New Rigid-Rod Poly(benzoxazole-imide)s Containing Chloro-Substituted *p*-Phenylene Units in the Main Chain

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ABSTRACT: A series of new poly(*o*-hydroxy amide-imide)s with high molecular weights were synthesized by low-temperature solution polycondensation from a preformed imide ring and chloro- or dichloro-substituted *p*-phenylene-containing diacid chlorides of 2,5-bis(trimellitimido)chlorobenzene or 1,4-bis(trimellitimido)-2,5-dichlorobenzene and three bis(*o*-amino phenol)s. All the poly(*o*-hydroxy amide-imide)s were readily soluble in a variety of organic solvents such as *N*-methyl-2-pyrrolidone and *N*,*N*-dimethylacetamide. Transparent and flexible films of these polymers were cast from their solutions. The cast films had tensile strengths ranging from 88 to 102 MPa and elongations at break of 8–12%. Subsequent thermal cyclodehydration of the poly(*o*-hydroxy amide-imide)s afforded novel poly(benzoxazole-imide)s. The poly(benzoxazole-imide)s exhibited glass-transition temperatures in the range of 310–338 °C and were stable up to 500 °C in nitrogen, with 10% weight-loss temperatures recorded between 550 and 570 °C in nitrogen. © 1999 John Wiley & Sons, Inc. J Polym Sci A: Polym Chem 37: 4151–4158, 1999

Keywords: poly(*o*-hydroxy amide-imide)s; poly(benzoxazole-imide)s; 2,5-bis(trimellitimido)chlorobenzene; 1,4-bis(trimellitimido)-2,5-dichlorobenzene

INTRODUCTION

Among the various heterocyclic polymers that have been investigated for high-performance applications, aromatic polyimides are used widely in the semiconductor and electronic-packaging industry because of their outstanding thermal stability, good insulation properties with low dielectric constant, good adhesion to common substrates, and superior chemical stability.[1,2] Aromatic polybenzoxazoles are another class of heterocyclic polymers that exhibit excellent thermooxidative stability, high-tensile modulus and strength, and superior chemical resistance.[3,4] A few rigid-rod polybenzoxazoles are reported to have potential for fabrication into high-modulus, high-strength fiber.[5,6] However, just as with aromatic polyimides, they are generally difficult to process because of their unmeltable behavior and poor solubility in conventional organic solvents. Consequently, potential applications have been limited, and many attempts have been tried to modify and improve their processibility.[7–13] One successful approach is to introduce flexible linkages into the polymer backbone to increase overall flexibility. Fluorinated monomers appear to be the key to soluble, high-performance materials in most of these synthetic efforts. For example, incorporation of the 2,2-hexafluoroisopropylidene (6F) group into the polybenzoxazole backbone enhanced solubility while retaining favorable thermooxidative stability and high glass-transition temperatures.[7,10–13] Similarly, soluble poly(ether benzoxazole)s have been generated and display properties intermediate between the two homopolymers.[14,15] Desired properties resulting from these materials include thermoplasticity, ex-

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cellent tensile properties, and enhanced toughness.

As part of an effort to develop high-performance and high-temperature-resistant polymers, we became interested in the potential usefulness of substituent-containing *p*-phenylene structure as a simultaneously bulky and symmetrical unit in the main chain. Recently, we reported that aromatic poly(amide-imide)s deriving both properties from a preformed imide ring containing the aromatic diimide-diacids 2,5-bis(trimellitimido)-chlorobenzene[16] and 1,4-bis(trimellitimido)-2,5-dichlorobenzene[17] had improved solubility with the retention of high thermal stability. The resulting poly(amide-imide) films were obtained with good mechanical properties. This article expands this approach to include mono- and dichloro-substitution on the central diamine components.

EXPERIMENTAL

Materials

Commercially obtained 2-chloro-*p*-phenylenediamine dihydrochloride (TCI Chemical Company), 2,5-dichloro-*p*-phenylenediamine (TCI Chemical Company), and trimellitic anhydride (TCI Chemical Company) were used without further purification. The aromatic bis(*o*-aminophenol) monomers, including 3,3′-dihydroxybenzidine, 3,3′-diamino-4,4′-dihydroxy-biphenyl, and 2,2-bis(3-amino-4-hydroxyphenyl)hexafluoropropane, were of high purity and used as received from TCI Chemical Company. *N*-Methyl-2-pyrrolidone (NMP) was purified by distillation under reduced pressure over calcium hydride and stored over 4-Å molecular sieves. 2,5-Bis(trimellitimido)chlorobenzene and 1,4-bis(trimellitimido)-2,5-dichlorobenzene were prepared by a two-stage procedure that included the ring-opening addition of 2-chloro-*p*-phenylenediamine or 2,5-dichloro-*p*-phenylenediamine with two equivalents of trimellitic anhydride, followed by cyclodehydration to the imidodicarboxylic acid by toluene–water azotropic distillation.[16,17] The reaction of the two diimide-diacids with excess thionyl chloride in the presence of a few drops of *N,N*-dimethylformamide (DMF) as catalyst afforded the corresponding diimide-diacid chlorides 1 and 2, respectively. Diacid chloride 1 was purified by recrystallization from a mixture of hexane and toluene to afford a white powder: mp = 239–240 °C, IR (KBr) 1,770 cm^{-1} (acid chloride C═O stretching), 1,790 cm^{-1} (imide, symmetric C═O stretching), and 1,735 cm^{-1} (imide, asymmetric C═O stretching).

ELEM. ANAL. calcd. for C24H9N2O6Cl3: C, 54.63%; H, 1.72; N, 5.31. Found: C, 54.51%; H, 1.69; N, 5.28.

Diacid chloride 2 was purified by recrystallization from toluene to give white needles: mp = 323–324 °C;, IR (KBr) 1,770 cm^{-1} (acid chloride C═O stretching), 1,790 cm^{-1} (imide, symmetric C═O stretching), and 1,745 cm^{-1} (imide, asymmetric C═O stretching).

ELEM. ANAL. calcd for C24H8N2O6Cl4: C, 51.28%; H, 1.43%; N, 4.98%. Found: C, 51.13%; H, 1.39%; N, 4.94%.

Polymer Synthesis

The low-temperature solution polycondensation technique was employed in this investigation. The following procedure for preparing poly(*o*-hydroxy amide-imide)s was typical. First, 2 mmol of bis(*o*-aminophenol) were dissolved in 10 mL of NMP and maintained at −10 to 0 °C in an ice–acetone bath. To this solution, 0.6 mL of propylene oxide was added, and then 2 mmol of diimide-diacid chloride were added. The reaction mixture was maintained at −10 to 0 °C for about 1 h and then at room temperature overnight (for ca. 10 h). As the polycondensation proceeded, the reaction mixture became increasingly viscous. The resulting polymer solution was poured slowly into 300 mL of methanol to give a fiber-like precipitate that was washed thoroughly with hot methanol, collected by filtration, and dried. Yields were usually quantitative. Inherent viscosities of the poly(*o*-hydroxy amide-imide)s were measured in NMP at a concentration of 0.5 g/dL at 30 °C.

Preparation of Poly(*o*-hydroxy amide-imide) Films and Thermal Conversion to Poly(benzoxazole-imide)s

A polymer solution for preparing the film was made by dissolving about 1.0 g of the poly(*o*-hydroxy amide-imide) sample in 10 mL of NMP. The solution was poured into a $\phi = 9$ cm glass culture dish that was placed in a 90 °C oven for 12 h to remove the solvent. The obtained semidried polymer film was stripped from the glass substrate and further dried *in vacuo* at 200 °C for 15 h.

Poly(benzoxazole-imide)s were obtained by thermal cyclodehydration of poly(*o*-hydroxy amide-imide)s at 380 °C *in vacuo* for 8 h on preformed

films. The inherent viscosities of the poly(benzoxazole-imide)s thus obtained were measured in concentrated sulfuric acid at a concentration of 0.5 g/dL at 30 °C.

Measurements

IR spectra were recorded on a Jasco IR-7000 infrared spectrometer. Elemental analyses were run in a Perkin-Elmer Model 2400 C, H, N analyzer at National Taiwan University (Taipei). Inherent viscosities were measured at 0.5 g/dL with a Cannon-Fenske viscometer thermostated at 30 °C. A Sinku Riko DSC-7000 differential scanning calorimeter equipped with a Sinku Riko TA-7000 thermal analyzer was used to determine thermal transitions with a heating rate of 20 °C/min. Glass-transition temperatures (T_g) were read at the middle of the changes in heat capacities. For thermogravimetric analysis (TGA), a DuPont 951 thermogravimetric analyzer was used. Experiments were carried out on 9- to 11-mg samples heated in flowing nitrogen or air (50 cm^3/min) at a heating rate of 10 °C/min. Wide-angle X-ray diffraction measurements were performed at room temperature (ca. 25 °C) on a Scintag-Xgen-4000 X-ray diffractometer using Ni-filtered CuK_α radiation (40 kV, 15 mA). The scanning rate was 2 °/min over a range of $2\theta = 5$ to 40 °. Tensile properties of solution-cast films were determined using an Instron universal tester, Model HT-9102 (Hung Ta Instrument Co., Taiwan), with a load cell of 10 kg. A gauge of 2 cm and a crosshead speed of 5 cm/min were used for this study. Measurements were performed at room temperature with film specimens (6 cm long, 0.5 cm wide, ca. 0.1 mm thick). Averages of at least five individual determinations are reported here.

RESULTS AND DISCUSSION

Polymer Synthesis

Polybenzoxazoles may be synthesized easily in a one-step procedure using poly(phosphoric acid),[5] phosphorous pentoxide/methanesulfonic acid,[18] or trimethylsilyl phosphate[19] as the condensing medium. Isolation and thermal cyclodehydration of soluble poly(*o*-hydroxy amide)s, which is derived from polycondensation of diacid derivatives and bis(*o*-aminophenol)s in polar solvent, is an alternative method for the production of polybenzoxazole films and fibers.[20] In this work, six

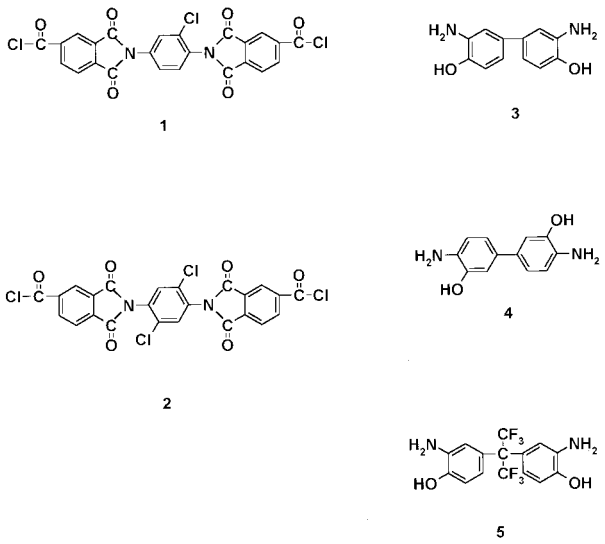

Figure 1. Structures and codes of monomers.

poly(*o*-hydroxy amide-imide)s (6–11) were prepared readily by the low-temperature solution polycondensation of diimide-diacid chlorides 1 or 2 with three structurally different bis(*o*-aminophenol)s (3–5) in NMP solution in the presence of propylene oxide as the acid acceptor at −10 to 0 °C. These conditions are preferred over the use of triethylamine as scavenger for the preparation of higher molecular-weight aramids.[21] Structures and codes of the monomers and poly(*o*-hydroxy amide-imide)s are given in Figures 1 and 2, respectively. The results of the polymerizations are summarized in Table I. The inherent viscosities of the resulting poly(*o*-hydroxy amide-imide)s were in the range of 0.80 to 2.56 dL/g. The 6F-containing bis(*o*-aminophenol) (5) derived poly(*o*-hydroxy amide-imide)s 10 and 11 were attained with high molecular weights, as evidenced by inherent viscosity values of around 0.8 dL/g. Apparently, silylation of bis(*o*-aminophenol) (5), as reported by Maruyama et al.,[7] is not a necessary step to attain high-molar-mass poly(*o*-hydroxy amide-imide)s in the low-temperature solution polycondensation reaction. All these poly(*o*-hydroxy amide-imide)s were readily soluble in amide-type solvents such as NMP and *N*,*N*-dimetylacetamide (DMAc) and afforded freestanding films by means of solution casting. The films obtained were all flexible and tough. Polymers 6 to 9 were brownish, whereas Polymers 10 and 11 were light yellow in color.

Formation of poly(*o*-hydroxy amide-imide)s was confirmed by means of IR spectroscopy and elemental analysis. The polymers exhibited broad

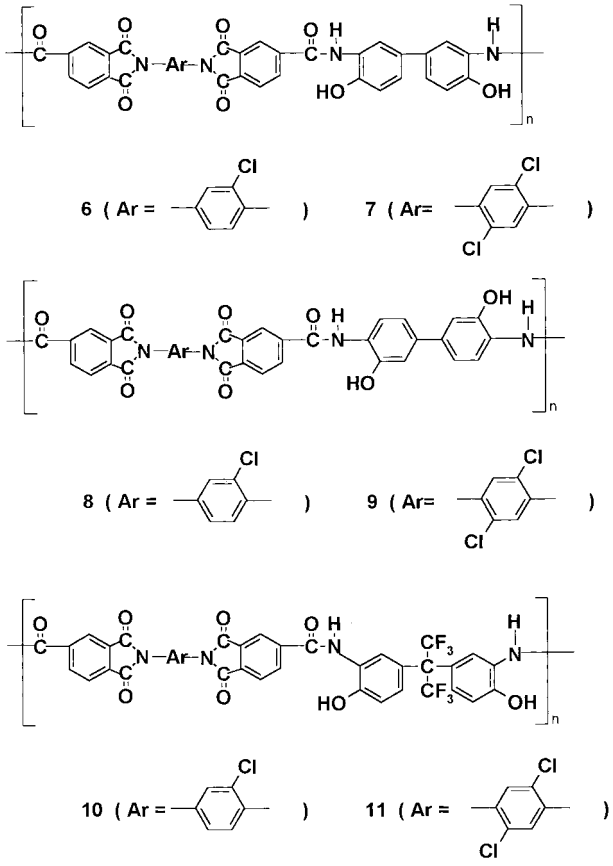

Figure 2. .Structures and codes of poly(*o*-hydroxy amide-imide)s.

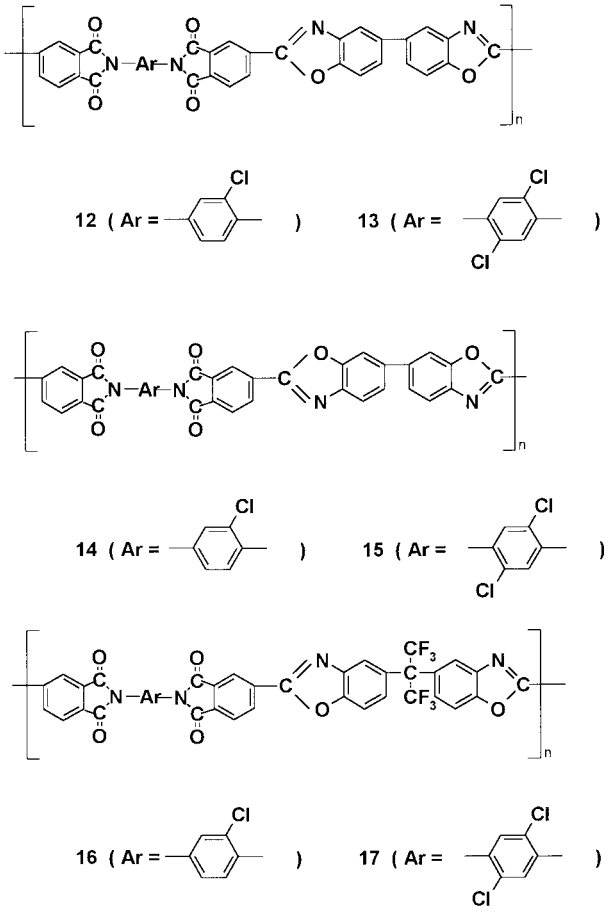

Figure 3. Structures and codes of poly(benzoxazole-imide)s.

absorption bands in the region of 2,500 to 3,500 cm^{-1} (O—H and N—H stretching), 1,666 cm^{-1} (amide C═O stretching), 1,790 cm^{-1} (imide, symmetric C═O stretching), and 1,745 cm^{-1} (imide, asymmetric C═O stretching). Conversions to poly(benzoxazole-imide)s (Fig. 3) were carried out on films at 380 °C *in vacuo* for about 8 h. The con-

Table I. Synthesis of Poly(*o*-hydroxy amide-imide)s and Poly(benzoxazole-imide)s

	Poly(*o*-hydroxy amide-imide)		Poly(benzoxazole-imide)				
					Elemental Analysis (%)[c]		
Monomer	Code	η_{inh} (dL/g)[a]	Code	η_{inh} (dL/g)[b]	C	H	N
1 + 3	6	2.15	12	1.87	67.38 (68.09)	2.52 (2.38)	8.60 (8.82)
2 + 3	7	2.56	13	1.63	64.01 (64.59)	2.25 (2.11)	8.15 (8.37)
1 + 4	8	1.77	14	1.62	67.44 (68.09)	2.54 (2.38)	8.56 (8.82)
2 + 4	9	1.83	15	1.50	63.88 (64.59)	2.32 (2.11)	8.12 (8.37)
1 + 5	10	0.80	16	0.90[a]	59.11 (59.67)	2.05 (1.93)	6.91 (7.14)
2 + 5	11	0.85	17	0.87[a]	56.64 (57.16)	1.83 (1.72)	6.62 (6.84)

[a] Measured at a concentration of 0.5 g/dL in NMP at 30°C.
[b] Measured at a concentration of 0.5 g/dL in concentrated sulfuric acid at 30°C, unless otherwise indicated.
[c] Data in parentheses are calculated values.

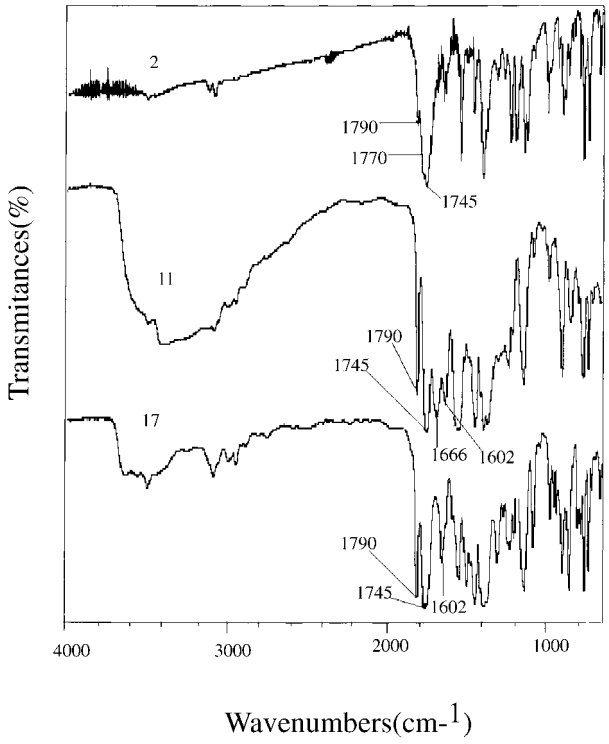

Figure 4. IR spectra of poly(*o*-hydroxy amide-imide) 11 and poly(benzoxazole-imide) 17.

version process was followed by changes in the IR spectra of the films. Figure 4 shows typical IR spectra of a representative pair of poly(*o*-hydroxy amide-imide) 11 and its corresponding poly(benzoxazole-imide) 17. Disappearance of the absorption bands around 2,500 to 3,500 and 1,660 cm^{-1} indicated completion of the cyclization process, whereas an absorption at 1,602 cm^{-1} was observed to be characteristic of a benzoxazole ring, which almost overlapped one of the absorption bands of the aromatic rings. Elemental analysis values of the transformed polymers agreed well with values calculated for the polymers with benzoxazole structures. There was sufficient difference in oxygen content between the poly(*o*-hydroxy amide-imide) and the resulting poly(benzoxazole-imide) to indicate essentially complete conversion. This reaction also was monitored by TGA and differential scanning calorimetry (DSC), which is discussed later. The poly(benzoxazole-imide)s thus obtained, except for 16 and 17, were insoluble in most organic solvents but soluble in concentrated sulfuric acid. The poly(benzoxazole-imide)s derived from diacid chlorides 1 and 2 had inherent viscosities of 0.87 to 1.87 dL/g in concentrated sulfuric acid, indicating that no thermal degradation or decomposition leading to molecular chain scission occurred during the conversion process or during the viscosity determination. This also indicates that the chloro-substituted *p*-phenylene linkage containing poly(benzoxazole-imide)s has good acid-resistance.

Polymer Characterization

All of the poly(*o*-hydroxy amide-imide)s were easily soluble in polar aprotic solvents such as NMP, DMAc, and DMF at room temperature (Table II). Nevertheless, they still exhibited excellent resistance to less polar solvents such as *m*-cresol, tetrahydrofuran (THF), acetone, and ethanol. The poly(*o*-hydroxy amide-imide)s 10 and 11 derived

Table II. Solubility Behavior of Poly(*o*-hydroxy amide-imide)s and Poly(benzoxazole-imide)s[a]

	Poly(*o*-hydroxy amide-imide)						Poly(benzoxazole-imide)					
Solvent[b]	6	7	8	9	10	11	12	13	14	15	16	17
Concd H_2SO_4	+	+	+	+	+	+	+	+	+	+	+	+
NMP	+	+	+	+	+	+	−	−	−	−	+	+
DMAc	+	+	+	+	+	+	−	−	−	−	+	+
DMF	+	+	+	+	+	+	−	−	−	−	−	−
DMSO	+h	+	+h	+h	+	+	−	−	−	−	−	−
m-Cresol	−	−	−	−	−	−	−	−	−	−	−	−
THF	−	−	−	−	+	+	−	−	−	−	−	−
Acetone	−	−	−	−	+	+	−	−	−	−	−	−
Ethanol	−	−	−	−	−	−	−	−	−	−	−	−

[a] Solubility: +, soluble at room temperature; +h, soluble on heating; −, insoluble even on heating.
[b] NMP: *N*-methyl-2-pyrrolidone; DMAc: *N,N*-dimethylacetamide; DMF: *N,N*-dimethylformamide; DMSO: dimethyl sulfoxide; THF: tetrahydrofuran.

from 6F-bis(*o*-aminophenol) 5 showed enhanced solubility and were even soluble in THF and acetone. This may be due to the bulky and flexible hexafluoroisopropylidene groups reducing chain packing and allowing solvent molecules to diffuse into the polymer chains easily.

The poly(benzoxazole-imide)s, however, dissolved only in cold sulfuric acid. Despite the fact that all the poly(benzoxazole-imide)s were amorphous, they were quite insoluble in organic solvents, with the exceptions of Polymers 16 and 17, which contained the bulky 6F group in the bis(*o*-aminophenol) moieties along the macromolecular backbone. Comparison of the solubility of 16 and 17 with those of the poly(benzoxazole-imide)s[12] that contained bulky 6F groups in both the imide and benzoxazole moieties indicates that introduction of chloro-substituted *p*-phenylene units into the polymer backbone greatly improves solubility of the poly(benzoxazole-imide)s in organic solvents.

Crystallinity of the prepared poly(*o*-hydroxy amide-imide) and poly(benzoxazole-imide) films was measured by a wide-angle X-ray diffraction study. Figure 5 reveals that all polymers were amorphous, probably because of the chloro-substituents on the *p*-phenylene linkages in the polymer backbones. However, all of the poly(benzoxazole-imide)s showed higher chain packing than the corresponding poly(*o*-hydroxy amide-imide)s because of the rigid and planar benzoxazole structure formation. Thus, the phenomena were reflected in their poor solubility, which is in agreement with the general rule that solubility decreases with increasing chain-packing density. All the poly(*o*-hydroxy amide-imide)s were solution-cast into tough, flexible films. The tensile strengths and the elongations at break (Table III) were in the range of 88 to 102 MPa and 8 to 10%, respectively. All the thermally converted poly(benzoxazole-imide) films were also flexible and tough, indicating that no thermal degradation leading to molecular chain scission occurred during the conversion process. However, most of the films shrank during the cyclization process, and their mechanical properties were not evaluated. Good-quality, smooth poly(benzoxazole-imide) films may be obtained if a carefully designed heating program is employed.

The thermal behavior of the poly(*o*-hydroxy amide-imide)s and poly(benzoxazole-imide)s was investigated by DSC and TGA. The poly(*o*-hydroxy amide-imide)s displayed glass transitions between 210 to 225 °C in the first DSC scan and

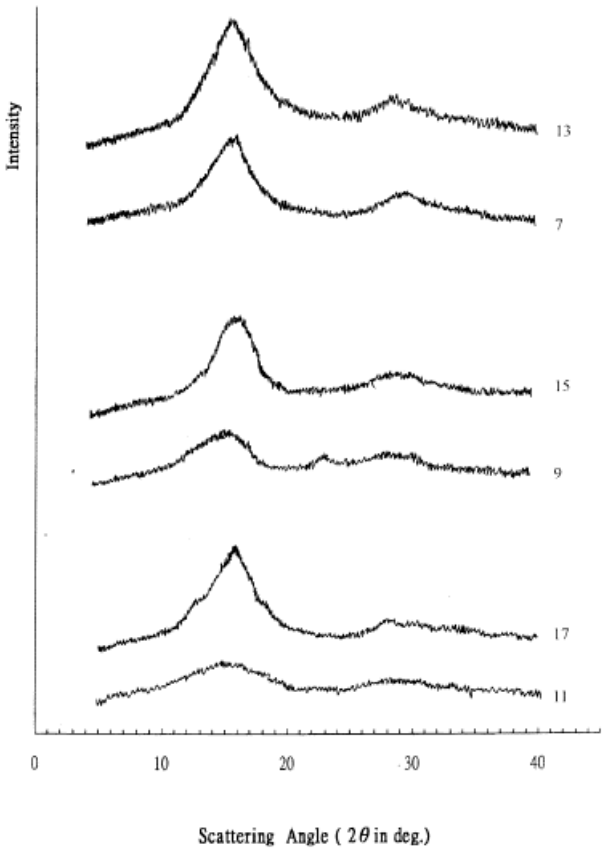

Figure 5. Wide-angle X-ray diffractograms of some poly(*o*-hydroxy amide-imide)s and poly(benzoxazole-imide)s.

exhibited strong endothermic peaks between 255 to 380 °C with peak temperatures around 322 to 340 °C. The latter two were attributed to loss of water during the conversion of the poly(*o*-hydroxy amide-imide) to poly(benzoxazole-imide). Figure 6 shows a typical pair of TGA curves for poly(*o*-hydroxy amide-imide) 7 and poly(benzoxazole-imide) 13. The TGA trace of poly(*o*-hydroxy amide-imide) 7 revealed that weight loss started at around 250 °C and came to an end at about 380 °C. This weight loss agreed well with the strong endothermic peak between 270 to 380 °C. Poly(benzoxazole-imide) 13 started to lose weight at around 500 °C and left 62% residual char when heated to 800 °C in nitrogen. The baseline-shift center at 225 °C in the first DSC heating trace is ascribed to the glass transition of poly(*o*-hydroxy amide-imide) 7. After rapid quenching from 450 °C, the poly(benzoxazole-imide) 13 formed *in situ* showed a distinct glass transition around 338 °C on the subsequent DSC heating trace. The increased T_g of poly(benzoxazole-imide) 13 when

Table III. Tensile Properties and Thermal Behavior of Poly(*o*-hydroxy amide-imide)s and Poly(benzoxazole-imide)s

Poly(*o*-hydroxy amide-imide)						Poly(benzoxazole-imide)			
	Tensile Properties of Polymer Films		DSC				DSC		TG
Code	Strength-to-Break in MPa	Elongation-to-Break (%)	T_g (°C)[a]	T_O (°C)[c]	T_P (°C)[d]	Code	T_g (°C)[e]	T_d (°C)[f]	Char Yield[g]
6	96	9	223	265	322	12	330	570 (535)	65.1
7	102	10	225	270	325	13	338	560 (530)	62.0
8	94	8	—[b]	268	338	14	331	565 (535)	64.5
9	101	10	—[b]	275	340	15	337	560 (530)	60.3
10	88	9	215	255	332	16	310	555 (485)	62.2
11	90	10	210	260	335	17	316	550 (460)	65.0

[a] Temperature at the middle point of baseline shift on the first DSC heating trace with a heating rate of 20°C/min in nitrogen.
[b] Not clearly observed.
[c] Extrapolated onset temperature of the endothermic peak.
[d] Endothermic peak temperature.
[e] Midpoint temperature of baseline shift on the second DSC heating trace of the sample after rapid cooling from 450°C in nitrogen.
[f] 10 wt % loss temperature at a heating rate of 10°C/min in nitrogen. Values in parentheses were observed in air.
[g] Residual wt % when heated to 800°C in nitrogen.

compared to its poly(*o*-hydroxy amide-imide) precursor 7 may be a reflection of enhanced chain rigidity due to the formation of benzoxazole rings. All the poly(*o*-hydroxy amide-imide)s and poly(benzoxazole-imide)s exhibited a similar DSC and TGA behavior, and their thermal data are summarized in Table 3. All the poly(benzoxazole-imide)s did not lose weight up to 500 °C in nitrogen, and the temperatures at which 10% weight loss was recorded ranged from 550 to 570 °C. All showed char yields greater than 60% when heated to 800 °C in nitrogen. Their glass-transition temperatures (T_g) were between 310 to 338 °C. The higher T_g and processability make these poly(benzoxazole-imide)s attractive as practical materials.

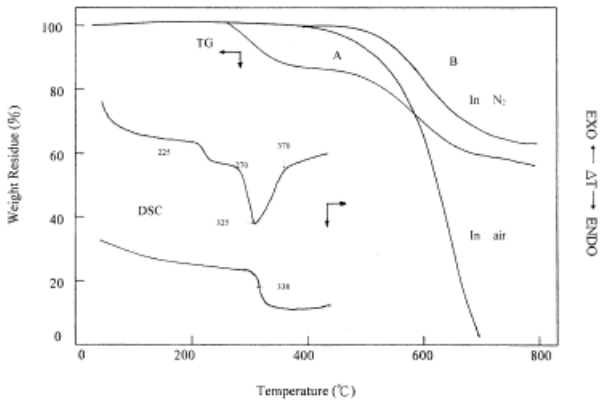

Figure 6. TGA and DSC curves for (A) poly(*o*-hydroxy amide-imide) 7 and (B) poly(benzoxazole-imide) 13.

CONCLUSIONS

Six poly(*o*-hydroxy amide-imide)s with high molar masses were prepared from the diacid chlorides of 2,5-bis(trimellitimide)chlorobenzene (1) and 1,4-bis(trimellitimide)-2,5-dichlorobenzene (2) with bis(*o*-aminophenol)s by the low-temperature solution polycondensation. All the poly(*o*-hydroxy amide-imide)s were easily soluble in a variety of organic solvents and were cast into flexible and tough films from their polymer solutions. They had T_gs between 210 to 225 °C and were transformed to the corresponding poly(benzoxazole-imide)s at elevated temperatures. The poly(benzoxazole-imide)s revealed a decreased solubility but higher T_gs when compared to their corresponding poly(*o*-hydroxy amide-imide) pre-

polymers. All the poly(benzoxazole-imide)s showed good thermal stability. Thus, the novel rigid-rod poly(benzoxazole-imide)s are promising candidates for new high-performance polymeric materials.

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