



ORIGINAL ARTICLE

New optically active poly(amide–imide)s from *N*-trimellitylimido-*L*-amino acid and 1,2-bis[4-aminophenoxy]ethane in the main chain: Synthesis and characterization



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Received 21 May 2011; accepted 2 September 2011

Available online 20 September 2011

KEYWORDS

Optically active;
Poly(amide–imide);
Polycondensation;
L-amino acid

Abstract Six new optically active poly(amide–imide)s (PAIs) with good inherent viscosities were synthesized from the direct polycondensation reaction of *N*-trimellitylimido-*L*-amino acids with 1,2-bis[4-aminophenoxy]ethane by direct polycondensation in a medium consisting of *N*-methyl-2-pyrrolidone (NMP)/triphenyl phosphite (TPP)/calcium chloride (CaCl₂)/pyridine (py). Diamine was synthesized by using a two-step reaction. At first 1,2-bis[4-nitrophenoxy]ethane was prepared from the reaction of two equimolars 4-nitrophenol and one equimolar 1,2-dibromo ethane and the dinitro compound was reduced by using Pd/C. Also *N*-trimellitylimido-*L*-amino acids were synthesized by the condensation reaction of trimellitic anhydride with two equimolars of various *L*-amino acids in acetic acid solution. The polymerization reactions produced a series of optically active PAIs with a high yield and good inherent viscosity. The resulted polymers were fully characterized by means of FTIR and ¹H NMR spectroscopy, elemental analyses, inherent viscosity, specific rotation, solubility tests, thermogravimetric analysis (TGA), and a derivative of thermogravimetric (DTG) analysis.

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1. Introduction

Aromatic poly(amide–imide)s possess desirable characteristics with the merits of both polyamides and polyimides, for example, high thermal stability and good mechanical properties as well as easy processability. Conventionally, aromatic poly(amide–imide)s can be prepared in several ways starting from trimellitic anhydride (TMA), such as two-step polycondensation from the acid chloride of TMA with aromatic diamines

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involving polyaddition and subsequent cyclodehydration (Imai et al., 1985), low temperature solution polycondensation of TMA-derived imide ring-preformed diacid chloride and aromatic diamines (Wrasidlo and Augl, 1969), polycondensation of TMA or TMA-derived imide ring-containing dicarboxylic acids with diisocyanates (Kakimoto et al., 1988), and phosphorylation polyamidation between TMA-derived imide ring-preformed dicarboxylic acids and aromatic diamines (Hsiao et al., 1994). Several series of novel poly(amide-imide) also have been readily prepared by direct polycondensation (Faghihi and Hagibeygi, 2005; Faghihi and Hajibeygi, 2004; Liou and Hsiao, 2001; Yang et al., 2001; Faghihi and Naghavi, 2005).

The synthesis and application of chiral polymers is of particular interest from the viewpoint of material science and newly considered topics. Chiral polymers have found successful uses in chromatographic separation of enantiomers, chiral liquid crystals, non-linear optical devices, optical switches, and biomedical devices, etc (Mallakpour and Kolahdoozan, 2007; Murano et al., 2000; Fu et al., 2007; Itsuno, 2005; Liaw et al., 2007; Hajipour et al., 2005, 2007; Faghihi et al., 2009, 2010a; Mallakpour et al., 2001). A direct and effective way for synthesizing the chiral polymers is to introduce chiral elements into the polymer backbone or side chains. The combination of poly(amide-imide)s with chiral elements is of synthetic interest and may also lead to chiral recognition membranes. Recently, we have synthesized optically active polymers by different methods (Faghihi et al., 2004, 2010b).

A triphenyl phosphite (TPP)-activated polycondensation (phosphorylation reaction) technique for the synthesis of polyamides was reported by Yamazaki et al. (1975). In this article, a series of new optically active PAIs **8a-f** containing dibenzalacetone moiety were synthesized by reactions of six chiral *N*-trimellitylimido-*L*-amino acids **3a-f** with 1,2-bis[4-

aminophenoxy]ethane **7** by direct polycondensation in a medium consisting of *N*-methyl-2-pyrrolidone (NMP)/triphenyl phosphite (TPP)/calcium chloride (CaCl₂)/pyridine (py).

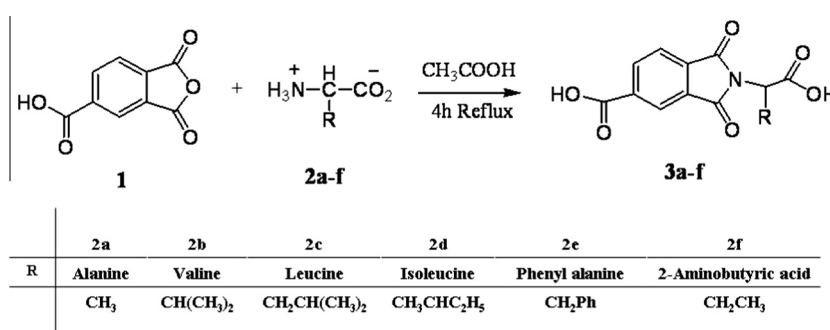
2. Experiments

2.1. Materials

Trimellitic anhydride **1**, *L*-alanine **2a**, *L*-valine **2b**, *L*-leucine **2c**, *L*-isoleucine **2d**, *L*-phenyl alanine **2e** and *L*-2-aminobutyric acid **2f**, 1,2-dibromo ethane **4**, and 4-nitrophenole **5** (from Merck) were used without further purification. Solvents: *N*-methyl-2-pyrrolidone (NMP; from Fluka), pyridine (from Acros) and triphenyl phosphite (TPP; from Merck) were used as received. Commercially available calcium chloride (CaCl₂; from Merck) was dried under vacuum at 150 °C for 6 h.

2.2. Techniques

¹H NMR and ¹³C NMR spectra were recorded on a Bruker 300 MHz instrument (Germany). Fourier transform infrared (FTIR) spectra were recorded on Galaxy series FTIR 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 Trade Mark 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 N₂ atmosphere at a rate of 10 °C/min. Elemental analy-



Scheme 1 Synthesis of diacids **3a-f**.

Table 1 Synthesis of chiral diacid derivatives **3a-f**.

Entry	Amino acid compound	R	Mp (°C)	Yield (%)	[α] _D ²⁵ ^a
3a	<i>L</i> -alanine	CH ₃	272–275	92	122.4
3b	<i>L</i> -valine	(CH ₃) ₂ CH	208–210	94	128.9
3c	<i>L</i> -leucine	(CH ₃) ₂ CHCH ₂	194–197	92	117.3
3d	<i>L</i> -isoleucine	(C ₂ H ₅)(CH ₃)CH	200–203	94	131.5
3e	<i>L</i> -phenyl alanine	PhCH ₂	215–218	90	128.7
3f	<i>L</i> -2-aminobutyric acid	CH ₃ CH ₂	234–237	95	139.8

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

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