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ORIGINAL ARTICLE

Optically active: microwave-assisted synthesis and characterization of L-lysine-derived poly (amide-imide)s

Ali Reza Alborzi · Saeed Zahmatkesh · Karim Zare · Javad Sadeghi

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Abstract L-lysine hydrochloride was transformed to ethyl L-lysine dihydrochloride. This salt was reacted with trimellitic anhydride to yield the corresponding diacid (**1**). Microwave-assisted polycondensation results a series of novel Poly (amide-imide)s (**PAI$_{a-i}$**). These polymers have inherent viscosities in the range of 0.23–0.66 dl g^{-1}, display optical activity from +8.02 to +15.11 (as there is no obvious regioselectivity between alpha and epsilon amino groups of the chiral diacid during the polymerization step then random orientation of diacid moieties along the polymer backbone can be predicted and the concept of "tacticity" cannot be addressed in this research), and are readily soluble in polar aprotic solvents. They start to decompose ($T_{10\%}$) above 362°C and display glass-transition temperatures at 119–153°C. All of the above polymers were fully characterized by UV, FT-IR and ^{1}H NMR spectroscopy, elemental analysis, thermogravimetric analyses, DSC, inherent viscosity measurement and specific rotation.

Keywords Thermal properties · Optically active · L-lysine · Poly (amide-imide) · Microwave-assisted polymerization

A. R. Alborzi · K. Zare · J. Sadeghi
Department of Chemistry, Islamic Azad University, Firouzabad, Fars, Iran

S. Zahmatkesh (✉)
Department of Science, Payame Noor University (PNU), Tehran, Iran
e-mail: zahmatkesh1355@yahoo.com

Introduction

The interest for developing new biodegradable and/or biocompatible polymers, especially polyesters and polyamides, has largely encouraged the use of monomers based on naturally occurring products (Gonsalves and Mungara 1996; Steinbuchel 2002). Both carbohydrate- (Thiem and Bachmann 1994; Varela and Orgueira 1999) and amino acid- (Gonsalves and Mungara 1996) derived monomers are being currently used as building blocks to generate novel polymeric structures with enhanced biodegradability.

L-Lysine in particular has been repeatedly used for making polyamides with potential as biomaterials (Gachard et al. 1997; Saotome and Schultz 1967; Crescenzi et al. 1968; Katsarawa et al. 1985; Espartero et al. 1993). For instance, L-lysine with good functionalities has been used to prepare some polytartaramides (Majo et al. 2004).

Optically active polymers are one of the most important classes of high performance engineering materials which are suitable candidates for use as the chiral stationary phases in high performance liquid chromatography (HPLC) (Nakano 2001; Cirilli et al. 2003; Coa et al. 2007; Mallakpour and Kowsari 2005; Yuan et al. 2005) as well as asymmetric catalysis applications (Itsuno 2005; Hu et al. 2001, 2002; HB et al. 2000; Canali et al. 1999). The synthesis and application of these polymers is a considerable topic, which has been paid more attention recently (Hajipour et al. 2005). Most of the natural polymers are optically active and have special chemical activities, such as catalytic properties that exist in genes, proteins and enzymes. Some other applications are construction of chiral media for asymmetric synthesis, chiral stationary phases for resolution of enantiomers in chromatographic techniques (Akelah and Sherrington 1981), chiral liquid crystals in ferroelectrics and nonlinear optical devices

(Wulff 1989; Fontanille and Guyot 1987). These synthetic polymers based on optically pure amino acids can induce crystallinity with their ability to form higher ordered structures that exhibit enhanced solubility characteristics (Birchall et al. 2001). These properties have caused them to be good candidate for drug delivery systems, biomimetic systems, biodegradable macromolecules, biomaterials, and also as chiral purification media (Mallakpour and Yousefian 2008). So, more considerations to improve different synthetic procedures of optically active polymers exist. Recently, we have synthesized optically active polymers by different methods (Hajipour et al. 2009; Zahmatkesh and Hajipour 2009, 2010).

In this research, we report the synthesis and characterization of nine novel Poly (amide-imide)s based on L-lysine through microwave-assisted polycondensation. These polymers showed good optical activity (+8.02 to +15.11). The outstanding characteristics of these polymers include thermal stability, good solubility, optical activity, potentially being ion exchangeable.

Materials and methods

The trimellitic anhydride (Merck) was recrystallized from acetic anhydride. 1,2-phenylene diamine, 1,3-phenylene diamine, 1,4-phenylene diamine and 2,6-diamino pyridine were purified by sublimation under the reduced pressure. 4,4′-diamino diphenyl sulfone, 4,4′-diamino diphenyl ether and 4,4′-diamino biphenyl were recrystallized from EtOH/H_2O. DMF was purified by distillation under reduced pressure over barium oxide. The other chemicals (Merck) were used as received. ^{1}H NMR spectra were recorded on an 500 MHz and ^{13}C NMR on an 125 MHz (Bruker Avance) instrument, using DMSO-d_6 as solvent and tetramethylsilane as shift reference (tube diameter 5 mm). IR spectra were recorded on a Shimadzu FT-IR-680 instrument using KBr pellets. UV spectra were recorded on a Perkin-Elmer lambada 5 instrument. Specific rotations were measured by an A. Kruss. Optronic P3002 RS (Germany) Polarimeter in DMF as solvent. Thermogravimetric analyses (TGA) were recorded on a Mettler TA4000 with heating rate of 6°C min^{-1} under air atmosphere. DSC analyses were preformed on a Mettler DSC-30 under nitrogen atmosphere. Inherent viscosities of polymers were measured by a standard procedure using a Cannon–Fenske Routine Viscometer (Germany) at 25°C using DMF as solvent. Melting points were measured in open capillaries with a IA9000 series digital Melting Point Apparatus. Elemental analyses were preformed in a Heraeus CHNS-RAPID instrument.

Monomer synthesis

Synthesis of Ethyl L-lysine dihydrochloride (Majo et al. 2004)

In a 50 ml round-bottomed flask equipped with a reflux condenser and a stirring bar, 8 ml of thionyl chloride was added drop wise to the stirring absolute ethanol (2.5 ml) at −10°C. L-lysine hydrochloride (7.3 g, 0.04 mol) was added to the mixture and refluxed for 6 h. The solvent was evaporated under reduced pressure and the residue was washed with diethyl ether for three times. Yield: 87%; m.p.: 136–137°C; IR (cm^{-1}): 3,421, 3,350–2,514, 2,019, 1,740, 1,603, 1,583, 1,501, 1,217, 851, 740; ^{1}H-NMR (D_2O, ppm): 1.07 (3H), 1.29 (2H), 1.49 (2H), 1.76 (2H), 2.78 (2H), 3.91 (1H), 4.08 (2H); Elemental analysis for $C_8H_{18}N_2O_2{\cdot}2HCl$, Calculated: C (38.87%), H (8.16%), N (11.33%), Found: C (38.62%), H (8.31%), N (11.40%).

*Synthesis of ethyl L-lysine-N,N′-ditrimellitoyl diacide (**1**)*

Into a 25-ml round-bottomed flask, 2,000 g (10.42×10^{-3} mol) of trimellitic anhydride, 1.282 g (5.21×10^{-3} mol) of Ethyl L-lysine dihydrochloride, a mixture of acetic acid/pyridine (5 ml, 3:2) and a stirring bar were placed. The mixture was stirred at room temperature for 2 h and then refluxed for 6 h. The solvent was removed under reduced pressure. 5 ml of cold concentrated HCl was added. A white precipitate was formed and filtered off. The white diacid (**1**) was extracted from this crude product with chloroform. Yield: 2.32 g (79%); m.p.: 168°C; $[\alpha]_D^{25} = +3.14°$ (0.050 g in 10 ml DMF); IR (cm^{-1}): 3,400–2,400, 1,702, 1,498, 1,418, 1,296, 1,148, 1,069, 1,018, 916, 803, 750, 748; ^{1}H-NMR (500 MHz, $CDCl_3$, ppm): 1.2 (3H), 1.3 (2H), 1.7 (1H), 1.8 (1H), 2.1 (1H), 2.2 (1H), 3.7 (2H), 4.2 (2H), 4.8 (1H), 7.9 (1H), 8.0 (1H), 8.1 (1H), 8.2 (1H), 8.4 (1H), 8.5 (1H); ^{13}C NMR (125 MHz, $CDCl_3$, ppm): δ 14.4, 23.6, 27.7, 28.2, 38.0, 52.4, 62.5, 123.8, 124.1, 124.8, 125.2, 132.2, 132.4, 134.9, 135.1, 136.2, 136.4, 136.5, 136.6, 166.8, 166.9, 167.6, 167.7, 169.3, 170.1, 170.2

*Synthesis of ethyl L-lysine-N,N′-ditrimellitoyl diacylchloride (**2**)*

Into a 25-ml round-bottomed flask were placed 0.558 g (1.0×10^{-3} mol) of diacid (**1**), 5 ml (an excess amount) of thionyl chloride and two drops of DMF. The mixture was stirred for 20 min and then refluxed for 2 h. Unreacted

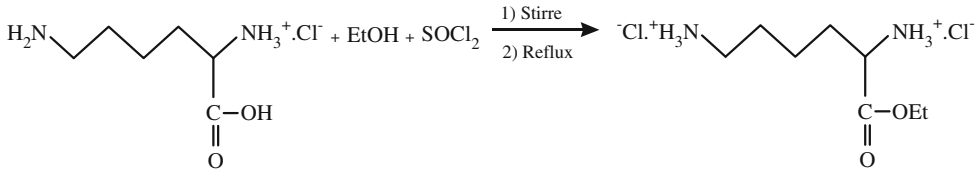
thionyl chloride was removed under reduced pressure and the residue was washed with *n*-hexane, to leave 0.486 g (82%) of white crystals. d.p.: 175°C. $[\alpha]_D^{25} = +4.08°$ (0.050 g in 10 ml DMF). IR (KBr cm^{-1}): 3,023, 2,784, 1,862, 1,785, 1,720, 1,464, 1,384, 1,226, 1,017, 922, 877, 754, 718, 680, 507.

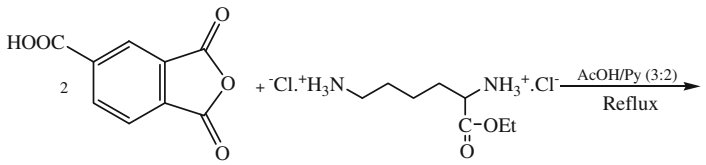
Diamine synthesis (Hajipour et al. 2007)

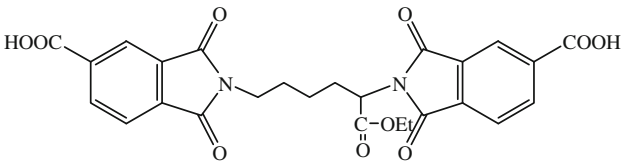
Synthesis of phenyl-2,6-bis(4-nitrophenyl) pyridine

Yield (%) = 60; m.p. (°C) >250; IR (cm^{-1}): 1,585, 1,543, 1,510, 1,375, 1,345, 1,090, 845, 815, 750, 740;

Scheme 1 Protection of L-lysine acidic group







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Scheme 2 Diacid **1** synthesis

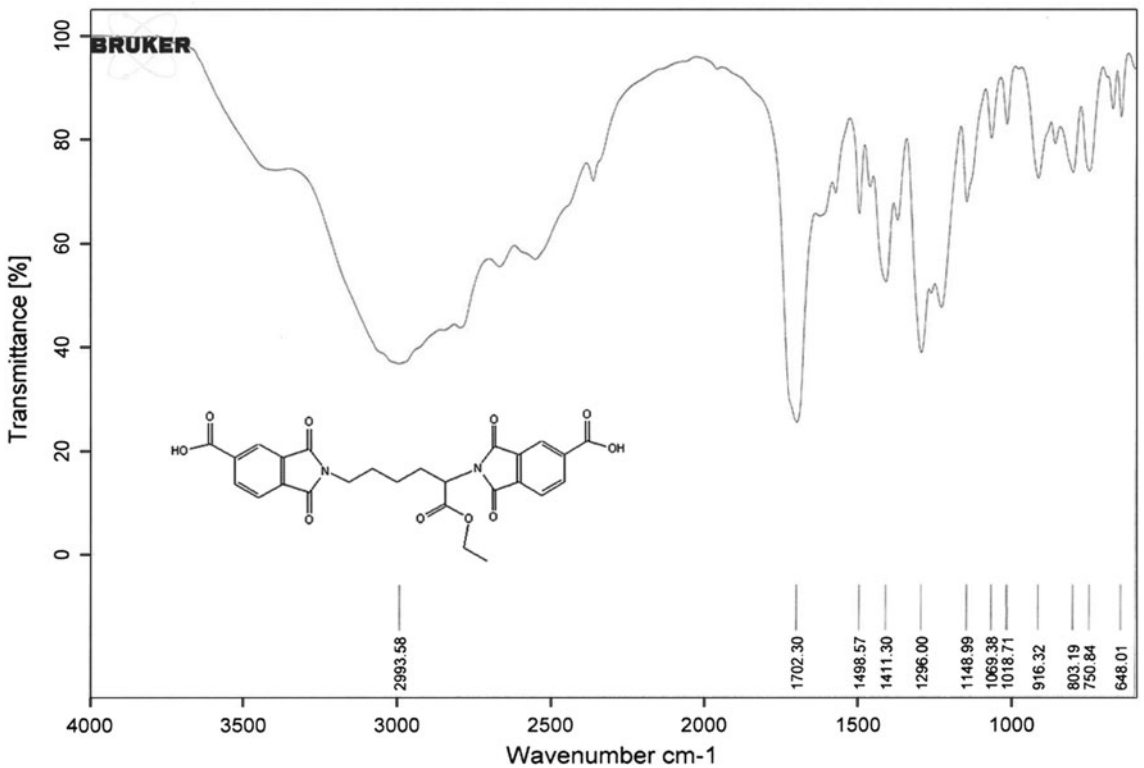


Fig. 1 IR spectrum of diacid **1**

^{1}H NMR δ: (ppm): 7.56–7.62 (m, 8H), 7.77–7.79 (s, 2H), 8.07 (dd, 2H), 8.37–8.44 (m, 3H); MS (*m*/*z*): 399, 398, 397 (M^{-+}, 100%), 351, 306, 305, 304, 302, 152, 151.

Synthesis of phenyl-2,6-bis(4-aminophenyl) pyridine

Yield (%) = 92; m.p. (°C) = 155; IR (cm^{-1}): 3,335, 3,200, 1,620, 1,580, 1,535, 1,505, 1,440, 1,385, 1,280,

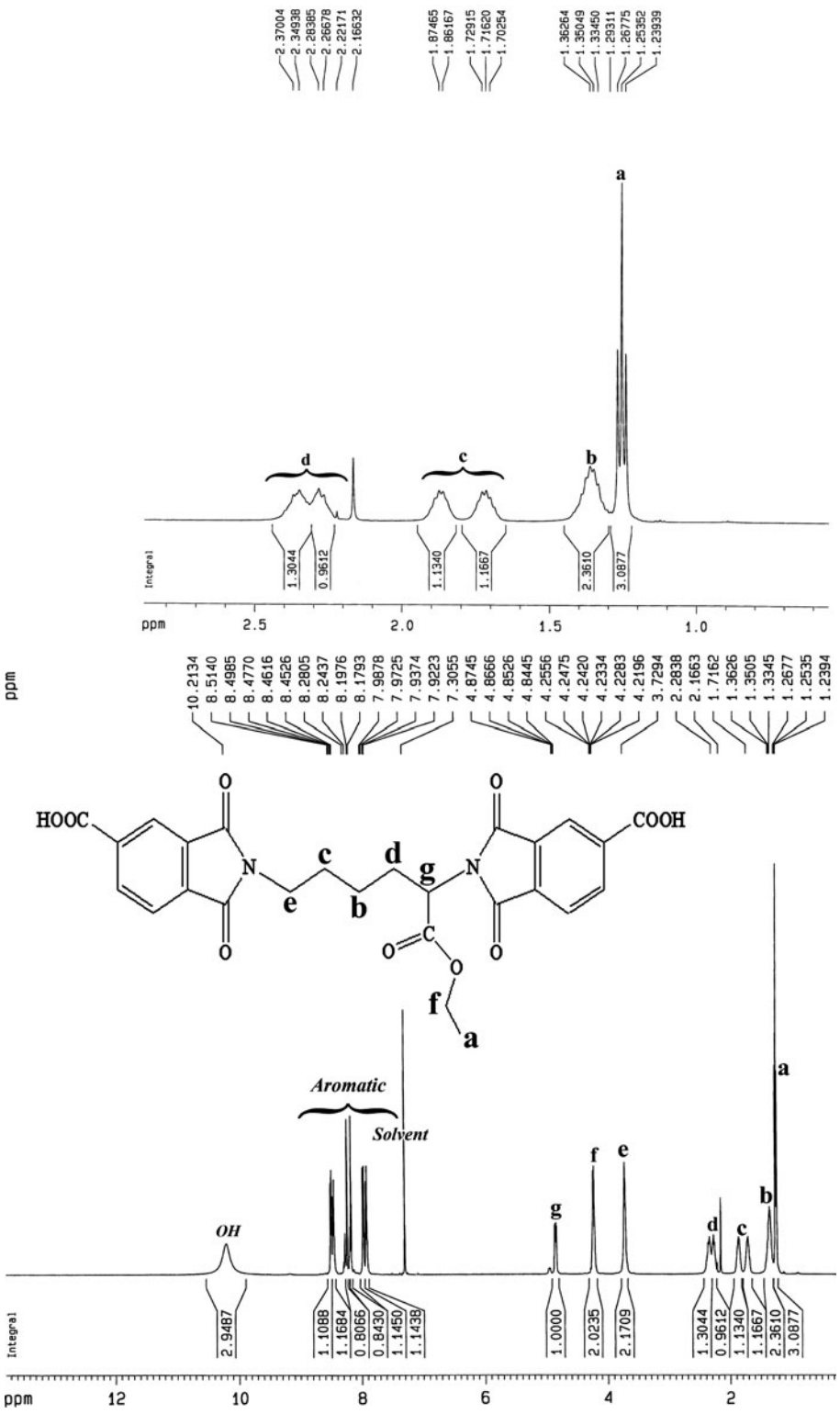


Fig. 2 ^{1}H NMR spectrum of diacid **1**

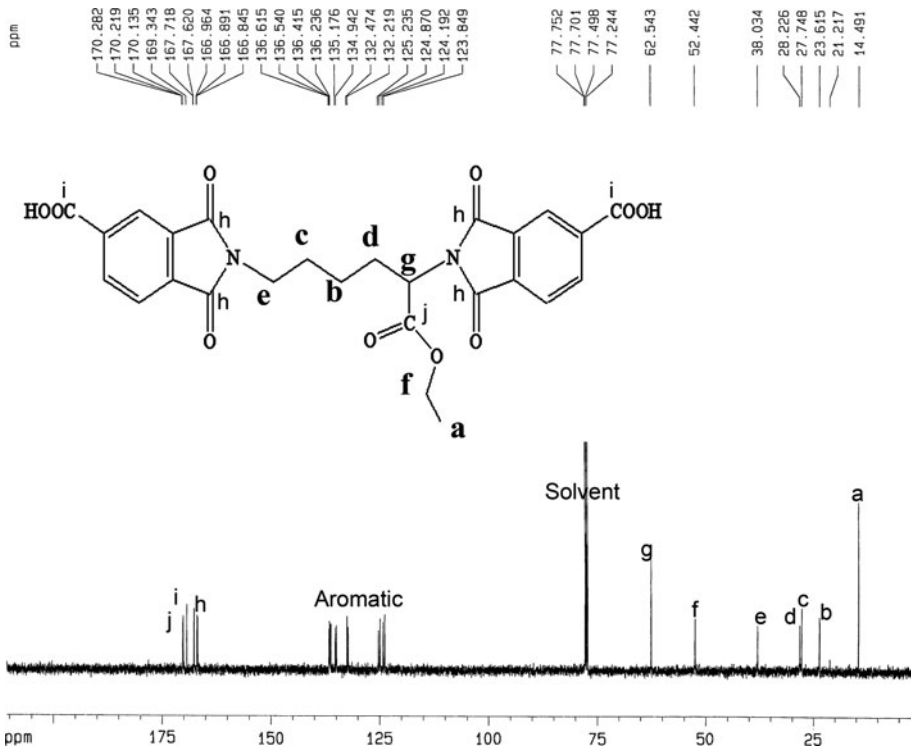
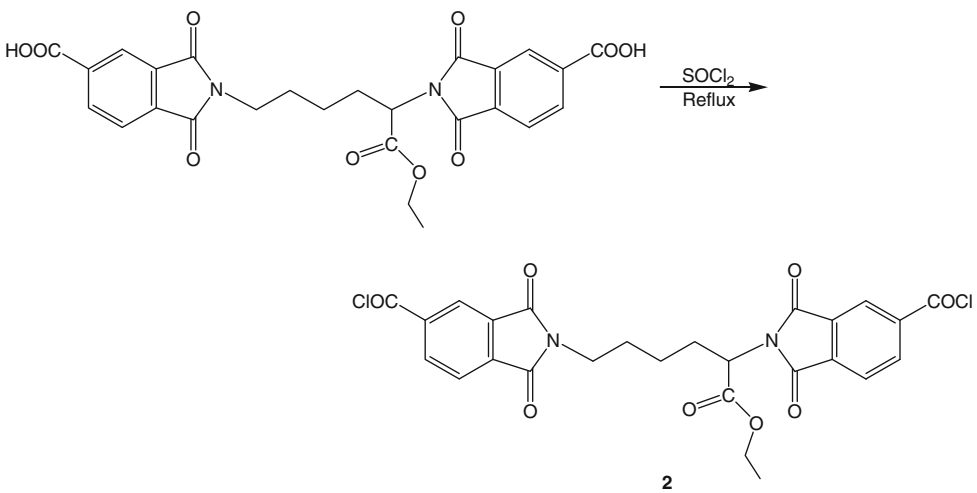


Fig. 3 ^{13}C NMR spectrum of diacid **1**



Scheme 3 Chlorination of diacid **1**

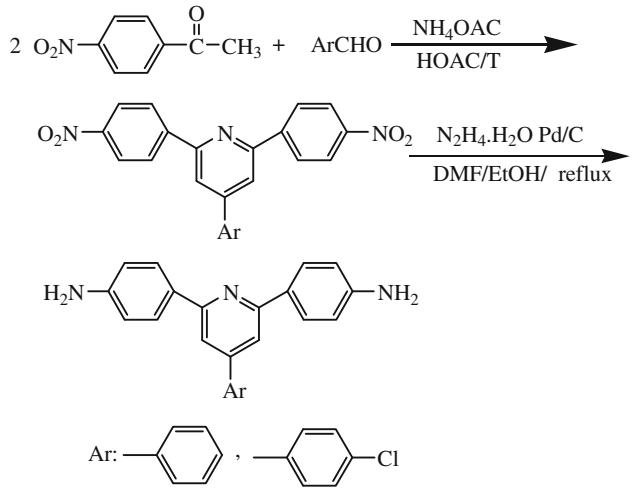
1,235, 1,175, 825, 750, 680; ^{1}H NMR δ: (ppm): 7.00–8.20 (m, 15H), 3.99 (NH2, s, 4H); MS (*m/z*): 340, 338, 337 ($M^{\cdot+}$, 100%), 336, 322, 245, 169.

Synthesis of 4-(p-chlorophenyl)-2,6-bis(4-nitrophenyl) pyridine

The same procedure was followed: recrystallized from EtOH, Yellow; Yield (%) = 55; m.p. (°C) >250; IR (cm^{-1}): 3,424, 1,595, 1,544, 1,515, 1,384, 1,343, 1,090, 1,010, 856, 822, 733, 688, 483; ^{1}H NMR δ: (ppm): 7.3 (2H), 7. 7 (2H), 7.9 (2H), 8.1 (2H), 8.4 (2H).

Synthesis of 4-(p-chlorophenyl)-2,6-bis(4-aminophenyl) pyridine

The same procedure was followed: recrystallized from EtOH, Bright yellow; Yield (%) = 90; m.p. (°C) = 378; IR (cm^{-1}): 3,500–3,000, 1,610, 1,603, 1,540, 1,519, 1,369, 1,233, 1,180, 1,091, 1,013, 824, 748, 518; ^{1}H NMR δ:



Scheme 4 Synthesis of phenyl-2,6-bis(4-aminophenyl) pyridine and 4-(*p*-chlorophenyl)-2,6-bis(4-aminophenyl) pyridine

(ppm): 3.6 (4H), 6.7 (4H), 7.3 (2H), 7.4 (2H), 7.5 (2H), 8.0 (4H); ^{13}C NMR δ: (ppm): 114.6, 115.3, 128.5, 128.8, 129.5, 130, 135, 138, 148.7, 157.6.

Microwave-assisted polymerization

General procedure into a porcelain dish, a mixture of diacyl chloride (1.0 mmol) and DABCO (1.0 mmol) as a catalyst was placed. After grounding the reagents for 5 min, diamine (1.0 mmol) was added and the mixture was ground for further 5 min. 0.25 ml of *O*-cresol as a solvent was added, and the mixture was ground for 5 min. The reaction mixture was irradiated in a microwave oven for 10 min (600 W, interval 10 s/min). The resulting homogenous glassy compound film was dissolved in DMF and then isolated by adding methanol/H_2O (50:50) and triturating, followed by filtration. It was washed several times with methanol and vacuum dried.

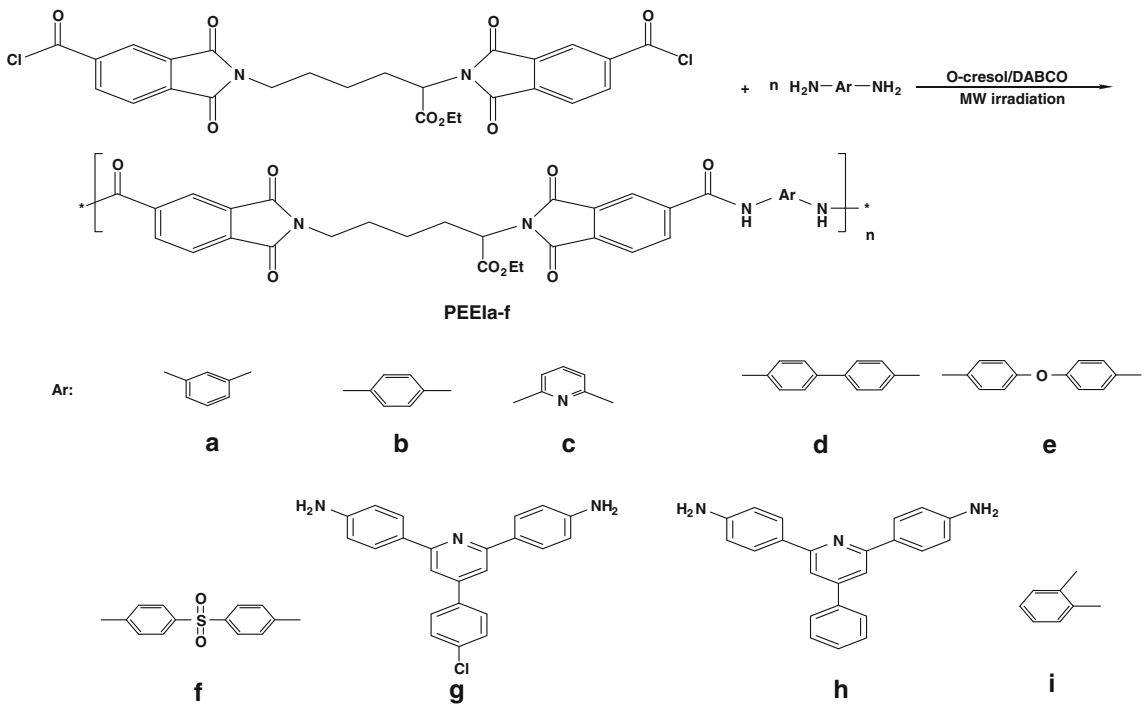
PAI$_a$: Pale yellow; yield (%) = 85; $[\alpha]_D^{25} = +10.25$; UV (λ max) = 268; IR (cm^{-1}): 3,375, 1,716, 1,544, 1,388, 1,250, 729; ^{1}H NMR δ: (ppm): 1.1 (3H), 1.3 (2H), 1.6 (2H), 2.1 (2H), 3.5 (2H), 4.1 (2H), 4.8 (1H), 6.7–8.5 (10H), 10.6 (2H).

PAI$_b$: Pale green; yield (%) = 90; $[\alpha]_D^{25} = +10.14$; UV (λ max) = 348; IR (cm^{-1}): 3,470, 1,775, 1,716, 1,517, 1,390, 1,318, 1,251, 730; ^{1}H NMR δ: (ppm): 1.1 (3H), 1.3 (2H), 1.6 (2H), 2.1 (2H), 3.5 (2H), 4.1 (2H), 4.8 (1H), 7.2–8.3 (10H), 8.5 (2H).

PAI$_c$: dark yellow; yield (%) = 70; $[\alpha]_D^{25} = +8.81$; UV (λ max) = 264; IR (cm^{-1}): 3,465, 2,933, 1,729, 1,717, 1,388, 1,281, 1,247, 1,172, 1,107, 726; ^{1}H NMR δ: (ppm): 1.1 (3H), 1.3 (2H), 1.6 (2H), 2.1 (2H), 3.5 (2H), 4.1 (2H), 4.8 (1H), 7.2–8.3 (9H), 8.4 (2H).

PAI$_d$: pale green; yield (%) = 85; $[\alpha]_D^{25} = +11.18$; UV (λ max) = 362; IR (cm^{-1}): 3,415, 1,776, 1,717, 1,501, 1,391, 1,249, 1,186, 1,106, 818, 730; ^{1}H NMR δ: (ppm): 1.1 (3H), 1.3 (2H), 1.6 (2H), 2.1 (2H), 3.5 (2H), 4.1 (2H), 4.8 (1H), 7.2–8.6 (14H), 10.6 (2H).

PAI$_e$: Pale yellow; yield (%) = 80; $[\alpha]_D^{25} = +12.01$; UV (λ max) = 264; IR (cm^{-1}): 3,371, 1,716, 1,539, 1,500,



Scheme 5 Microwave-assisted polymerization

Table 1 Optimization of microwave-assisted polycondensation on $\mathbf{PAI_a}$

Power	Time (min)	Interval (10 s min^{-1})	η_{inh} (dl g^{-1})	$[\alpha]_D^{25}$
900	15	−	0.31	+6.4
900	15	+	0.30	+6.4
600	10	+	0.36	+10.3
600	15	+	0.31	+6.3
300	20	+	0.29	+7.9
300	20	+	0.21	+8.5

Table 2 Solubility of polymers

Solvents	PAI_a	PAI_b	PAI_c	PAI_d	PAI_e	PAI_f	PAI_g	PAI_h	PAI_i
NMP	+	+	+	+	+	+	+	+	+
DMSO	+	+	+	+	+	+	+	+	+
DMAc	+	+	+	+	+	+	+	+	+
DMF	+	+	+	+	+	+	+	+	+
H_2SO_4	+	+	+	+	+	+	+	+	+
CH_2Cl_2	−	−	−	−	−	−	−	−	−
$CHCl_3$	−	−	−	−	−	−	−	−	−
EtOH	−	−	−	−	−	−	−	−	−
MeOH	−	−	−	−	−	−	−	−	−
H_2O	−	−	−	−	−	−	−	−	−

Concentration: 5 mg ml^{-1}, +, soluble at room temperature; −, insoluble at room temperature

1,388, 1,246, 1,176, 1,105, 833, 730; ^{1}H NMR δ: (ppm): 1.1 (3H), 1.3 (2H), 1.6 (2H), 2.1 (2H), 3.5 (2H), 4.1 (2H), 4.8 (1H), 6.6–8.4 (14H), 10.5 (2H).

PAI_f: Gray; yield (%) = 85; $[\alpha]_D^{25} = +13.46$; UV (λ max) = 264; IR (cm^{-1}): 3,370, 1,776, 1,716, 1,593, 1,532, 1,395, 1,320, 1,253, 1,148, 1,107, 835, 730; ^{1}H NMR δ: (ppm): 1.1 (3H), 1.3 (2H), 1.6 (2H), 2.1 (2H), 3.5 (2H), 4.1 (2H), 4.8 (1H), 6.1 (1H), 6.6 (2H), 7.5 (2H), 7.8 (2H), 7.9 (1H), 8.0 (1H), 8.1 (1H), 8.2 (1H), 8.3 (1H), 8.4 (1H),10.8 (1H), 13.8 (1H).

PAI_g: Bright yellow; yield (%) = 85; $[\alpha]_D^{25} = +12.61$; UV (λ max) = 263; IR (cm^{-1}): 3,362, 1,775, 1,716, 1,604, 1,525, 1,392, 1,250, 1,187, 843, 731; ^{1}H NMR δ: (ppm): 1.1 (3H), 1.3 (2H), 1.6 (2H), 2.1 (2H), 3.5 (2H), 4.1 (2H), 4.8 (1H), 6.6–9.2 (21H), 10.3 (2H).

PAI_h: Bright yellow; yield (%) = 90; $[\alpha]_D^{25} = +15.11$; UV (λ max) = 294; IR (cm^{-1}): 3,379, 1,775, 1,717, 1,603, 1,525, 1,387, 1,250, 1,094, 827, 731; ^{1}H NMR δ: (ppm): 1.1 (3H), 1.3 (2H), 1.6 (2H), 2.1 (2H), 3.5 (2H), 4.1 (2H), 4.8 (1H), 6.6–9.2 (20H), 10.2 (1H), 10.3 (1H).

PAI_i: Pale yellow; yield (%) = 65; $[\alpha]_D^{25} = +8.02$; UV (λ max) = 258; IR (cm^{-1}): 3,471, 1,776, 1,716, 1,388, 1,249, 732; ^{1}H NMR δ: (ppm): 1.1 (3H), 1.3 (2H), 1.6 (2H), 2.1 (2H), 3.5 (2H), 4.1 (2H), 4.8 (1H), 7.2-8.6 (10H), 10.3 (1H), 13.2 (1H).

Results and discussion

Ethyl L-lysine dihydrochloride was prepared with the reaction of a mixture of EtOH and thionyl chloride with L-lysine hydrochloride. L-lysine hydrochloride was added to the mixture drop wise at −10°C and then refluxed for 6 h. The dark solid was washed three times with diethyl ether to leave a bright with solid (87%). FT-IR spectroscopy shows a strong and broad peak at 3,350–2,514 cm^{-1} corresponding to the ammonium N–H stretching and a strong peak at 1,740 cm^{-1} corresponding to the C=O stretching of ester moiety. ^{1}H-NMR (D_2O, ppm) spectroscopy shows the corresponding peaks such as 3.91 (1H) due to the chiral center and 1.07 (3H) and 2.78 (2H) peaks due to the ethyl moiety (Scheme 1). The best solvent to prepare Ethyl L-lysine-*N*,*N*′-ditrimellitoyl diacide (**1**) was acetic acid/pyridine mixture, since we have the salt form of chiral diamine as starting material here. The best method for purification of this synthetic diacid was found to be extraction with chloroform (Scheme 2). The chemical structure of diacid **1** was confirmed by spectroscopic analysis (Figs. 1, 2, 3). FT-IR spectroscopy shows a strong and broad peak at 3,600–2,700 cm^{-1} corresponding to the COOH stretching, a strong peak at 1,740 cm^{-1} corresponding to the C=O stretching of ester moiety, a strong peak at 1,702 cm^{-1} corresponding to the symmetric C=O stretching of imidic moiety and two peaks at 1,411 and 750 cm^{-1} due to the cyclic imide groups. ^{1}H-NMR ($CDCl_3$ ppm) spectroscopy shows 14 peaks and the corresponding peaks such as 4.8 (1H) due to the chiral center and 10.21 (2H) due to the acidic moiety. ^{13}C-NMR ($CDCl_3$ ppm) spectroscopy shows 26 peaks due to the 26 different carbons. Corresponding white diacyl chloride (**2**) was prepared in refluxing $SOCl_2$ for 2 h (Scheme 3). FT-IR spectroscopy shows the corresponding carbonyl stretching of acyl chloride at 1,862 cm^{-1}. Synthesis of phenyl-2,6-bis(4-aminophenyl) pyridine and 4-(*p*-chlorophenyl)-2,6-bis(4-aminophenyl) pyridine is presented in Scheme 4.

Microwave-assisted polymerization was applied to prepare the poly (amide-imide)s (Scheme 5). To optimize the polymerization conditions, we did six experiments on $\mathbf{PAI_a}$. The optimum condition is as follow: power = 600 W; time = 10 min with interval times of 10 s min^{-1} of running. It is found that at higher power, the lower viscosity and lower specific rotation is obtained; which can be attributed to polymer degradation. At lower power, the higher specific rotation but lower viscosity is obtained; the results are shown in Table 1. All polymers have been obtained in good yields (65–90%) and high viscosity (0.23–0.66) with optical activity (+8.02 to +15.11) and being thermally stable [$T_{(10\%)}$ 0.362–425°C] High speed and high yield are the advantages of this polymerization method. As there is no obvious regioselectivity between

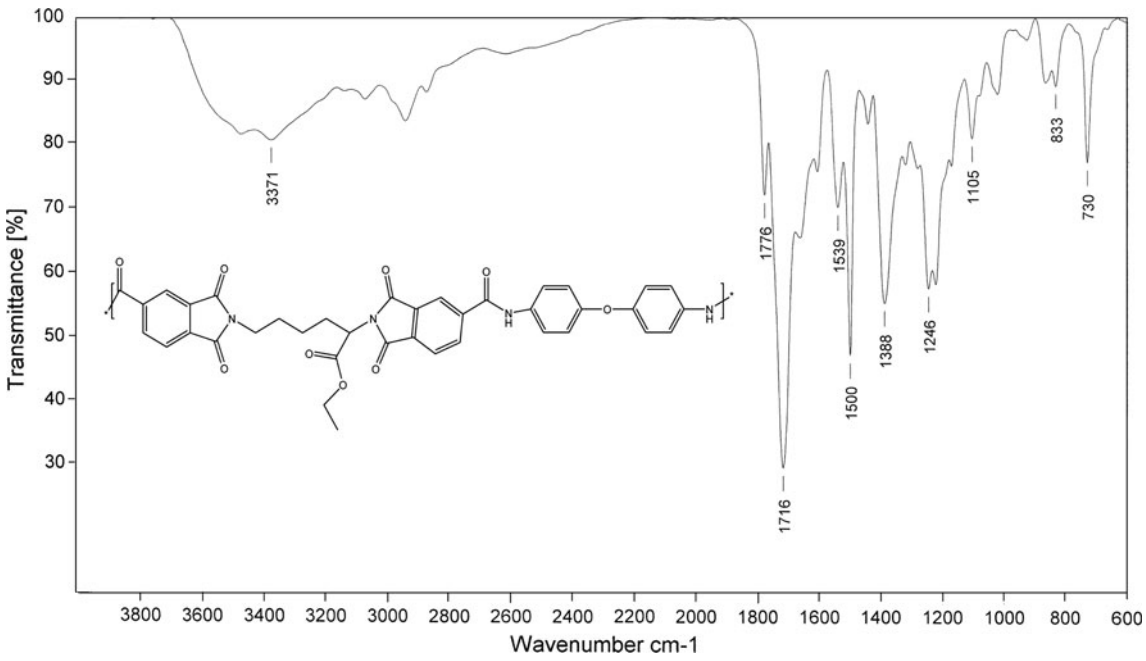
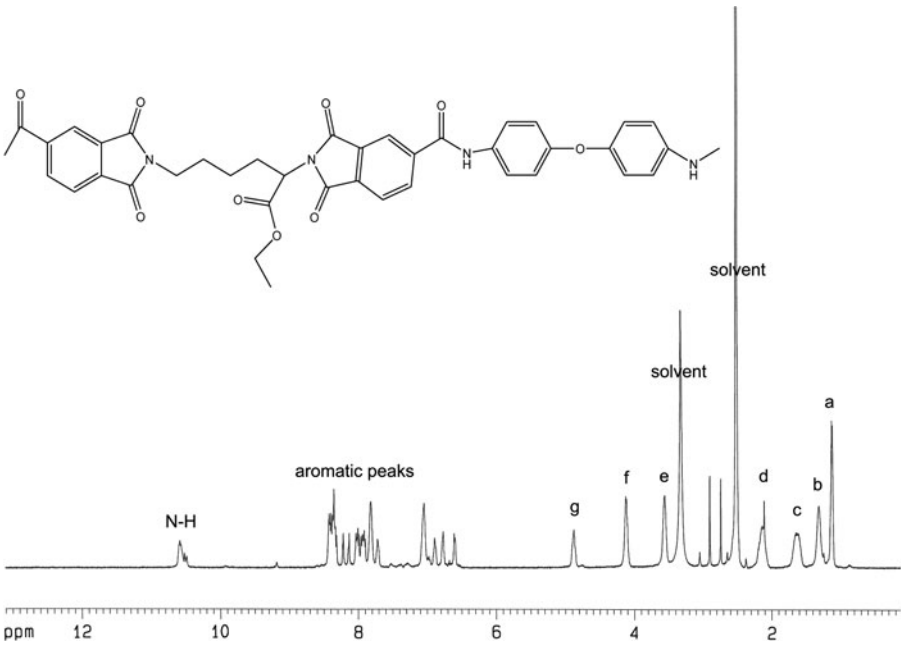


Fig. 4 FT-IR spectrum of $\mathbf{PAI_e}$

Fig. 5 ^{1}H NMR spectrum of $\mathbf{PAI_e}$



alpha and epsilon amino groups of the chiral diacid during the polymerization step, then random orientation of diacid moieties along the polymer backbone can be predicted and the concept of "tacticity" cannot be addressed in this research. Head-to-tail regiorandomness may likely affect some physical properties of the polymers such as crystallinity. These polymers are organosoluble in common polar aprotic solvents (Table 2). The novel polymers were fully characterized by spectroscopic analysis (UV, FT-IR, ^{1}H NMR), elemental analysis, thermal analysis (TGA, DSC), viscometric measurements, optical rotation measurements and solubility tests. As an example, the corresponding spectra of $\mathbf{PAI_e}$ are represented in Figs. 4, 5, 6, 7, respectively. Thermal properties of these polymers are investigated by TGA/DTG under air atmosphere at 6°C min^{-1} and by DSC under nitrogen atmosphere at

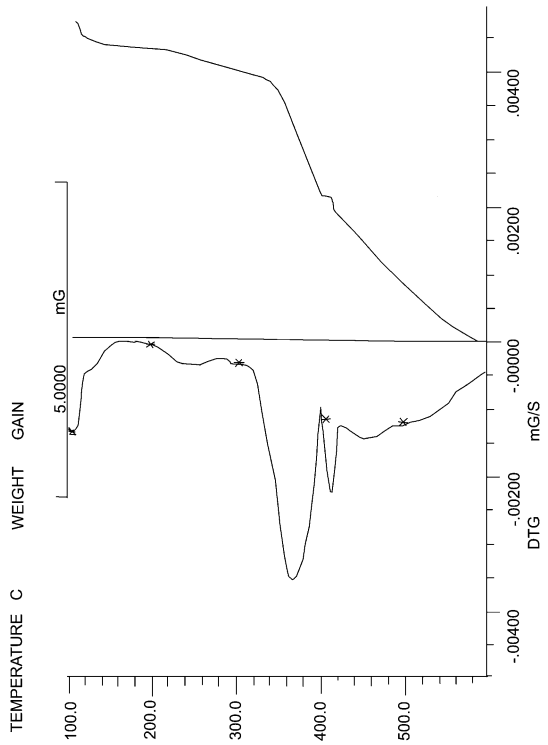


Fig. 6 TGA spectrum of $\mathbf{PAI_e}$

10°C min^{-1} (Table 3). In FT-IR spectra of these polymers some characteristic peaks could be seen, including the N–H stretching of amide group at around 3,350 cm^{-1}, C=O asymmetric stretching of imide group, the C=O symmetric stretching of imide and amide groups at 1,727–1,716 cm^{-1}, C–N stretching at around 1,387 cm^{-1}, C–N stretching at 1,530 cm^{-1}. All of these **PAI**s exhibited strong absorption at around 1,380 and 720 cm^{-1}, which shows the presence of the heterocyclic imide groups. The ^{1}H NMR spectra of **PAI**s shows a peak at 4.8 ppm due to the chiral center. The polymers show a maximum absorption in UV spectra at around 264–348 nm. The elemental analysis result for $\mathbf{PAI_d}$ is also in good agreement with calculated/expected percentages of carbon, hydrogen and nitrogen contents in the polymer-repeating unit (calculated for $C_{38}H_{28}N_4O_8$: C (68.20), H (4.20), N (8.30); found: C (67.43), H (4.56), N (7.65)). These polymers can be partially hydrolyzed to present the pendent carboxylic acid groups

Table 3 Thermal behavior of polymers

Polymer	Decomposition temperature (°C) $T^a_{10\%}$	Glass-transition temperature (T_g)[b]	Char yield (%)[c]
PAI_a	370	–	3.9
PAI_b	362	–	2.6
PAI_c	365	–	2.9
PAI_d	363	–	3.8
PAI_e	370	–	3.2
PAI_f	425	–	3.1
PAI_g	400	142	4.6
PAI_h	420	–	4.4

[a] Temperature at which 10% weight loss was recorded by TGA at a heating rate of 10°C min^{-1} under air atmosphere

[b] Glass-transition temperature

[c] Percentage weight of material left after TGA analysis at maximum temperature 600°C under air atmosphere

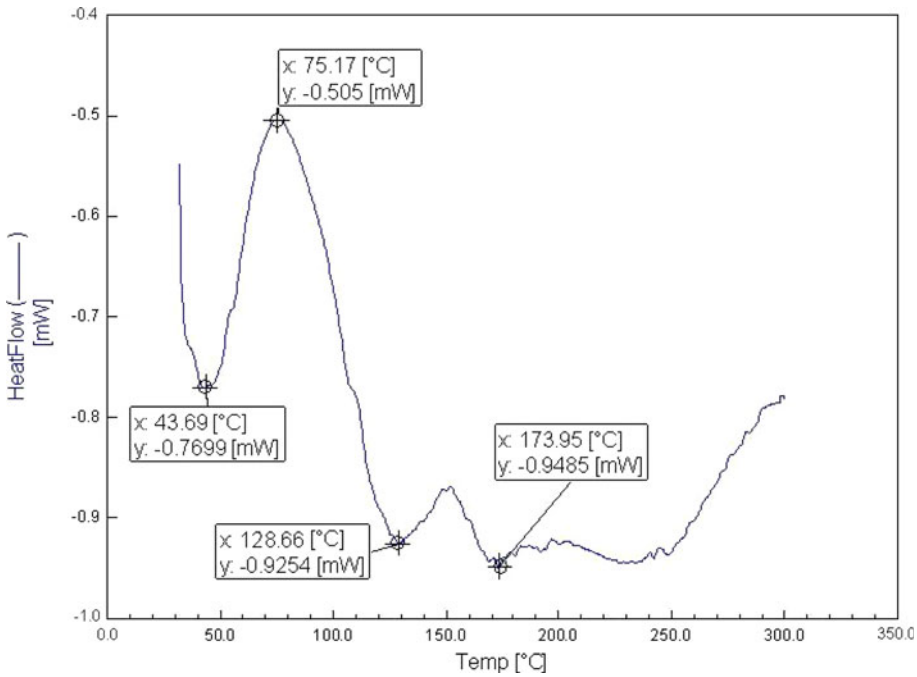


Fig. 7 DSC spectrum of $\mathbf{PAI_e}$

(ion-exchangeable polymers). Solubility and the flexibility of these polymers are enhanced by incorporating the soft segment in the polymer backbone.

Conclusions

A fast and effective procedure to prepare a serious of optically active poly (amide-imide)s containing ethyl L-lysine ester has been introduced. Since there is no obvious regioselectivity between alpha and epsilon acyl chloride groups of the lysine ester containing diacid during the polymerization step then random orientation of lysine moieties along the polymer backbone can be predicted. These polymers are very soluble, optically active, potentially ion exchangeable and thermally stable. The resulting polymers are identified by FT-IR, UV, ^{1}H NMR spectroscopy and elemental analysis; and characterized by yield of reaction, inherent viscosity, and specific rotation.

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