140.18, 167.28, 168.03, 168.62; elemental analysis calculated for $C_{22}H_{20}N_2O_7$: C, 62.26%; H, 4.75%; N, 6.60%. Found: C, 61.60%; H, 4.72%; N, 6.52%.



Scheme 1 Synthesis of optically active monomer **7** in three steps

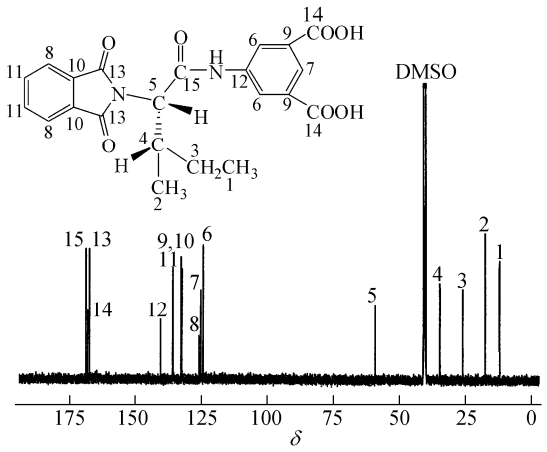


Fig. 1 ^{13}C-NMR (125 MHz) spectrum of diacid **7** in DMSO-d_6 at R.T.

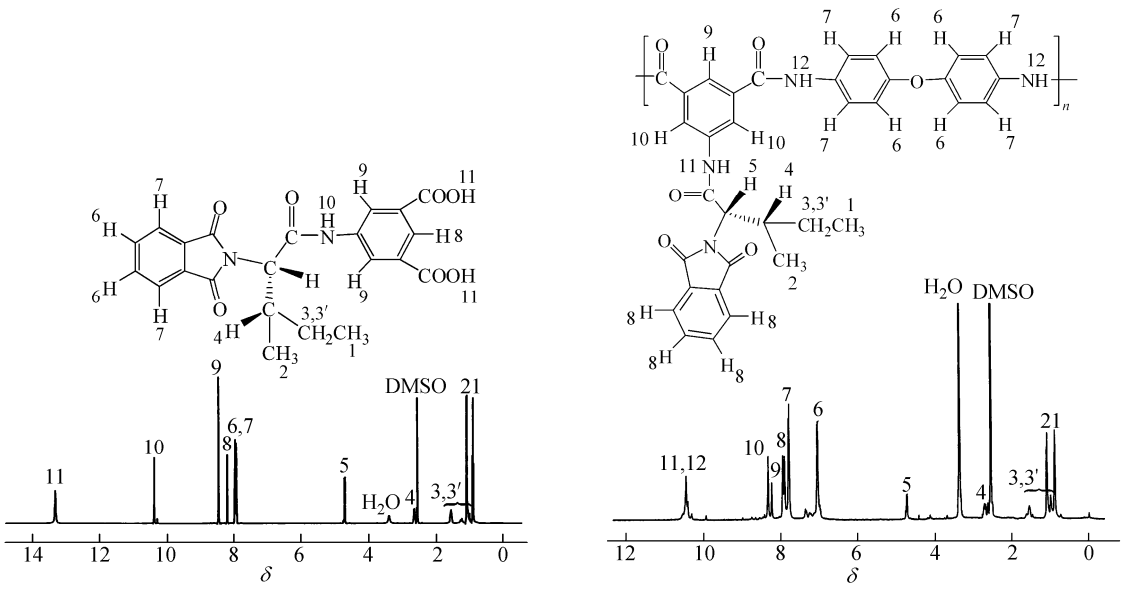


Fig. 2 ^{1}H-NMR (500 MHz) spectrum of PA9D and diacid **7** in DMSO-d_6 at R.T.

Polymer Synthesis

A typical preparation of aromatic PAs was as follows: 0.19 g (4.52×10^{-4} mol) of diacid **7**, 0.08 g (4.52×10^{-4} mol) of diamine **8C** and 0.06 g (5.00×10^{-4} mol) of calcium chloride were placed in a 10 mL round-bottomed flask and then 0.5 mL (5.10×10^{-3} mol) of NMP, 0.29 g (9.00×10^{-4} mol) of triphenyl phosphite (TPP) and 0.15 mL (1.80×10^{-3} mol) of Py were added. The mixture was refluxed for 4 h under nitrogen atmosphere. The viscous reaction solution was poured into 75 mL of methanol. The precipitated polymer was collected by filtration and was dried at 80°C for 15 h under vacuum to leave 0.18 g (85%) of PA9C. Other PAs were synthesized by the aforementioned procedure.

RESULTS AND DISCUSSION

Monomer Synthesis

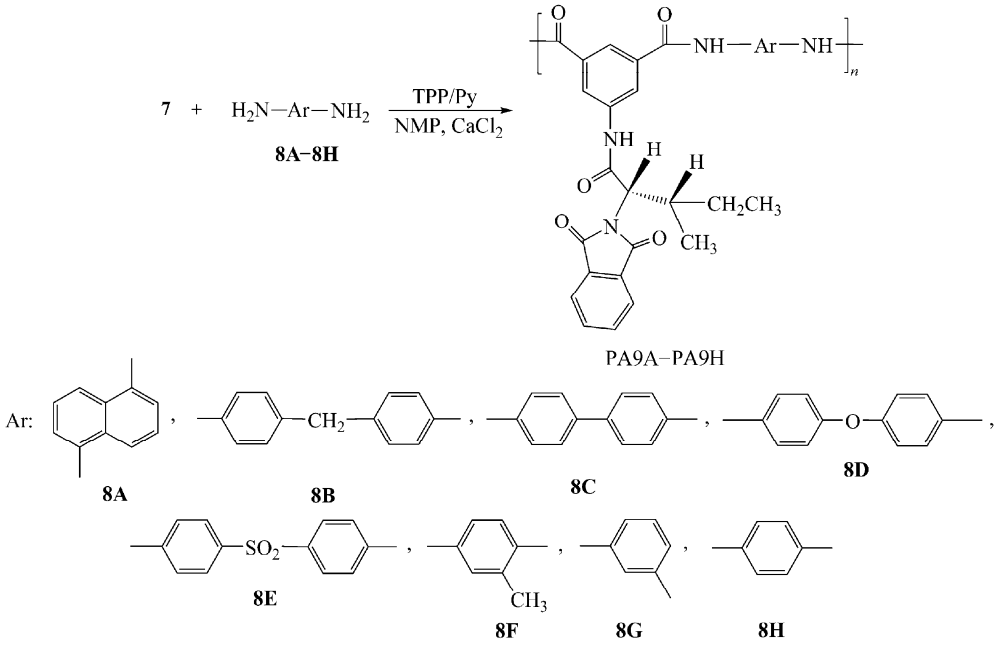
(2*S*,3*S*)-5-(3-Methyl-2-phthalimidylpentanoylamino) isophthalic acid (**7**) was prepared by the procedure as shown in Scheme 1[33]. The asymmetric diacid compound **7** was synthesized in three steps starting from **1** and **2**, which produced compound **4**. The condensation reaction of the same equimolar acid chloride **5** with 5-aminoisophthalic acid (**6**) gave the chiral monomer **7**. Chemical structure and purity of compound **7** were confirmed using elemental analysis, ^{13}C-NMR (Fig. 1) and ^{1}H-NMR (Fig. 2) spectroscopic techniques.

Polymer Synthesis

PA9A–PA9H were synthesized by solution polycondensation of an equimolar mixture of monomer 7 with eight different diamines (**8A**–**8H**) in NMP and in the presence of TPP/Py/$CaCl_2$ as shown in Scheme 2. Under the polymerization conditions employed, the reaction mixture was refluxed under nitrogen atmosphere, and then the resulting viscous mixtures were precipitated in methanol, which furnished PA9A–PA9H in high yields and moderate inherent viscosities (Table 1).

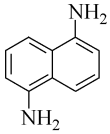
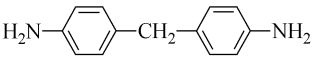
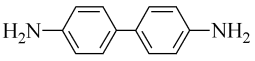
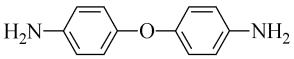
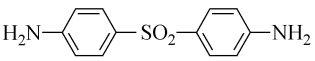
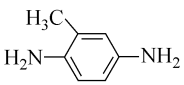
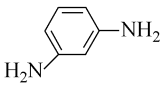
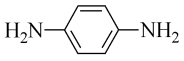
The resulting PAs were characterized by FTIR, ^{1}H-NMR and elemental analysis techniques. The results are shown in Tables 2 and 3. The ^{1}H-NMR spectra of diacid **7** and PA9D are shown in Fig. 2 for comparison in which all peaks confirm their chemical structures. The ^{1}H-NMR of PA9D showed peak for CH_3 (1) which appeared as multiplet at $\delta = 0.85$. Peaks for CH_2 (2) appeared as doublet at $\delta = 1.06$ according to their coupling with CH (4). The peaks in the region of $\delta = 0.95$–1.51 are related to diastereotopic hydrogens of CH_2 (3,3′) which appeared as multiplet. Peaks in the region of $\delta = 2.59$–2.67 are related to CH (4). Peak at $\delta = 4.70$ is assigned for CH (5) which appeared as doublet. The aromatic protons appeared in the region of $\delta = 6.98$–8.31.

The peaks in the region of δ = 10.45–10.55 are assigned for NH of amide groups. The elemental analysis results were in good agreement with calculated percentages for carbon, hydrogen and nitrogen contents in PA repeating unit (Table 3).



Scheme 2 Polycondensation reactions of monomer **7** with different diamines

Table 1. Reaction conditions for the polymerization of monomer **7** with different diamines and some physical properties of PAs [a]

Polymer	Diamine	Yield (%)	η (dL/g)	$[\alpha]_D^{25,\,e}$	$[\alpha]_{Hg}^{25,\,e,f}$
PA9A		70	0.44[b]	+12.4	+48.4
PA9B		80	0.42[c]	–20.6	–27.2
PA9C		71	0.45[d]	–18.7	–15.2
PA9D		82	0.45[b]	–	+40.9
PA9E		75	0.22[b]	–15.4	–28.5
PA9F		65	0.28[b]	+8.6	+44.9
PA9G		72	0.21[b]	+6.4	+50.2
PA9H		85	0.22[b]	+25.2	+40.9

[a] NMP was used as solvent and TPP/Py/$CaCl_2$ was used as a condensing agent; [b] Measured at a concentration of 0.5 g/dL in DMF at 25°C; [c] Measured at concentration of 0.5 g/dL in DMF containing 2% *W*/*W* LiCl (soluble fraction) at 25°C; [d] Measured at concentration of 0.5 g/dL in DMF containing 4% *W*/*W* LiCl (soluble fraction) at 25°C; [e] Measured under the same conditions as inherent viscosity; [f] Wide range was used (no filter was used for the Hg lamp).

Table 2. Characterization of PAs

Polymers	FTIR peaks (cm^{-1})	^{1}H-NMR peak (δ)
PA9A	3319 (m), 2963 (m), 1778 (m), 1713 (s), 1669 (s), 1599 (m), 1532 (s), 1494 (s), 1380 (s), 1332 (m), 1268 (m), 1079 (w), 872 (w), 784 (w), 717 (w)	
PA9B	3330 (m), 2959 (m), 1772 (w), 1717 (s), 1661 (s), 1590 (m), 1513 (s), 1442 (w), 1385 (s), 1318 (w), 1246 (w), 918 (w), 818 (w)	
PA9C	3354 (m), 2960 (m), 1776 (w), 1717 (s), 1669 (s), 1595 (m), 1505 (s), 1386 (s), 1323 (w), 1247 (w), 1080 (w), 824 (w), 722 (w)	
PA9D	3297 (m), 2964 (w), 1772 (w), 1716 (s), 1662 (m), 1597 (m), 1498 (s), 1384 (m), 1229 (m), 832 (w), 719 (w)	0.85 (distorted t, 3H, CH_3), 1.06 (d, 2H, CH_2, J = 6.5 Hz), 0.95–1.51 (m, 2H, CH_2), 2.59–2.67 (m, 1H, CH), 4.72 (d, 1H, CH, J = 8.0 Hz), 6.98 (distorted dd, 4H, Ar—H), 7.78 (distorted dd, 4H, Ar—H), 7.93 (distorted d, 4H, Ar—H), 8.22 (s, 1H, Ar—H), 8.31 (s, 2H, Ar—H), 10.45–10.55 (s, 3H, NH)
PA9E	3331 (m), 2956 (w), 1773 (w), 1715 (s), 1678 (s), 1592 (s), 1523 (s), 1447 (w), 1400 (s), 1386 (s), 1320 (s), 1245 (s), 1183 (w), 1152 (s), 1108 (s), 1071 (m), 902 (w), 836 (w), 722 (m), 695 (m), 571 (m)	
PA9F	3299 (m), 2965 (m), 1774 (w), 1715 (s), 1669 (s), 1596 (s), 1522 (s), 1446 (m), 1384 (s), 1284 (m), 1071 (w), 895 (w), 719 (m), 530 (w)	
PA9G	3318 (m), 2956 (m), 1778 (w), 1716 (s), 1663 (s), 1605 (s), 1540 (s), 1485 (s), 1442 (m), 1382 (s), 1331 (m), 1241 (s), 1080 (m), 912 (w), 720 (w), 686 (w)	0.83 (distorted t, 3H, CH_3), 1.03 (distorted d, br, 2H, CH_2), 0.92–1.51 (m, 2H, CH_2), 2.58 (m, 1H, CH), 4.74 (distorted d, 1H, CH), 6.80 (m, 1H, Ar—H), 7.01–7.32 (m, br, 3H, Ar—H), 7.86 (distorted dd, 4H, Ar—H), 8.21 (s, 1H, Ar—H), 8.30 (s, 2H, Ar—H), 10.30 (s, 1H, NH), 10.48 (s, 2H, NH)
PA9H	3319 (m), 2965 (m), 1778 (m), 1714 (s), 1666 (s), 1604 (s), 1541 (s), 1486 (s), 1443 (m), 1382 (s), 1330 (m), 1244 (s), 1081 (m), 909 (w), 720 (w), 688 (w)	

All the polymers showed FTIR absorption peaks at 3300, 2960, 1770–1780, 1713–1717, 1661–1678, 1380–1386 and 717–722 cm^{-1} corresponding to the amide N—H, aliphatic C—H, amide carbonyl groups and imide, respectively. FTIR spectrum of PA9D is shown in Fig. 3.

Table 3. Elemental analysis of PA9B and PA9C

Polymer		Formula		C (%)	H (%)	N (%)
Without terminal groups	PA9B	$C_{35}H_{30}N_4O_5$	Calcd.	71.66	5.15	9.55
		586.64 g/mol	Found	70.98	5.11	9.44
	PA9C	$C_{34}H_{28}N_4O_5$	Calcd.	71.32	4.93	9.78
		572.61 g/mol	Found	71.04	4.72	9.65
With terminal groups	PA9B	$C_{35}H_{32}N_4O_6$	Calcd.	69.52	5.33	9.27
		604.65 g/mol	Found	70.98	5.11	9.44
	PA9C	$C_{34}H_{30}N_4O_6$	Calcd.	69.14	5.12	9.49
		590.63 g/mol	Found	71.04	4.72	9.65

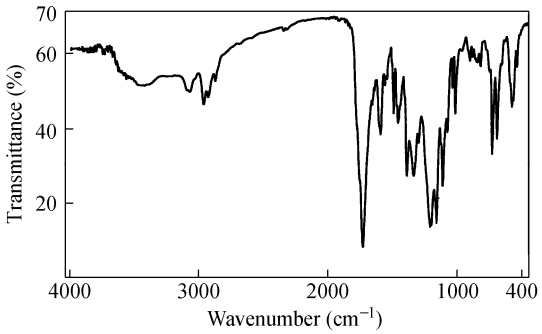


Fig. 3 FTIR (KBr) spectrum of PA9D

The incorporation of chiral units into polymer side chains was confirmed by measuring their specific rotation with diverse source of lamps and it was shown that polymers prepared by the reaction of the same monomer with different diamines showed different specific rotations (Table 1). This is a normal behavior for all optically active compounds, that optical rotation is highly dependent on the chemical structures of the resulting polymeric materials, any small changes in the chemical structures of any chiral molecules have substantial random effect on the optical rotations and is not predictable[34]. Therefore, in this investigation during the polymerization of diacid **7** with different aromatic diamines different molecular structures could be formed. All of the PAs demonstrate optical rotation and thus are optically active.

Thermal Properties

Thermal properties of PAs were evaluated with TGA under a nitrogen atmosphere (Table 4). Thermal stability of the polymers was studied based on 5% and 10% weight loss temperatures (T_5, T_{10}) of the polymers and residues at 600°C (char yield). Figure 4 shows the TGA curves for PA9B and PA9C. TGA data show that, the thermal stability of the resulting polymers are rather high. It could be related to aromatic and rigid structure of diamines and bulky imide pendant groups. Because of the more rigidity of PA9C it should show higher thermal stability.

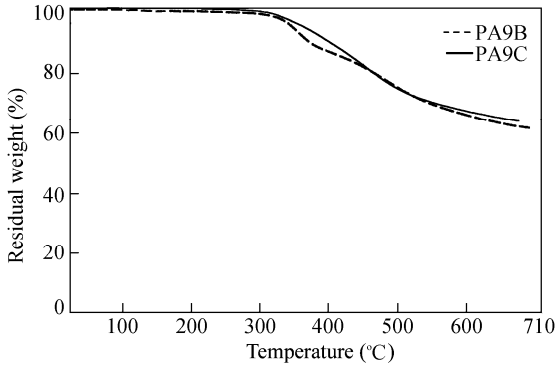


Fig. 4 TGA thermograms of PA9B and PA9C under a nitrogen atmosphere at heating rate of 10 K/min

Table 4. Thermal properties of PA9B and PA9C

Polymer	T_5 (°C)[a]	T_{10} (°C)[a]	Char yield (%)[b]
PA9B	330	370	62
PA9C	345	385	64

[a] Temperature at which 5% and 10% weight loss was recorded by TGA at heating rate of 10 K/min under a nitrogen atmosphere;

[b] Weight percentage of material left undecomposed after TGA analysis at a temperature of 600°C under a nitrogen atmosphere

Solubility of PAs

The solubility properties of PAs were studied in different solvents (Table 5). Polymers could be dissolved in the amide type solvents, such as NMP, DMAc and DMF. Differences in solubility of PAs were based on the polymer's structure. They are insoluble in solvents such as water, methanol, chloroform and dichloromethane. The PA9A–PA9H show good solubility due to the fact that some diamines are flexible, and the diacid chiral monomer contains a bulky phthalimidyl group that exhibits a steric hindrance, which prevents close chain packing and allows the solvent molecules to diffuse into the polymer. Since the resulting polymers are optically active, they have potential to be used as chairal stationary phase in GC for the separation of racemic mixtures.

Table 5. Solubility of PAs[a]

Solvent	PA9A	PA9C	PA9D	PA9E	PA9F
DMF	+++	±	+++	+++	+++
DMF 4% LiCl	+++	++	+++	+++	+++
NMP	+++	±	+++	+++	+++
DMAc	+++	±	+++	+++	+++
o-Cresol	+++	+	+++	+++	+++
DMSO	+++	+	+++	+++	+++
H_2SO_4	+++	+++	+++	+++	+++
EtOH	–	–	–	–	–
H_2O	–	–	–	–	–
Acetone	–	–	–	–	–
$CHCl_3$	–	–	–	–	–
CH_2Cl_2	–	–	–	–	–
Cyclohexane	–	–	–	–	–

[a] Solubility: measured at a polymer concentration of 10 mg/mL; + Soluble at boiling temperature; ++ Soluble at boiling water bath temperature; +++ Soluble at R.T.; ± Partially soluble; – Insoluble

CONCLUSIONS

In this work, novel thermally stable and optically active PAs containing a rigid phthalimido and flexible amino acid group, using Yamazaki phosphorylation reaction were synthesized. The main advantage of this polycondensation reaction is that, this process is a one-pot reaction and it would not be required to prepare more active materials such as diacid chlorides. The resulting polymers have inherent viscosities in the range of 0.21–0.45 dL/g. On the other hands, by introducing pendant units into the chain of polymer backbone, should disturb interchain hydrogen bonding, inherent macromolecular rigidity, diminish packing efficiency and crystallinity that caused superior solubility. So, these polymers are readily soluble in polar amidic solvents. Since, these optically active polymers have natural amino acids in the polymer architecture; they are expected to be biodegradable and therefore may be classified under environmentally friendly polymers. The obtained polymers show high thermal stability although slightly diminished compared with that of related aromatic PAs which do not have any bulky units.

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