

Microwave-Assisted Polycondensation of 4,4'-(Hexafluoroisopropylidene)-*N,N'*-bis(phthaloyl-L-leucine) Diacid Chloride with Aromatic Diols

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ABSTRACT: A new facile and rapid polycondensation reaction of 4,4'-(hexafluoroisopropylidene)-*N,N'*-bis(phthaloyl-L-leucine) diacid chloride (**1**) with several aromatic diols such as phenol phthalein (**2a**), bis phenol-A (**2b**), 4,4'-hydroquinone (**2c**), 1,4-dihydroxyanthraquinone (**2d**), 1,8-dihydroxyanthraquinone (**2e**), 1,5-dihydroxy naphthalene (**2f**), dihydroxy biphenyl (**2g**), and 2,4-dihydroxyacetophenone (**2h**) was performed by using a domestic microwave oven in the presence of a small amount of a polar organic medium such as *o*-cresol. The polymerization reactions proceeded rapidly, compared with the conventional solution polycondensation, and was completed within 10 min, producing a series of optically active poly(ester-imide)s with quantitative yield and high inherent viscosity of 0.50–1.12 dL/g. All of the above polymers were fully characterized by IR, elemental analyses, and specific rotation. Some structural characterization and physical properties of this optically active poly(ester-imide)s are reported. © 2000 John Wiley & Sons, Inc. *J Appl Polym Sci* 77: 3003–3009, 2000

Key words: microwave-assisted rapid polycondensation; optically active polymers; poly(ester-imide)s; microwave oven; inherent viscosity

INTRODUCTION

Polyimides are one of the most superior engineering plastics and characterized by high thermal stability, good mechanical properties, and excellent chemical resistance. Because of the highly coplanar and symmetric aromatic imide structure, polyimides usually possess high glass transition temperature and high melting temperature which induce serious difficulties in

melt processing. Five ways have been proposed to decrease the melting point of polyimides: 1. introducing a substituted group,^{1,2} 2. introducing a meta or ortho-substituted aromatic structure,^{3–8} 3. adding a flexible swivel linking group in the main chain,^{9–12} 4. introducing a crank shaft unit,^{13–15} and 5. introducing a flexible spacer.^{16–21} The effects of introducing a flexible spacer have been reported in the investigations of polyesters,¹⁵ poly(ester-imide)s,²² and poly(ester-amide)s.^{8,16–21}

Since 1775, photochemical, catalytic, sonic and high-pressure techniques²³ have been added to the chemists' repertoire for accelerating chemical reactions. In this decade, a new technique provided an alternative to conventional conductive heating for introducing energy into reactions. The microwave dielectric heating effect uses the abil-

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ity of some liquids and solids to transform electromagnetic energy into heat and thereby drive chemical reactions. This *in situ* mode of energy conversion has many attractions to the chemist, because its magnitude depends on the properties of the molecules. This allows some control of the materials' properties and may lead to selectivity of the reaction. Application of microwave should not be confused with gas phase reactions where a microwave discharge can create a plasma with a very high temperature and can cause dramatic fragmentation and recombination reactions.

Much important work has been performed on the synthesis of the useful processes using microwave dielectric heating effects. These include the preparation of samples for organic chemistry,^{24–26} preparation of samples for analysis,^{27–29} application to waste treatment,^{30,31} polymer technology,^{32–34} drug release targeting,³⁵ ceramics,³⁶ and alkane decomposition.³⁷

On the other hand, synthesis and application of optically active polymers are the newly considered topics that have been given more attention recently. The polymers with chiral structures are biologically very important; other applications are: 1. constructing chiral media for asymmetric synthesis, 2. chiral stationary phases for resolution of enantiomers in chromatographic techniques, and 3. chiral liquid crystals in ferroelectric and nonlinear optical devices.^{38–41} Recently, we have synthesized optically active polymers by different methods.^{42–45}

In this article we report a facile method for rapid synthesis of aromatic optically active poly(ester-imide)s by using a domestic microwave oven and comparing with conventional solution polymerization results.

EXPERIMENTAL

Apparatus

The apparatus used for the polycondensation was a Samsung domestic microwave oven (2450 MHz, 900 W; South Korea). IR spectra were recorded on a Shimadzu 435 IR spectrophotometer (Japan). Spectra of solids were performed using KBr pellets. Vibrational transition frequencies are reported in wave number (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-Fensck Routine Viscometer (Ger-

many). Specific Rotations were measured by a Perkin Elmer-241 Polarimeter (Germany). Thermal gravimetric analysis (TGA) data for polymers were taken on a Stanton-650 TGA (UK) under N_2 atmosphere at a rate of $20^\circ\text{C}/\text{min}$ and differential scanning calorimetry (DSC) data were recorded on a DSC-PL-1200 instrument (UK) under N_2 atmosphere at a rate of $20^\circ\text{C}/\text{min}$ by the Iran Polymer Institute.

Reagents and Monomers

All chemicals were purchased from Fluka Chemical Co., Aldrich Chemical Co., and Riedel-de-Haen AG. 4,4'-(hexafluoroisopropylidene) bis(phthalic anhydride) was kindly obtained from Hoescht-Celanese as electronic grade material and was used as received. 1,4-dihydroxyanthraquinone (**2d**), 1,8-dihydroxyanthraquinone (**2e**), and 1,5-dihydroxy naphthalene (**2f**) were purified by recrystallization from ethanol, acetic acid, and nitromethane, respectively. The other diols were used as obtained without further purification.

4,4'-(hexafluoroisopropylidene)-*N,N'*-bis(phthaloyl-L-leucine) diacid chloride (**1**) was prepared by the condensation of L-leucine with 4,4'-(hexafluoroisopropylidene) bis(phthalic anhydride), followed by reaction with thionyl chloride, according to our previously reported procedure.⁴⁵ The yield of the diacid chloride **1** was 96.1%, m.p. = 118°C and $[\alpha]_{\text{D}}^{25} = -29.92^\circ\text{C}$ [0.052 g in 10 mL *N,N*-dimethylformamide (DMF)].

Preparation of Poly(ester-imide)s

The polymerization reaction was performed by reaction of monomer **1** with different diols via two methods.

Solution Polymerization

To a three-necked reactor equipped with thermometer and purging nitrogen gas, was added a mixture of one equimolar of monomer **1** with diols **2a–2c**, 1,2-dichlorobenzene was also charged in the reaction mixture in 0.7 molar concentration. The reaction solution was stirred at refluxing temperature for 24 h and the resulting polymer solution was poured into 50 mL of water. The precipitated polymer was filtered and dried under vacuum for 15 h at 100°C .

Microwave-Assisted Polymerization

In this method, the sample cell was a mortar containing a mixture of reactants dissolved in

Table I Some Reaction Conditions and Physical Properties of Polymer 3a Resulting from the Reaction of Diacid Chloride 1 with Diol 2a in Different Solvents Under Microwave Irradiation

Diols	Solvent	ϵ	b.p.	v/t^a	Polymer	
					Yield (%)	η_{inh}^b
2a	1,2-dichlorobenzene	9.93	179	0.1/5	27.3	0.14
2a	<i>m</i> -cresol	11.5	200	0.1/5	48.0	0.16
2a	<i>o</i> -cresol	11.5	191	0.1/5	57.5	0.21

^a v , volume (mL); t , irradiation time (min).^bMeasured at a concentration of 0.5 g/dL in DMF at 25°C.

various solvents that was placed on the center of the turn table in the microwave oven with a power of 2450 MHz that was irradiated in a specific time listed in Tables I and II and Figure 1. The resulting polymer solution was triturated into 20 mL of methanol. The precipitated polymer was washed thoroughly with methanol and dried at 80°C under vacuum for 15 h. Other poly(ester-imide)s were synthesized analogously and characterized by IR analysis.

Polymer 3a

IR (KBr): 3460 (w, sh), 3390 (w, br), 3075 (w), 3025 (w), 2950 (m), 2910 (m, sh), 2860 (w), 1755 (s, br), 1720 (s, br), 1680 (s, sh), 1670 (m, sh), 1610 (m), 1600 (m, sh), 1500 (w), 1490 (w), 1455 (w), 1435 (w), 1375 (m), 1325 (w), 1280 (m, sh), 1250 (m, br), 1200 (w, br), 1170 (w), 1145 (w, sh), 1105 (s), 1090 (s, sh), 1055 (m, sh), 1035 (w, sh), 1010 (w), 955 (m, br), 920 (m), 830 (m, br), 795 (w), 735 (m), 720 (m) 690 (w) cm^{-1} .

Polymer 3b

IR (KBr): 3460 (m, br), 3075 (w), 3025 (w), 2950 (s), 2910 (s, sh), 2860 (m), 1760 (s, br), 1735 (s, sh), 1710 (s, br), 1620 (m, sh), 1610 (w), 1590 (m), 1510 (s), 1490 (w), 1460 (w), 1435 (w), 1375 (m, br), 1335 (w, sh), 1295 (m), 1250 (m), 1200 (w, br), 1170 (w), 1140 (w), 1120 (w, sh), 1105 (s), 1060 (m, sh), 1040 (w, sh), 1015 (w), 990 (w), 960 (m), 925 (m), 855 (m, sh), 830 (m), 790 (m), 745 (s), 715 (s) cm^{-1} .

Polymer 3c

IR (KBr): 3460 (m, br), 3060 (w), 3020 (w), 2950 (s), 2910 (s, sh), 2860 (m), 1755 (s, br), 1715 (s, br), 1650 (w, sh), 1620 (m), 1600 (w), 1580 (m), 1505 (s), 1485 (w), 1460 (w), 1435 (w), 1375 (m, br),

1330 (w), 1295 (m), 1250 (m), 1195 (w, br), 1170 (w), 1140 (w), 1125 (w, sh), 1105 (s), 1055 (m, sh), 1035 (w, sh), 990 (w), 960 (m), 925 (m), 855 (m, sh), 845 (m), 790 (m), 745 (s), 715 (s) cm^{-1} .

Polymer 3d

IR (KBr): 3470 (w), 3070 (w), 3030 (w), 2950 (s), 2920 (s, sh), 2860 (m), 1775 (s, br), 1710 (s, br), 1625 (m), 1585 (s), 1570 (m, sh), 1540 (w, sh), 1490 (s, sh), 1445 (s, br), 1370 (m, br), 1345 (w, sh), 1290 (w), 1255–1185 (m, br), 1165 (w), 1140 (w), 1100 (m), 1065 (m, sh), 1025 (w, sh), 985 (w, sh), 960 (m), 925 (w), 870 (m, sh), 850 (m, sh), 835 (m), 780 (m, sh), 760 (m, sh), 745 (m), 715 (m), 695 (m, sh), 650 (w, sh), 575 (w) cm^{-1} .

Polymer 3e

IR (KBr): 3470 (w), 3075 (w), 3040 (w), 2930 (m), 2920 (m, sh), 2860 (w), 1760 (s, sh), 1755 (w, sh),

Table II Some Physical Properties of Poly(ester-imide) 3a–3h Under μw Irradiation

Diols	Polymer Code	Polymers ^a		
		Yield (%)	η_{inh}^b	$[\alpha]^b$
2a	3a	96.2	1.11	–27.8
2b	3b	91.0	0.58	–17.0
2c	3c	95.9	0.72	–21.2
2d	3d	97.4	0.42	–18.4
2e	3e	98.0	0.53	–13.4
2f	3f	92.3	0.4	–24.9
2g	3g	94.1	0.47	–20.6
2h	3h	96.5	0.52	— ^c

^aAll reactions were performed in *o*-cresol under 10-min irradiation time.^bMeasured at a concentration of 0.5 g/dL in DMF at 25°C.^cUndefined.

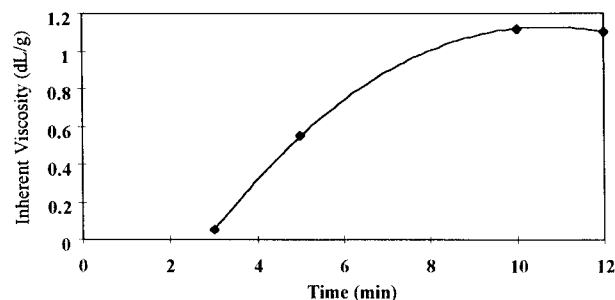


Figure 1 Time dependence of inherent viscosity of the poly(ester-imide) **3a**.

1715 (s, br), 1700 (s, sh), 1670 (w, sh), 1625 (w), 1585 (w), 1488 (m), 1460 (w), 1435 (w), 1378 (m), 1335 (w), 1295 (m), 1255 (m), 1220 (w, sh), 1210 (w, br), 1190 (w, sh), 1170 (w), 1140 (w), 1105 (s), 1060 (w), 985 (w), 960 (m), 925 (w), 855 (m), 845 (m), 790 (w), 745 (m), 715 (m) cm^{-1} .

Polymer 3f

IR (KBr): 3470 (m, br), 3080 (w), 3030 (w), 2950 (s), 2920 (s, sh), 2860 (m), 1760 (s, br), 1755 (w), 1715 (s, br), 1685 (m, sh), 1670 (w, sh), 1620 (w), 1600 (w), 1580 (w), 1505 (w, sh), 1485 (m), 1435 (w), 1375 (m), 1330 (w, sh), 1295 (w), 1255 (m), 1200 (w, br), 1170 (w), 1140 (w), 1105 (s), 1060 (m), 1040 (w, sh), 990 (w, sh), 960 (m), 925 (w), 855 (m, br), 785 (m), 745 (s), 720 (s) cm^{-1} .

Polymer 3g

IR (KBr): 3460 (w), 3080 (w), 3030 (w), 2950 (s), 2920 (s, sh), 2860 (m), 1760 (s, br), 1715 (s, br), 1640 (w), 1620 (m), 1585 (w), 1490 (w), 1460 (w), 1435 (w), 1375 (m, br), 1330 (w), 1295 (m), 1250 (m, br), 1200 (w, br), 1170 (w), 1140 (w), 1125 (w, sh), 1105 (s), 1060 (m), 1040 (m, sh), 1025 (w, sh),

980 (m, sh), 960 (m), 925 (m), 855 (m), 845 (m, sh), 790 (m), 745 (s), 715 (s) cm^{-1} .

Polymer 3h

IR (KBr): 3460 (w), 3400 (w), 3070 (w), 3020 (w), 2950 (s), 2920 (s, sh), 2860 (m), 1755 (s, br), 1720 (s, br), 1635 (m, sh), 1610 (m), 1585 (m, sh), 1490 (m), 1460 (w), 1435 (w), 1370 (m, br), 1290 (w), 1250 (w, br), 1210 (w, br), 1190 (w), 1170 (w), 1140 (w), 1105 (s), 1060 (m), 1040 (w, sh), 980 (m, sh), 960 (m), 925 (m), 855 (m, br), 845 (m), 790 (w), 745 (s), 735 (s), 690 (w, sh), 655 (w), cm^{-1} .

RESULTS AND DISCUSSION

Polymer Synthesis

At first, a series of new poly(ester-imide)s **3a–3c** were prepared from diacid chloride **1** and various aromatic diols **2a–2c** by solution polycondensation in 1,2-dichlorobenzene under refluxing for 24 h. The yield and inherent viscosity of the resulting polymers **3a–3c** are shown in Table III.

The long reaction time, high temperature, low yield, and low inherent viscosity are the arguments that caused us to choose a simple and fast method for polymerization reaction. Microwave radiation energy is very useful for high efficiency utilization of heat energy. So the application of this technique produces polymers with quantitative yield and high inherent viscosity in short time. The heating effect utilized in microwave-assisted organic transformations is due to dielectric constant ϵ and dielectric loss tangent δ of materials. It is particularly convenient that qualitatively, the larger the dielectric constant, the greater the coupling with microwave irradiation. Thus the use of a small amount of a polar solvent, which is heated when irradiated in a microwave

Table III Some Physical Properties of Poly(ester-imide) **3a–3c** Prepared Under Solution Condition at Refluxing Temperature for 24 h in 1,2-Dichlorobenzene

Diols	Polymer	
	Yield (%)	η_{inh} (dL/g) ^a
2a	86.8	0.18
2b	77.7	0.12
2c	78.1	0.09

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

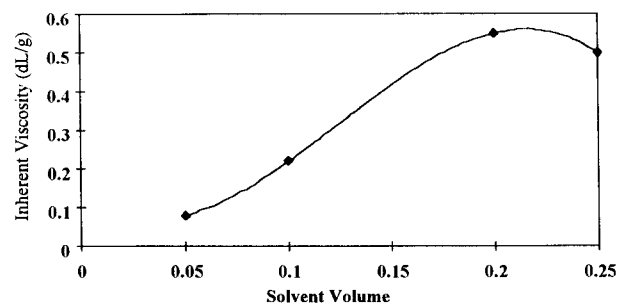
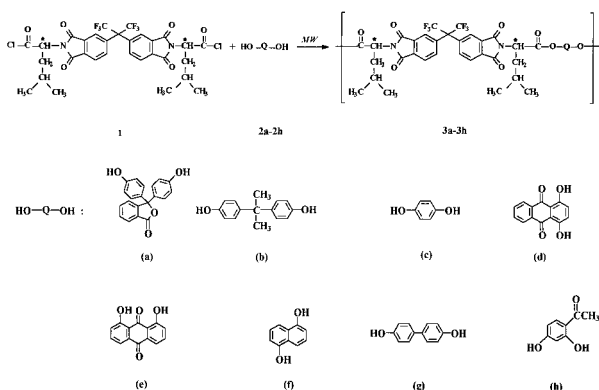


Figure 2 Concentration dependence of inherent viscosity of the poly(ester-imide) **3a**.



Scheme 1

oven, acts: 1. as a primary absorber, and 2. as a solvent for both the starting and resulting materials. Therefore, the solvent allows induction of effective homogeneous heating to convert the monomers to polymers.

Here we have used 1,2-dichlorobenzene, *m*-cresol, and *o*-cresol as solvents. Table I shows that by increasing the dielectric constant and boiling point of the solvent, the inherent viscosities increase. According to these observations, we found that solvent polarity is another parameter that has a significant effect on the inherent viscosity. *o*-Cresol and *m*-cresol have nearly the same ϵ and boiling point, but the first one is more polar. Therefore, the polymers synthesized in *o*-cresol have higher inherent viscosities in com-

parison with the polymers produced in *m*-cresol as a solvent. So we chose *o*-cresol as an appropriate solvent.

To optimize the influence of the amount of *o*-cresol, i.e., monomer concentration, on the inherent viscosity, the different values of solvent were added to the sample cell. For this purpose, 0.1 g of diacid chloride **1** with 1 equimolar diol **2a** were irradiated at a constant time (5 min). Figure 2 exhibits that, by increasing the solvent volume from 0.05 to 0.2 mL at a constant irradiation time, the inherent viscosities increased from approximately 0.08 to 0.55 dL/g, and after that by increasing the solvent volume, the inherent viscosities decreased. Thus, after optimizing the monomer concentration, the favored reaction time was obtained by changing microwave irradiation time in the presence of 0.2 mL *o*-cresol and measuring the viscosity values.

Figure 1 shows that by increasing the irradiation time up to 10 min, the inherent viscosity has increased sharply and above this time its value decreased smoothly.

According to these results the optimum condition was selected for the synthesis of the other poly(ester-imide)s. Thus 0.1 g of diacid chloride **1** with 1 equimolar of eight aromatic diols in 0.2 mL *o*-cresol ($[C] = 0.7$ mol/L) were irradiated 10 min and the resulting mixture was precipitated in methanol to obtain polymer with quantitative yield, as shown in Scheme I.

Table IV Elemental Analysis of Poly(ester-imide)s 3a-3h

Polymer	Formula		Elemental Analysis (%)		
			C	H	N
3a	$(C_{51}H_{38}F_6N_2O_{10})_n$	Calcd	64.29	4.02	2.94
		Found	64.60	4.10	2.70
3b	$(C_{46}H_{40}F_6N_2O_8)_n$	Calcd	64.03	4.67	3.25
		Found	64.60	4.90	3.10
3c	$(C_{37}H_{30}F_6N_2O_8)_n$	Calcd	59.68	4.06	3.76
		Found	62.20	4.60	3.60
3d	$(C_{45}H_{32}F_6N_2O_{10})_n$	Calcd	61.79	3.69	3.20
		Found	64.40	4.30	2.60
3e	$(C_{45}H_{32}F_6N_2O_{10})_n$	Calcd	61.79	3.69	3.20
		Found	63.20	4.30	2.80
3f	$(C_{41}H_{32}F_6N_2O_8)_n$	Calcd	61.97	4.06	3.53
		Found	63.00	4.40	3.30
3g	$(C_{36}H_{31}F_6N_5O_6)_n$	Calcd	62.93	4.18	3.41
		Found	63.20	4.60	3.30
3h	$(C_{44}H_{36}F_6N_4O_7)_n$	Calcd	59.54	4.10	3.56
		Found	62.00	4.30	3.50

Table V Solubility of Poly(ester-imide)s 3a–3h

Solvents	3a	3b	3c	3d	3e	3f	3g	3h
DMAc	+	+	+	+	+	+	+	+
DMF	+	+	+	+	+	+	+	+
THF	+	+	+	+	+	+	+	+
DMSO	±	±	±	±	±	±	±	±
MeOH	–	–	–	–	–	–	–	–
EtOH	–	–	–	–	–	–	–	–
CHCl ₃	–	–	–	–	–	–	–	–
CH ₂ Cl ₂	–	–	–	–	–	–	–	–
H ₂ O	–	–	–	–	–	–	–	–

+ , Soluble at room temperature; – , insoluble; ± , partially soluble.

Polymer Characterization

Table II summarizes some physical properties of these poly(ester-imide)s. Comparing inherent viscosities of microwave irradiation technique with the solution method shows that the internal heat generation of both the solvent and the monomers under microwave irradiation is much more effective than the conventional external heating. Therefore, the microwave irradiation is a convenient method to produce poly(ester-imide)s with high viscosity values in a shorter polymerization time during condensation polymerization (Tables II and III). The structures of these polymers were confirmed as poly(ester-imide)s by means of elemental analysis and IR spectroscopy. Elemental analysis values of the resulting polymers are listed in Table IV.

IR spectra of all polymers are approximately similar, because they possess the same functional

Table VI Thermal Properties of Some Aromatic Poly(ester-imide)s

Polymer	DSC [T_g (°C)]	IDT (°C) ^a	TGA		Char Yield (%) ^d
			T_5^b	T_{10}^c	
3e	81	288	344	370	32.3
3f	106	311	348	369	19.9
3h	93	202	285	344	4.2

^a IDT (initial decomposition temperature) is the temperature at which initial loss of mass was observed.

^b Temperature at which 5% weight loss was recorded by TGA at a heating rate of 20°C/min in N₂.

^c Temperature at which 10% weight loss was recorded by TGA at a heating rate of 20°C/min in N₂.

^d Weight percent of the material left undecomposed after TGA at maximum temperature 600°C in N₂.

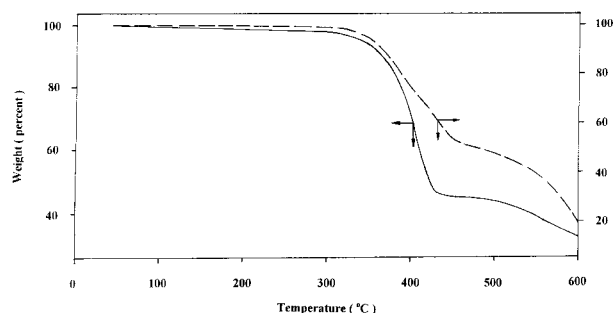


Figure 3 TGA thermograms of poly(ester-imide) **3e** (—) and **3f** (---) with a heating rate of 20°C·min⁻¹ in nitrogen atmosphere.

groups and similar structures. For example the bands around 2950 and 2860 cm⁻¹ result from the stretching of the —CH₂— group. The intensity of the C=O group stretching for the ester groups centered at 1715–1720 cm⁻¹ is strong and broad whereas that for the imide rings at 1755–1760 cm⁻¹ is strong and broad also, but lower than the ester C=O groups. The bands around 1200 and 1060 cm⁻¹ result from aromatic O—C=O stretching.

The solubility of poly(ester-imide)s is listed in Table V. Most of the polymers are soluble in organic solvents such as DMF, *N,N*-dimethylacetamide (DMAc), and tetrahydrofuran (THF) at room temperature, and are insoluble in solvents such as chloroform, methylene chloride, methanol, ethanol, and water.

Thermal Properties

The thermal behavior of poly(ester-imide)s **3e**, **3f**, and **3h** were evaluated by means of DSC and TGA, respectively. Figure 3 shows TGA thermograms of poly(ester-imide)s **3e** and **3f** with a heating rate of 20°C/min in nitrogen. The glass transition temperature, initial decomposition temperature, 5 and 10% weight loss (T_5 , T_{10}), and char yield at 600–C for poly(ester-imide)s **3e**, **3f**, and **3h** are summarized in Table VI.

CONCLUSIONS

A series of novel optically active and high molecular weight aromatic poly(ester-imide)s have been successfully prepared by using microwave irradiation technique from the polycondensation reactions of diacid chloride **1** with several diols in the presence of a small amount of a

polar organic medium such as *o*-cresol that acts as a primary microwave absorber. The resulting polymers have high inherent viscosities in a range of 0.50–1.12 dL/g and also show optical rotation. Therefore, they are optically active and have potential to be used in a proper column chromatography technique for the separation of enantiomeric mixtures. Furthermore, the microwave-assisted step-growth polymerization proceeded rapidly, compared with the conventional solution polycondensation, and was completed within a short period of time.

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