Synthesis and Properties of New Polyarylates from 1,4-Bis(4-carboxyphenoxy)naphthyl or 2,6-Bis(4-carboxyphenoxy)naphthyl and Various Bisphenols

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ABSTRACT: New 1,4-naphthyl and 2,6-naphthyl-containing polyarylates having inherent viscosities up to 1.28 dL/g were synthesized by the high-temperature solution polycondensation from the acid chloride of 1,4-bis(4-carboxyphenoxy)naphthyl or 2,6-bis(4-carboxyphenoxy)naphthyl and various bisphenols. Most of the resulting polyarylates showed amorphous characteristics and were readily soluble in common organic solvents such as N,N-dimethylacetamide (DMAc), N-methyl-2-pyrrolidone (NMP), o-chlorophenol, and chloroform. Transparent, flexible, and colorless films of these polymers could be cast from the DMAc solutions. Their cast films had tensile strengths ranging from 54.9 to 84.2 MPa, elongations at break from 5.3% to 19.0%, and initial modulus from 2.0 to 2.8 GPa. These polymers had glass transition temperatures in the range of 172–280°C and began to lose weight around 400°C, with 10% weight loss being recorded at about 450°C in air. Dynamic mechanical analysis (DMA) reveals that the polyarylates containing isopropylidene linkages have three transitions on the temperature scale between −100 and 300°C. However, only two transitions were observed in the other polyarylates without isoproylidene linkage. J Polym Sci A: Polym Chem 37: 645–652, 1999

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INTRODUCTION

Interest in molecular engineering of physical properties for high-performance polymers persists because small alteration of molecular structure of a polymer can dramatically influence performance and processing properties. Generally, alteration of high-performance polymers focuses on changes in substitution pattern of aromatic cyclic units comprising the bulk of such polymers. Change in chain stiffness or rigidity via modification of primary and secondary structures is particularly effective.[1] It is well known that wholly aromatic polyesters (polyarylates) possess high thermal stability and excellent mechanical properties.[2] There are numerous examples in the literature of use of biphenyl-4,4′-diyl or 2,6-naphthyl units to promote liquid crystallinity in polyesters.[3–6] However, the high melting temperatures or softening temperatures and limited solubility in organic solvents of most polyarylates make their processing into articles difficult unless flexible spacers are included in the systems, thereby lowering the thermal stability. One of the

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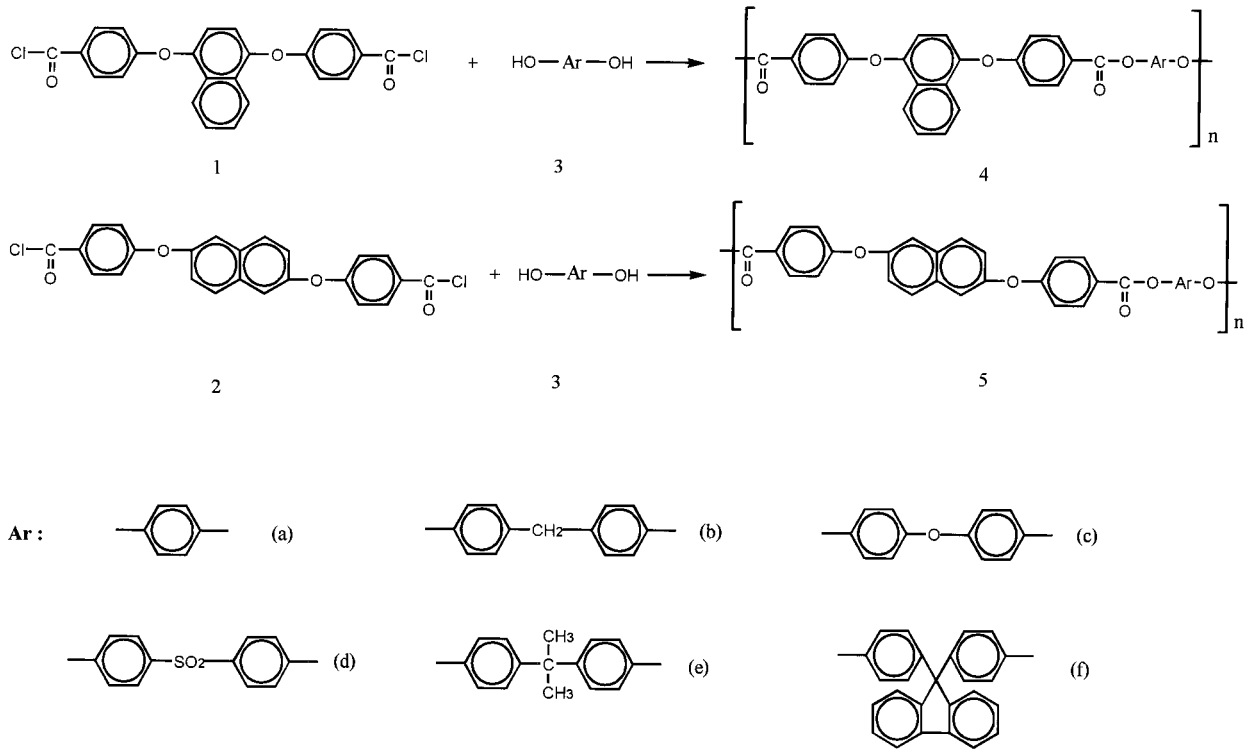

Scheme 1.

successful approaches to increase solubility and processability with simultaneous preservation of thermal stability would be the introduction of bulky, kink, crank, and noncoplanar structure into the polymer backbone. Some organosoluble polyarylates have been demonstrated by using aromatic diacid chlorides having biphenyl-2,2′-diyl or 1,1′-binaphthyl-2,2′-diyl unit.[7–9] We also have already reported that the synthesis of new organosoluble polyarylates having high glass transition temperatures by the incorporation of 1,6-diamantane structure along the polymer main chain backbone.[10]

Recently, in continuation of these studies, we are interested in the potential usefulness of naphthyl-containing structure as simultaneously bulky and symmetrical unit in the polymer main chain. We have already reported that the aramids derived both from 1,4-bis(4-carboxyphenoxy)naphthyl and 2,6-bis(4-carboxyphenoxy)naphthyl[11,12] had improved solubility with retention of high thermal stability. These resulting transparent and flexible aromatic polyamide films were obtained with good mechanical properties. Therefore, the diacid chlorides of 1,4-bis(4-carboxyphenoxy)naphthyl and 2,6-bis(4-carboxyphenoxy)naphthyl would be potential monomers for the preparation of high-performance polyarylates. In this article, we describe a successful synthesis of novel soluble polyarylates from both the diacid chloride (1) or (2) and various bisphenols by the high-temperature solution condensation (Scheme 1). The solubility, tensile properties, crystallinity, and thermal properties of the obtained polymers will also investigated.

EXPERIMENTAL

Materials

Various bisphenols were obtained commercially and purified by recrystallization. They included hydroquinone (3_a), 4,4′-dihydroxydiphenylmethane (3_b), 4,4′-dihydroxydiphenylether (3_c), bis(4-hydroxyphenyl)sulfone (bisphenol S, 3_d), 2,2′-bis(4-hydroxyphenyl)propane (bisphenol A, 3_e), and 9,9-bis(4-hydroxyphenyl)fluorene (fluorene bisphenol, 3_f). o-Dichlorobenzene was purified by distillation under reduced pressure over calcium hydride.

Monomer Synthesis

Diacid Chlorides (1 and 2) of 1,4-Bis(4-carboxyphenoxy)naphthyl (1,4-NCA) and 2,6-Bis(4-carboxyphenoxy)naphthyl (2,6-NCA)

1,4-NCA and 2,6-NCA were prepared by the nucleophilic fluorodisplacement reaction of p-fluorobenzonitrile with 1,4-dihydroxynaphthalene and 2,6-dihydroxynaphthalene, respectively, followed by the alkaline hydrolysis reaction of the dinitrile compounds in potassium hydroxide solution, according to previously reported procedures.[11,12] The reaction of dicarboxylic acids (1,4-NCA) and (2,6-NCA) with thionyl chloride in the presence of a few drops dimethylformamide (DMF) as a catalyst afforded diacid chlorides (1, 1,4-NCL) and (2, 2,6-NCL) respectively. Diacid chloride 1 was purified by recrystallization from a mixture of hexane and toluene to afford white needles (80%, yield); mp 167–168°C; IR (KBr) 1769 cm^{-1} (C═O). EIMS m/z (%) 436 (M^+, 95), 401 (100), 183 (90); ^{13}C-NMR (100 MHz, DMSO-d_6): δ 116.45, 116.73, 121.98, 125.31, 127.60, 127.66, 131.82, 147.46, 161.81, 166.69. ANAL. Calcd for $C_{24}H_{14}O_4Cl_2$: C, 65.90%; H, 3.20%. Found: C, 66.40%; H, 3.35%. Diacid Chloride 2 was purified by recrystallization from toluene to give white needles (75%, yield); mp 185–186°C; IR (KBr) 1768 cm^{-1} (C═O). EIMS m/z (%) 436 (M^+, 70), 401 (100), 183 (80); ^{13}C-NMR (100 MHz, DMSO-d_6): δ 116.04, 117.43, 121.21, 125.45, 129.88, 131.28, 131.75, 152.54, 161.03, 166.72. ANAL. Calcd for $C_{24}H_{14}O_4Cl_2$: C, 65.90%; H, 3.20%. Found: C, 66.44%; H, 3.37%.

Polymer Synthesis

Polyester 4_c from 1 and 3_c

A typical example of polycondensation is given below: A stoichiometric mixture of 1.094 g (2.5 mmol) of 1 and 0.506 g (2.5 mmol) of 3_c in 8 mL of o-dichlorobenzene was heated with stirring at 200°C under nitrogen for 20 h. After the solution was allowed to cool to ambient temperature, then the viscous solution was poured slowly into 300 mL of methanol with stirring. The polymer that precipitated was collected by filtration, washed thoroughly with hot methanol, and dried under reduced pressure at 150°C for 15 h. The polyarylate 4_c was obtained quantitative and had an inherent viscosity of 1.28 dL/g, measured at a concentration of 0.5 g/dL in o-chlorophenol at 30°C. The polyarylate film was obtained by casting from the DMAc solution onto a glass plate and drying at 150°C for 15 h under vacuum. The IR spectrum of 4_c (film) exhibited a characteristic absorption band at 1735 cm^{-1} (C═O).

The other polyarylates were prepared by analogous procedure.

Measurements

A Bio-Rad FTS-40 FTIR spectrophotometer was used to record spectra of the KBr pellets. In a typical experiment, an average of 20 scans per sample was made. MS spectra were obtained using a JEOL JMS-D300 mass spectrometer. ^{13}C-NMR spectra were recorded on a Bruker AM-300 WB Fourier transform nuclear magnetic resonance spectrometer. Elemental analyses were run in a Perkin-Elmer Model 2400 C, H, N analyzer. The inherent viscosities were measured with an Ubbelohde viscometer thermostated at 30°C at 0.5 g/dL concentration in o-chlorophenol. Qualitative solubility was determined using 0.01 g of polymer in 2 mL of solvent. Differential scanning calorimetry (DSC) and thermogravimetry (TG) were performed with Sinku Riko DSC-7000 and Rigaku thermoflex TGA-8110 coupled to a Rigaku TAS-100 thermal analysis station, respectively. Glass transition temperature, T_g, was read at the middle of the change in the heat capacity, and was taken from the second heating scan after rapid cooling and the melting temperature, T_m, was taken from the endothermic peaks. Thermomechanical analysis (TMA) was carried out with a DuPont 9900 thermalanalyzer system. TMA was recorded under a fixed load (2 g) at a heating rate of 5°C/min. The softening temperature temperature, T_s, was estimated from the inflection point on the displacement. Dynamic mechanical analysis (DMA) was performed on a DuPont 9900 thermal analyzer system. A sample 10 mm in length, 2 mm in width, and approximately 0.08 mm in thickness was used. The dynamic shear modulus was measured at a resonance mode. Wide-angle X-ray diffraction patterns were obtained at room temperature on a Philips PW 1730-10 X-ray diffractometer using nickel-filtered Cu Kα radiation. Tensile properties were determined stress-strain curves with a Toyo Baldwin Instron UTM-III-500 with a load cell of 10 kg at a drawing speed of 5 cm/min. Measurements were performed at room temperature with film specimens (6 cm long, 0.5 cm wide, and 0.1 mm thick), and an average of at least five individual determinations was reported.

RESULTS AND DISCUSSION

Monomer Synthesis

The naphthalene unit-containing new polymer-forming diacid chlorides (1, 1,4-NCL) and (2, 2,6-NCL) were successfully synthesized from 1,4-bis(4-carboxyphenoxy)naphthyl and 2,6-bis(4-carboxyphenoxy)naphthyl, respectively, with thionyl chloride. The yield was high, with up to 80% after recrystallization. The chemical structures of all of the newly synthesized compounds were confirmed by means of elemental analysis, the characteristic peaks in the NMR spectra, and IR spectroscopic techniques. Concerning the synthesis of dicarboxylic acid chlorides 1 and 2, the disappearance of a characteristic absorption bands at around 1680 cm^{-1} (C═O) and 2500–3500 cm^{-1} (O—H) on the IR spectra revealed completion of the chlorination of the corresponding dicarboxylic acid compounds, and the most characteristic band of diacid chlorides (1,4-NCL and 2,6-NCL) shifted from 1680 cm^{-1} (free acid) to near 1770 cm^{-1} (C═O stretching). According to the basis of the shielding effect of carbon, the positions of chemical shifts for carbon were readily assigned from the ^{13}C-NMR experiments compound 1 and 2. The ^{13}C-NMR spectra of 1 and 2 exhibited exactly 10 peaks due to symmetry and were consistent with the calculated values. The elemental analysis values of all of the compounds were also in good agreement with the calculated values for the proposed structures.

Polymer Synthesis

The high-temperature solution polycondensation is a convenient method for the preparation of polyarylate on a laboratory scale.[13] Two series of new 1,4-naphthyl and 2,6-naphthyl containing high-molecular-weight polyarylates, (4_a–4_f) and (5_a–5_f), were prepared from the diacid chlorides, 1 and 2, respectively, with various bisphenols 3^c–3_f by the high-temperature solution polycondensation reaction (Scheme 1). Generally, the molecular weight of the polyesters obtained from the reaction is highly dependent on the monomer concentration, reaction temperature, and reaction time. Hence, the effects of these reaction parameters were first studied to find out a suitable reaction condition according to previously reported procedures.[7] Various polyarylates 4 and 5 were prepared from 1 and 2 with the corresponding aromatic diols 3 by using the most favorable reaction conditions.

Table I. Synthesis of Polyarylates[a]

Polymer Code	η_{inh}[b] (dL/g)	M_n[c] ($\times 10^4$)	M_w/M_n[c]	Remarks[d]
4a	—	—	—	P
4b	1.07	7.1	3.2	S
4c	1.28	8.9	2.3	S
4d	0.39	2.2	1.2	S
4e	0.61	4.8	1.7	S
4f	1.24	13.7	1.9	S
5a	—	—	—	P
5b	1.17	9.5	3.4	S
5c	0.57	—	—	P
5d	0.42	2.0	1.2	S
5e	0.88	6.3	2.2	S
5f	0.70	4.6	2.1	S

[a] Polymerization was carried out with 2.5 mmol of each monomer in 8 mL of o-dichlorobenzene at 200°C for 20 h.
[b] Measure at a concentration of 0.5 g/dL in o-chlorophenol at 30°C.
[c] Determined by GPC on the basis of polystyrene calibration in NMP.
[d] Appearance of the polymerization system: S, homogeneous solution; P, polymer precipitation.

The results of the polycondensation are listed in Table I. All the polyarylates were obtained in almost quantitative yield. The polyarylates had inherent viscosities ranging from 0.39 to 1.28 dL/g. The GPC curves of polyarylates indicated that M_n values were 20,000–137,000 relative to standard polystyrene, and M_w/M_n values, a measure of molecular distribution, were 1.2–3.4. The molecular weight of polyarylates 4_a, 5_a, and 5_c were low. This was probably due to the fact that the poor solubility of these polymers resulted in an early precipitation during the course of polymerization, which retarded further polymerization. The unsatisfactory results obtained from bis(4-hydroxyphenyl)sulfone (3_d) might be attributed to its lower nucleophilicity owing to the presence of the electron-withdrawing sulfonyl group. Transparent films of the polyarylates could be obtained by casting the resulting polymers from DMAc solutions. The formation of polyarylates was confirmed by means of IR spectroscopy. In the FTIR spectra, the polyarylates exhibited characteristic ester absorption bands at near 1735 cm^{-1}.

Polymer Characterization

The solubility of the polyarylates was studied in various solvents, and the results are summarized

Table II. Solubility of Polyarylates[a]

Solvent	Polymer				
	4b–4f	5d–5f	5b	5c	4a, 5a
N-Methyl-2-pyrrolidone	+ +	+ +	+ +	+	−
N,N-Dimethylacetamide	+ +	+ +	+ +	+	−
o-Chlorophenol	+ +	+ +	+ +	+	−
Chloroform	+ +	+ +	+	−	−
Tetrahydrofuran	+ +	+ +	±	−	−
m-Cresol	+	+	−	−	−
1,4-Dioxane	±	±	−	−	−
Methanol	−	−	−	−	−
Acetone	−	−	−	−	−

[a] + +, Soluble at room temperature; +, soluble on heating; ±, swelling on heating; −, insoluble.

in Table II. Besides polyarylates 4_a and 5_a with symmetric and rigid p-phenylene diol structure was quite insoluble in any organic solvents. All the other polyarylates were highly soluble in organic solvents such as NMP, DMAc, and o-chlorophenol. Polymers 4_d–4_f and 5_d–5_f, containing sulfone, isopropylidene, and fluorene group, respectively, showed excellent solubility in all test solvents, even in chloroform and tetrahydrofuran. X-ray diffraction results, as discussed below, revealed that polymers 4_a, 5_a, and 5_c had higher crystallization tendency among all the polymers. The solubility behavior was consistent with the results of X-ray diffraction studies. Thus, the solubility of polyarylates was found to be greatly improved by the introduction of a bulky, cranked, and symmetrical 1,4-naphthyl and 2,6-naphthyl units into the polymer backbone. The solubility studies also revealed that polyarylates 4 had better solubilities than polyarylates 5 in many organic solvents, where the bulky effect of 1,4-naphthyl structure is greater than that of 2,6-naphthyl. Transparent and colorless films for polymers 4 and 5 could be cast onto glass plates from their DMAc solutions.

The structural characterization was made first by X-ray methods with the prepared powders or films. Polymers 4_a and 5_a show sharp diffraction peaks which indicate the presence of a high degree of crystallinity, as shown in Figure 1. Since the diffraction patterns of 4_a and 5_a are rather similar in the positions of their peak angles, similarity in lattice parameters for both polymers is expected. Polymer 5_c shows the semicrystalline diffraction pattern and exhibits crystalline peaks appearing (2θ) at around 20°, whereas all of the other polymers were completely amorphous. Thus, the amorphous nature of these polymers was reflected in their good solubility, which is in agreement with the general rule that solubility decreases with increasing crystallinity, and this could be attributed to the incorporation of bulky and cranked 1,4-naphthyl or 2,6-naphthyl unit

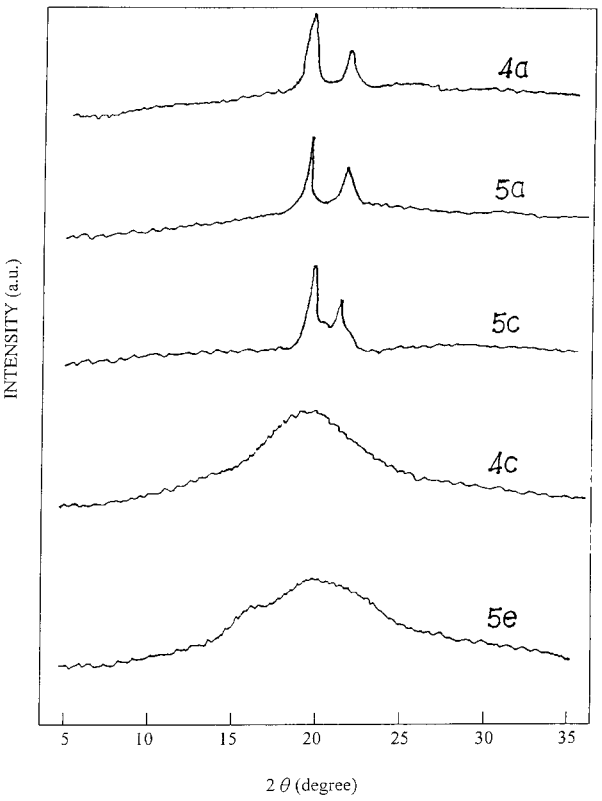

Figure 1. X-ray diffraction diagrams of polyarylates.

Table III. Tensile Properties of Polyarylate Films[a]

		Polymer						
		4b	4c	4e	4f	5b	5e	5f
Yield strength	(MPa)	80.7	55.8	—	—	54.0	65.2	97.0
Tensile strength	(MPa)	75.8	56.2	81.8	82.3	54.9	76.4	84.2
Elongation at break	(%)	9.8	6.1	6.6	5.3	12.4	19.0	9.2
Tensile modulus	(GPa)	2.8	2.0	2.5	2.6	2.3	2.6	2.0

[a] Polymer films were prepared by slow evaporation of their DMAc solution at 90°C for 12 h, then vacuum-dried at 150°C for 10 h.

along the polymer backbone. Polymers 4 and 5 could be processed into good-quality films and were subjected to tensile test. The mechanical properties were determined with an Instron machine. All the tensile properties are also summarized in Table III. All specimens showed high yield or tensile strengths up to 97.0 MPa. Except for polymers 4_e and 4_f, all other polymers yielded during tensile testing and had fairly high elongation to break, indicative of high toughness. Polymers 4_d and 5_d could also be cast into films but were too brittle to be measured.

The thermal behavior of the polymers was evaluated by DSC, TMA, DMA, and TG. The thermal properties of the polymers are summarized in Table IV. Representative DSC and TG curves of polymer 4_e are shown in Figure 2. All the other polymers showed similar patterns of decomposition with no significant weight loss up to 410°C in both air and nitrogen atmospheres, and the temperatures at 10% weight loss were above 465°C on the TG curves in both atmospheres. Thus, introduction of 1,4-naphthyl and 2,6-naphthyl units into polymer backbone resulted in polyarylates with high thermal stability. In addition, there was no obvious difference in thermal stability between polyarylates 4 and polyarylates 5.

The influence of residual water or solvent and history of thermal annealing are sometimes observed in the first heating scan of DSC. Quenching from elevated temperature to room temperature yields samples with similar heat histories so that the glass transition temperatures (T_gs) of these polyarylates could be easily measured in the DSC charts of the second heating trace, and observed in the range of 172–280°C, depending on

Table IV. Thermal Properties of Polyarylates

Polymer Code	T_s[a] (°C)	T_g[b] (°C)	T_g[c] (°C)	T_d[d]		T_{10}[d]		Char yield[e] (%)
				Air	N_2	Air	N_2	
4a			192	400	410	465	505	48
4b	182	212	175	390	400	450	475	49
4c	180	209	186	400	410	465	500	42
4d			208	400	400	450	450	37
4e	202	224	204	410	420	465	490	35
4f	231	310	263	400	410	475	490	50
5a			202	400	400	475	510	40
5b	172	205	172	395	395	470	475	41
5c			182	395	390	455	495	41
5d			188	395	395	450	450	47
5e	197	225	205	400	400	450	490	35
5f	260	310	280	400	400	485	490	48

[a] Softening temperature measured by TMA at a heating rate of 5°C/min.
[b] Glass transition measured by DMA using the shear mode at a heating rate of 5°C/min.
[c] From the second heating traces of DSC measured conducted at a heating rate of 10°C/min in nitrogen.
[d] T_d and T_{10} are decomposition of initial and 10% weight loss, respectively, measured by TG at a heating rate of 10°C/min.
[e] Char yield at 800°C in nitrogen.

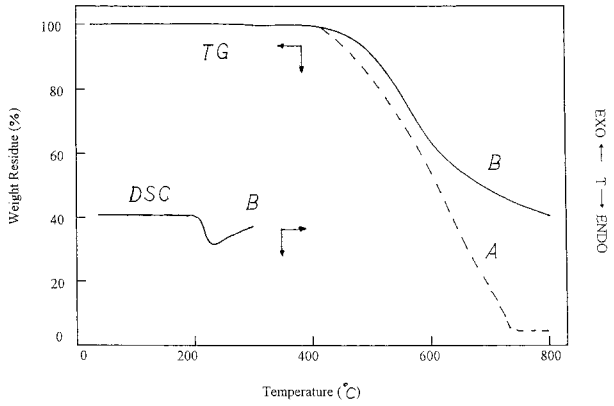

Figure 2. DSC and TG curves for polyarylate 4e at a heating rate 10°C/min (A) in air, (B) in nitrogen.

the structure of bisphenol component, and decreased with decreasing rigidity of polymer backbone, or with introduction of flexible linkages in the polymer main chain. The large window between T_g and the decomposition temperature makes these polymers attractive for practical processing comparing with the commercial polyarylate U-polymer that has a T_g of about 190–195°C.

Thermal properties of polyarylates 4c, 4e, and 4f measured by TMA are shown in Figure 3. When the temperature reaches 202°C, the film of 4_e softens and displays a softening transition temperature (T_s). The appearance of a softening transition at 202°C is in very good agreement with the glass transition temperature (T_g), as obtained by DSC. All other polymers also show softening transition temperatures between 172 and 260°C.

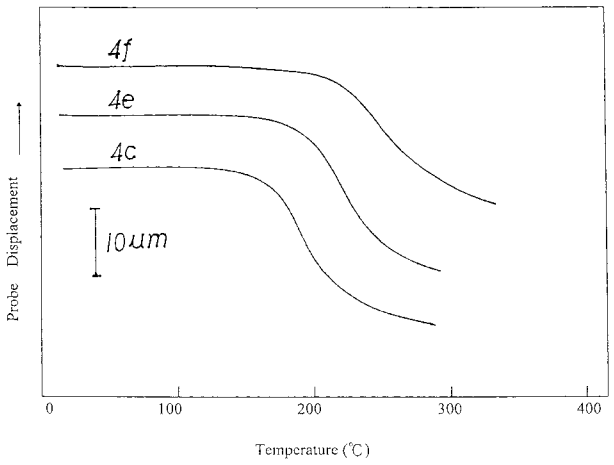

Figure 3. TMA curves of polyarylate films.

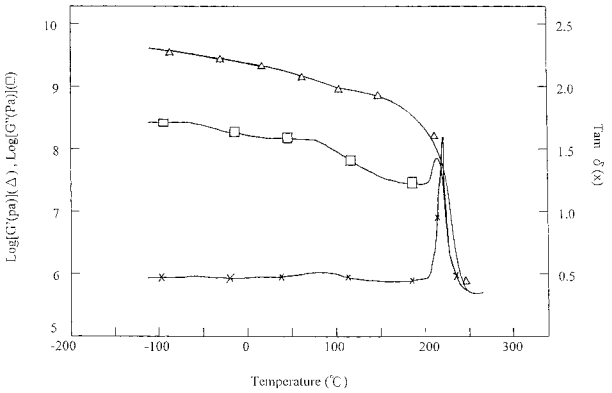

Figure 4. Mechanical-dynamic spectra of 5_e at a heating rate 5°C/min.

More detailed information can be obtained from the dynamic mechanical measurements taken of the film as a function of temperature. Films of about 80 μm thickness were prepared by casting from DMAc solutions, and were studied between −100°C and to 350°C. Figure 4 presents the mechanical relaxation spectra of polyarylate 5_e. Three relaxations appeared at ca. −50, 80, and 225°C, based on tan δ and G″ peaks. The relaxation process can be assessed by verifying results obtained for other related polymers.[14–17] The low-temperature transition named γ (ca. −50°C) is a typical relaxation for polyesters. This relaxation is attributed to the reorientation of the carbonyl groups. The second transition, named β, at 80°C, is associated with approximately a quarter of one order of magnitude step decrease in G′. Small transition peak in tan δ and broad disperse peak in G″ also appear in β relaxation. This transition has been assigned to the mobility of the side chains accompanied by some conformational rearrangements of the main chain.[14–16] A comparison of the mechanical properties behavior of the system studied here with that of another polyester, studied by Kallitsis,[14,15] showed that the main difference was the relatively high temperature of β transition appearing in the relatively short side chain (methyl group) in this article. In addition, the glass transition at around 225°C is associated with an approximately 2 orders of magnitude decrease in G′. The mechanical relaxation spectra of 4_e resemble those of 5_e. Three relaxation transitions were also observed in the polymer 4_e, based on tan δ and G″ peaks. However, the β transition of 4_e shifted to 100°C. The glass transition of 4_e appeared at 224°C.

Figure 5 illustrates the mechanical relaxation spectra of 4_c. Two relaxation transitions appeared

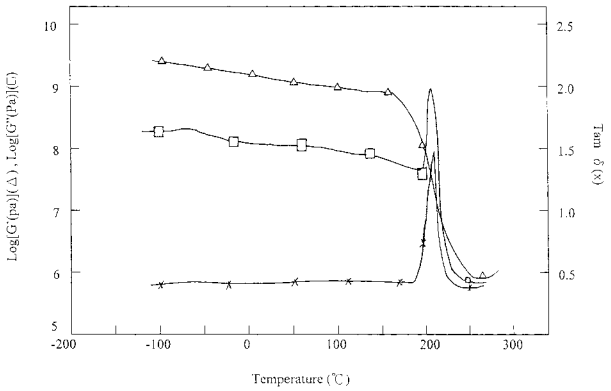

Figure 5. Mechanical-dynamic spectra of 4_c at a heating rate 5°C/min.

at ca. −50 and 209°C, based on tan δ and G″ peaks. A broad disperse γ peak appeared at around −50°C in tan δ and G″. The reason for such a transition resembles that of the γ transition of 5_e. However, the β subglass transition around 80°C was not observed in 4_c. This finding is attributed to the fact that 4_c does not contain aliphatic side group. The glass transition of 4_c at 209°C is associated with an approximately 2 orders of magnitude decrease in G′. The mechanical relaxation spectra of the other polyesters (4_b, 4_f, 5_b, and 5_f) resembles those of 4_c. Table IV also summarizes the glass relaxation temperatures of 4 and 5. The relatively high glass transition temperatures of 4_f and 5_f appeared at 310°C. This finding is due to the fact that the rigid and bulky fluorene groups inhibited the rotation of bonds, resulting in an increasing chain stiffness.

CONCLUSIONS

New polyarylate-forming dicarboxylic acid chlorides having bulky and cranked structure were successfully synthesized starting from 1,4-bis(4-carboxyphenoxy)naphthyl and 2,6-bis(4-carboxyphenoxy)naphthyl with thionyl chloride. These diacid chlorides (1 and 2) monomers were subjected to the high-temperature solution polycondensation with various bisphenols, giving high-molecular-weight polyarylates. The introduction of bulky and cranked 1,4-naphthyl or 2,6-naphthyl unit into the polymer backbone highly improved solubility of the polyarylates in organic solvents. The polyarylates had glass transition temperatures of 172–280°C and 10% weight losses above 450°C in air, indicating high thermal stability. Thus, these polyarylates are considered to be high thermally stable polymeric materials. Transparent and colorless films of these polymers could be cast, and display good mechanical properties. The short methyl side group in the bisphenol A-based polyarylates also induce the subglass transition appearing at about 80°C.

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