Synthesis and Characterization of Novel Poly(amide-ester-imide)s Based on Bis(p-Amidobenzoyl Chloride)-N-trimellitylimido-L-leucine

Shadpour E. Mallakpour*, Abdol-Reza Hajipour and Ramin Vahabi
Organic Polymer Chemistry Research Laboratory, College of Chemistry, Isfahan University of Technology, Isfahan, 84156, I.R. Iran

Received 16 May 2001; accepted 5 August 2001

ABSTRACT

N-Trimellitylimido-L-leucine 3 was reacted with thionyl chloride and diacid chloride 4 was obtained in quantitative yield. The reaction of this diacid chloride with p-aminobenzoic acid was performed in dry THF and bis(p-amidobenzoic acid)-N-trimellitylimido-L-leucine 5 was obtained as an optically active aromatic imide-amide diacid in high yield. The diacid 5 was converted to diacid chloride 6 in quantitative yield by reacting with thionyl chloride. The polycondensation reaction of the new monomer imide-amide diacid chloride 6 with several aromatic diols such as bisphenol-A 7a, 4,4'-hydroquinone 7b, phenol phthalein 7c, 1,4-dihydroxyanthraquinone 7d, 1,8-dihydroxyanthraquinone 7e, and 2,4-dihydroxyacetophenone 7f was carried out under phase transfer conditions. The resulting novel poly(amide-ester-imide)s (PAEI)s having inherent viscosities 0.10–0.29 dL/g were obtained in high yield and are optically active and thermally stable. All of the above compounds were fully characterized by IR spectroscopy, elemental analyses and specific rotation.

Key Words: bis(p-amidobenzoic chloride)-N-trimellitylimido-L-leucine, poly(amide-ester-imide)s, phase transfer catalysis, optically active polymers, inherent viscosities, thermally stable polymers

INTRODUCTION

Aromatic polyimides and their copolymers are well known as high-performance polymer materials for their excellent mechanical and electrical properties, high thermal and thermooxidative stability, and outstanding solvent resistance [1, 2]. However, most aromatic polyamides are intractable materials that do not melt before thermal decomposition, therefore, would be difficult for processing. One way to decrease the melting point of polyimids is incorporation of a flexible spacer which has been reported in the synthesis of poly(ester-imide)s [3].

The synthesis and application of optically...
active polymers are the newly considerable topics, which have been surrounded with much attention recently, as polymers with chiral structures are biologically very important. Most of the natural polymers are optically active and have special chemical activities such as catalytic properties that exist in genes, proteins and enzymes. Recently, we have synthesized optically active polymers by different methods [4-16].

In previous papers [17-18] we synthesized a series of novel optically active poly(ester-imides) PEIs. The resulting polymers are optically active and have inherent viscosities in a range of 0.35-1.12 dL/g.

In this article for the first time we wish to report the synthesis and characterization of new optically active PAEIs from aromatic diols and bis(p-amidobenzoic acid)-N-trimellitylimido-L-leucine 5, a monomer containing preformed imide and amide groups, under phase transfer conditions.

**EXPERIMENTAL**

**Materials**

All chemicals were purchased from Fluka Chemical Co., Aldrich Chemical Co. and Riedel-deHaen AG. Trimellitic anhydride 1 was synthesized by the dehydration of 1,2,4-benzene tricarboxylic acid with acetic anhydride in boiling acetic acid. 1,4-Dihydroxyanthraquinone 7d, and 1,8-dihydroxyanthraquinone 7e were purified by recrystallization from ethanol, acetic acid, respectively. The other diols were used as obtained without further purification.

**Techniques**

Proton nuclear magnetic resonance (\(^1\)H NMR, 90 MHz) spectra were recorded on a Varian EM-390 instrument. Tetramethylsilane (TMS) was used as an internal reference. IR Spectra were recorded on a Shimadzu 435 IR spectrophotometer. Spectra of the 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), shoulder (sh), strong (s) and broad (br). Inherent viscosities were measured by a standard procedure using a Cannon Fensk routine viscometer. Specific rotations were measured by a Perkin-Elmer-241 polarimeter. Thermal gravimetric analysis (TGA) data for polymers were taken on a Mettler TGA-50 in N\(_2\) atmosphere at a rate of 10 °C/min. Elemental analyses were performed by Research Institute of Petroleum Industry, Tehran, I.R. Iran.

**Monomer Synthesis**

**N-Trimellitylimido-L-leucine Diacid Chloride 4**

Into a 50 mL round-bottomed flask 1.00 g (3.27x10\(^{-2}\) mol) of N-trimellitylimido-L-leucine 3, 10 mL (an excess amount) of thionyl chloride and a stirring bar were placed. The stirrer was started and the mixture was refluxed for 1 h. The reaction mixture was stirred at room temperature for 2 h. The thionyl chloride was removed via distillation and 20 mL of n-hexane was added and the mixture was heated and n-hexane was distilled off and the solid was collected, dried under vacuum to give 1.12 g (100 %) of white solid, mp = 91-92 °C, \([\alpha]_D^{25} = -48.2^\circ\) (0.050 g in 10 mL DMF). IR (KBr): 2950 (s), 2910 (m), 2860 (m), 1800 (s), 1780 (s), 1750 (s), 1720 (s), 1620 (w), 1470 (m), 1420 (w), 1380 (s), 1340 (m,sh), 1270 (w), 1210 (m), 1180 (m), 1170 (m), 1130(m), 1100 (s), 1075 (m), 1050 (m), 990 (s), 940 (m), 920 (w), 860 (m), 840 (s), 820 (m), 770 (s), 720 (m), 710 (m), 680 (m), 620 (w), 600 (w), 540 (w), 500 (w), 470 (w), cm\(^{-1}\). Elemental analysis calculated for C\(_{15}\)H\(_{13}\)C\(_{12}\)N\(_{2}\)O\(_{4}\): C, 52.65%; H, 3.83% and N, 4.09%. Found: C, 53.20%; H, 4.10% and N, 4.20%.

**Bis(p-Amidobenzoic Acid)-N-trimellitylimido-L-leucine 5**

Into a 25 mL round-bottomed flask 0.30 g (8.77x10\(^{-4}\) mol) of N-trimellitylimido-L-leucide diacid chloride 4, 5 mL of dry THF and a stirring bar were placed. The stirrer was started and the mixture was cooled in an ice bath and a solution of 0.24 g (1.75 x 10\(^{-4}\) mol) of p-aminobenzoic acid in 4 mL of dry THF was added dropwise in a period of 5 min. A yellow precipitate was formed then it was turned to light pink colour. The reaction mixture was stirred at room temperature for over night and a white precipitate was formed. The reaction mixture was concentrated up to 2 mL and the solid was filtered off and dried under vacuum to give 0.453 g (95%) of white solid.

Recrystallization from hot methanol gave white crystal, mp> 275 °C (dec.), \([\alpha]_D^{25} = +17.6^\circ\) (0.050 g in
10 mL DMF). IR (KBr): 3450 (m, sh), 3250 (s, br), 3100 (s, br), 2950 (s), 2850 (s, sh), 2650 (m), 2550 (m), 1770 (m), 1720 (s), 1680 (s), 1600 (s), 1520 (s), 1410 (s), 1370 (s), 1310 (s), 1290 (s), 1250 (s), 1170 (s), 1100 (m), 1080 (m), 1010 (w), 990 (w), 920 (w), 850 (m), 770 (m), 720 (m), 690 (w), 650 (w), 600 (w), 540 (w), cm⁻¹; ¹H NMR (DMSO-d₆, TMS, 90 MHz): δ 1.00 (m, br, 6H), 1.76 (s, br, 3H), 4.80 (m, br, 1H), 7.40-9.20 (m, 11H), 10.50 (s, 1H), 10.90 (s, 1H), 12.70 (s, br, 2H) ppm. Elemental analysis calculated for C₂₉H₂₅N₃O₈: C, 64.08%; H, 4.64% and N, 7.73%. Found: C, 63.80%; H, 5.10% and N, 7.30%.

**Bis(p-Amidobenzoyl Chloride)-N-trimellitylimido-L-leucine**

Into a 50 mL round-bottomed flask 0.20 g (3.68x10⁻⁴ mol) of diacid 5, 5 mL (an excess amount) of thionyl chloride, 10 mL of ethyl acetate and a stirring bar were placed. The stirrer was started and the mixture was refluxed for 1 h and the reaction mixture was stirred at room temperature for further 2 h. The thionyl chloride was removed via distillation and 20 mL of n-hexane was added and the mixture was heated and n-hexane was distilled off and the solid, was collected, dried under vacuum to give 0.213 g (100%) of white solid, mp=105 °C, [α]D₂⁵ = −0.8 (0.050 g in 10 mL DMF). IR (KBr): 3320 (m), 3180 (m), 3100 (m), 2950 (m), 2900 (m), 2850 (m), 1775 (s), 1720 (s), 1620 (w), 1590 (s), 1520 (s), 1470 (m), 1405 (s), 1370 (s), 1320 (s), 1250 (s), 1210 (s), 1170 (s), 1090 (m), 1010 (s), 880 (s), 840 (m), 810 (m), 770 (w), 720 (m), 640 (m), 620 (w), 600 (w), 500 (w), cm⁻¹. Elemental analysis calculated for C₂₉H₂₃Cl₂N₃O₆: C, 60.01%; H, 3.99% and N, 7.24%. Found: C, 60.90%; H, 4.20% and N, 7.00%.

**Polymerization: Synthesis of Polymer 8a**

The PAEIs were prepared by phase transfer technique using the following general procedure. Taking polymer 7a as an example. Into a 25 mL round-bottomed flask fitted with a magnetic stirrer, was placed 0.0393 g (1.723x10⁻⁴ mol) of diol 6a, 0.0392 g (1.723x10⁻⁴ mol) of benzyltriethyl ammonium chloride and 1 mL of 0.33 M NaOH. The stirrer was started and to this mixture a solution of 0.10 g (1.723x10⁻⁴ mol) of diacid chloride in 4 drops of DMF and 0.4 mL of dry chloroform was added dropwise in a period of 1 min. The reaction mixture was stirred at room temperature for 1.5 h. The resulted polymer was poured into 50 mL of methanol. The precipitated polymer was collected by filtration, and dried at 80 °C for 6 h under vacuum to leave 0.1053 g (83.0 %) of white solid 7a, mp >260 °C (dec.); IR (KBr): 3320 (m), 2940 (m), 2900 (m, sh), 2850 (m, sh), 1775 (m), 1720 (s), 1680 (m, sh), 1590 (s), 1510 (s), 1405 (m), 1375 (m), 1320 (m), 1260 (s), 1220 (m), 1165 (s), 1060 (m), 1010 (m), 850 (m, sh), 760 (m), 720 (m), 690 (w), 620 (w), 510 (w), cm⁻¹.

The other PAEIs 8b-8f were prepared in a procedure similar to that above.

**Polymer 8b**

A grey solid polymer with mp=>250 °C (dec.), was obtained; IR (KBr): 3320 (m), 3150 (m, sh), 3050 (m), 2950 (m), 2900 (m, br), 2850 (m), 1770 (m), 1715 (s), 1595 (s), 1520 (s), 1410 (m), 1380 (m), 1250 (m), 1220 (m), 1160 (s), 1050 (m), 980 (m), 880 (w), 850 (m), 760 (m), 720 (m), 690 (m), 620 (w), 510 (w), cm⁻¹.

**Polymer 8c**

A white solid polymer with mp=>260 °C (dec.), was obtained; IR (KBr): 3300 (m), 3150 (m, sh), 3050 (m), 2900 (m), 2850 (m), 1770 (m), 1720 (s), 1685 (m), 1595 (s), 1510 (s), 1460 (m), 1430 (w), 1405 (m), 1370 (m), 1320 (m), 1250 (m), 1200 (s), 1160 (s), 1115 (m), 1100 (m), 1060 (m), 1010 (m), 960 (m), 925 (m), 840 (m), 750 (m), 720 (m), 690 (m), 580 (w), 530 (w), cm⁻¹.

**Polymer 8d**

An orange solid polymer with mp = >190 °C (dec.), was obtained; IR (KBr): 3300 (m, br), 2950 (m), 2900 (m), 2850 (m), 1775 (m), 1720 (s), 1670 (s), 1630 (m), 1590 (s), 1520 (s), 1450 (m), 1405 (m), 1370 (m), 1360 (m), 1310 (m), 1250 (s), 1210 (s), 1160 (s), 1050 (m), 1020 (m), 1000 (m), 850 (m), 785 (m), 750 (m), 720 (m), 690 (m), 650 (w), 570 (w), cm⁻¹.

**Polymer 8e**

A pale-green solid polymer with mp=>190 °C (dec.), was obtained; IR (KBr): 3320 (m), 3200 (m), 3050 (m), 2950 (m), 2900 (m), 2850 (m), 1775 (s), 1720 (s).
Synthesis and Characterization of Novel Poly(amide-ester-imide)s Based on N-Trimellitylimido-L-leucine (3) has been reported in literature, and we used a modified procedure [19] for preparation of the diacid (3). Thus, the unsymmetric diacid compound (3) was synthesized by the condensation reaction of one equimolar of anhydride (1) with one equimolar of L-leucine (2) in toluene and in the presence of triethylamine as a base (Scheme I).

The diacid 3 was reacted with thionyl chloride and the diacid chloride 4 was obtained in high yield (Scheme II). The chemical structure and purity of the optically active diacid chloride 4 was proved using elemental analysis and IR spectroscopic techniques. The reaction of this diacid chloride 4 with p-amino-benzoic acid was performed in dry THF at 0°C.

The resulting novel optically active aromatic imide-amide diacid 5 was obtained in high yield (Scheme II) and its chemical structure and purity were proved using elemental analysis, IR, and 1H NMR spectroscopic techniques.

The diacid 5 was reacted with an excess amount of thionyl chloride and diacid chloride 6 was

Polymer 8f
A grey solid polymer with mp = >190 °C (dec.), was obtained; IR (KBr): 3200 (m), 3200 (m), 3100 (m), 2950 (m), 2900 (m), 2850 (m), 1780 (s), 1720 (s), 1640 (s), 1590 (s), 1520 (s), 1410 (s), 1370 (s), 1320 (s), 1240 (s), 1220 (s), 1160 (s), 1125 (s), 1070 (m), 1050 (s), 980 (m), 850 (m), 790 (w), 760 (m), 720 (m), 690 (m), 630 (w), 600 (w), 560 (w), cm⁻¹.

RESULTS AND DISCUSSION

Monomer Synthesis

\[
\text{HO-C} \quad \text{O} \quad \text{O} \quad \text{+} \quad \text{NH}_2\text{CHCOH} \quad \text{Toluene} \quad \text{Et}_3\text{N} \quad \text{HO-C} \quad \text{NHCHCOH} \\
\text{CH}_2 \quad \text{CH} \quad \text{CH}_3 \quad \text{CH}_2 \quad \text{CH} \quad \text{CH}_3
\]

Scheme I

or other isomer

3

[Image 0x0 to 405x580]
obtained in quantitative yield (Scheme II). The chemical structure and purity of the optically active diacid chloride 4 was proved using elemental analysis and IR spectroscopic techniques.

**Polymer Synthesis**

PAEIs 8a-8f were synthesized by the phase transfer polycondensation reactions of an equimolar mixture of monomer 6 with six different aromatic diamines 7a-7f using benzyltriethyl ammonium chloride as a phase transfer agent (Scheme III).

The synthesis and some physical properties of these novel optically active PAEIs are listed in Table 1. All the polymerizations readily proceeded in a heterogeneous condition and the resulted polymers were precipitated in methanol. All of the polymers were obtained in good yields with moderate inherent viscosities of 0.10–0.29 dL g⁻¹ and showing optical rotation, therefore they are optically active. We also tried the polymerization of diacid chloride 6 with diol 7a under solution conditions, thus the monomer 6 was allowed to react with diol 7a in N-methyl pyrrolidone.
Synthesis and Characterization of Novel Poly(amide-ester-imide)s Based on

6 + HO−Ar−OH \xrightarrow{\text{NaOH/BTEAC}} H_2O/DMF, CHCl_3

\[ 7a-7f \]

\[ \begin{array}{c}
\text{O} \\
\text{C} \\
\text{Ar} \\
\text{O} \\
\text{NH} - \text{C} \\
\text{CH_2} \\
\text{CH} \\
\text{C_3} \\
\text{H} \\
\end{array} \]

\[ \text{n} \] \[ 8a-8f \]

Scheme III

(NMP) at low temperature and then it was heated up to 120 °C under nitrogen atmosphere, but the resulted product was soluble in methanol and no polymeric material was obtained. Similarly the above reaction was repeated under refluxing NMP for 10 min, but no polymeric material was obtained.

Polymer Characterization

The formation of PAEIs was confirmed by IR spectroscopy analysis. As an example, the IR spectrum of PAEIs 7b (Figure 1) showed the characteristic absorptions of amide and imide groups around 3300, 1780, 1715 and 1660 cm\(^{-1}\), peculiar to N−H stretching

| Table 1. Synthesis and some physical properties of PAEIs 8a−8f. |
|------------------|------------------|------------------|
| **Diol** | **Polymer** | **Polymer Yield (%)** | **\( \eta_{\text{inh}} \text{ (dL/g)}^{a} \)** | **[\alpha]_D^{b} \text{ cm}^{-1} \)** | **Colour\(^c \)** |
| 7a | 8a | 83 | 0.29 | -0.4 | W |
| 7b | 8b | 94 | 0.22 | -0.8 | G |
| 7c | 8c | 75 | 0.12 | -0.6 | W |
| 7d | 8d | 77 | 0.16 | -1.6 | P-O |
| 7e | 8e | 78 | 0.10 | -1.0 | P-G |
| 7f | 8f | 60 | 0.10 | -0.6 | G |

(a) Measured at a concentration of 0.5 g/dL in DMF at 25 °C; (b) Measured at a concentration of 0.5 g/dL in DMF at 25 °C; (c) W= white, G= grey, P-O= pale-orange, P-G= pale-grey
Table 2. Elemental Analysis of PAEIs 8a–8f.

<table>
<thead>
<tr>
<th>Polymer</th>
<th>Formula</th>
<th>Elemental analysis (%)</th>
<th>Moisture intake (%)*</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>C</td>
<td>H</td>
</tr>
<tr>
<td>8a</td>
<td>((\text{C}44\text{H}32\text{N}3\text{O}8)_{n})</td>
<td>Calcd 71.82</td>
<td>5.07</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Found 68.60</td>
<td>5.00</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Corr(^a) 70.60</td>
<td>4.85</td>
</tr>
<tr>
<td>8b</td>
<td>((\text{C}35\text{H}22\text{N}3\text{O}8)_{n})</td>
<td>Calcd 68.06</td>
<td>4.41</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Found 63.80</td>
<td>4.50</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Corr(^a) 65.99</td>
<td>4.35</td>
</tr>
<tr>
<td>8c</td>
<td>((\text{C}34\text{H}22\text{N}3\text{O}10)_{n})</td>
<td>Calcd 71.26</td>
<td>4.27</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Found 69.30</td>
<td>4.30</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Corr(^a) 71.04</td>
<td>4.19</td>
</tr>
<tr>
<td>8d</td>
<td>((\text{C}35\text{H}22\text{N}3\text{O}10)_{n})</td>
<td>Calcd 69.07</td>
<td>3.91</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Found 64.70</td>
<td>4.10</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Corr(^a) 66.27</td>
<td>4.00</td>
</tr>
<tr>
<td>8e</td>
<td>((\text{C}35\text{H}22\text{N}3\text{O}10)_{n})</td>
<td>Calcd 69.07</td>
<td>3.97</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Found 65.40</td>
<td>4.40</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Corr(^a) 67.00</td>
<td>4.29</td>
</tr>
<tr>
<td>8f</td>
<td>((\text{C}35\text{H}22\text{N}3\text{O}10)_{n})</td>
<td>Calcd 67.37</td>
<td>4.43</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Found 65.30</td>
<td>4.50</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Corr(^a) 67.08</td>
<td>4.38</td>
</tr>
</tbody>
</table>

(a) Moisture intake (%) = \((\text{W}_0 - \text{W})/\text{W}_0 \times 100\), \(\text{W}_0\) = weight of polymer sample after standing at room temperature and \(\text{W}\) = weight of polymer sample after being dried in vacuum at 100 °C for 10 h; (b) Corrected value for C and N = found value × (100 + moisture intake)/100, and corrected value for H = found value × (100 - moisture intake)/100.

and carbonyls stretching of imide, ester and amide, respectively. All of these PAEIs exhibited absorption at 1380 cm\(^{-1}\) and 710–720 cm\(^{-1}\), which show the presence of the imide heterocycle in these polymers. Elemental analysis values of the resulting polymers...
Synthesis and Characterization of Novel Poly(amide-ester-imide)s Based on

Table 3. Solubility of PAEIs 8a–8f.

<table>
<thead>
<tr>
<th>Solvents</th>
<th>8a</th>
<th>8b</th>
<th>8c</th>
<th>8d</th>
<th>8e</th>
<th>8f</th>
</tr>
</thead>
<tbody>
<tr>
<td>DMAc</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
</tr>
<tr>
<td>DMF</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
</tr>
<tr>
<td>NMP</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
</tr>
<tr>
<td>DMSO</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
</tr>
<tr>
<td>H2SO 4</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
</tr>
<tr>
<td>MeOH</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>EtOH</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>CHCl 3</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>CH2Cl 2</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>H2O</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

(a) Concentration: 5 mg mL⁻¹; (+) soluble at room temperature, (-) insoluble.

are listed in Table 2. PAEIs derived from monomer 6 may range in colour from white to pale-orange.

The solubility of PAEIs was tested quantitatively in various solvents and are listed in Table 3. All of the PAIs are soluble in organic solvents such as DMF, DMAc, DMSO, NMP and H2SO 4 at room temperature, and are insoluble in solvents such as chloroform, methylene chloride, methanol, ethanol and water.

Thermal Properties
The thermal properties of PAEIs 8a–8c were evaluated by means of TGA/DTG in nitrogen atmosphere. All of these polymers show similar decomposition behaviour (Figures 2–4). Table 4 summarizes the thermal properties of some of the PAEIs. The PAEIs exhibited good resistance to thermal decomposition around 300°C in nitrogen and began to decompose gradually above that temperature. The temperature of 5 % weight loss for all the polymers ranged from 235 to 306 °C, the temperature of 10 % weight loss for all the polymers ranged from 345 to 376 °C and the residual weight for these polymers at 600 °C ranged from 33.37 % to 39.37 % in nitrogen atmosphere.

Figure 3. TGA/DTG Thermograms of PAEIs (8b) in N2 atmosphere.

Figure 4. TGA/DTG Thermograms of PAIs (8c) in N2 atmosphere.
Table 4. Thermal behaviour of aromatic PAEIs 8a–8c.

<table>
<thead>
<tr>
<th>Polymer</th>
<th>Decomposition temperature (°C) T&lt;sub&gt;s&lt;/sub&gt;</th>
<th>Char yield (%)&lt;sup&gt;b&lt;/sup&gt;</th>
</tr>
</thead>
<tbody>
<tr>
<td>8a</td>
<td>241</td>
<td>33.37</td>
</tr>
<tr>
<td>8b</td>
<td>306</td>
<td>38.16</td>
</tr>
<tr>
<td>8c</td>
<td>235</td>
<td>39.37</td>
</tr>
</tbody>
</table>

(a) Temperature at which 5% weight loss was recorded by TGA at a heating rate of 10°C/min in N<sub>2</sub>; (b) Percentage weight of material left undecomposed after TGA analysis at maximum temperature 800°C in N<sub>2</sub>.

CONCLUSION

A series of optically active PAEIs having inherent viscosities of 0.10–0.29 dL/g were synthesized for the first time by phase transfer catalysis of the optically active bis(p-amidobenzyol chloride)-N-trimellitylimido-L-leucine 6 as a diacid chloride having a pre-formed imide ring and amide group as an “enlarged” monomer containing one chiral L-leucine group with some aromatic diol. These aromatic PAEIs show optical rotation and are readily soluble in various organic solvents and have good thermal stability.

ACKNOWLEDGEMENTS

We wish to express our gratitude to the Research Affairs Division of Isfahan University of Technology, Isfahan, for financial support. We thank Amin Pharmaceutical Center, Isfahan, I.R. Iran for recording optical rotations.

REFERENCES

N,N'-bis(phthaloyl-L-leucine) diacid chloride with aromatic
of novel optically active poly(ester-imide)s with benzo-
phenone linkages by microwave-assisted polycondensation", 
19. Mallakpour S. E., Hajipour A. R. and Roohipour-fard, R.,
"Direct polycondensation of N-trimellitylimido-L-leucine with