Syntheses and Properties of New Poly(amide-imide)s Based on 1,4-Bis(4-aminophenoxy)naphthalene and Various Diimide-Diacids

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ABSTRACT: A series of new alternative poly(amide–imide)s (PAIs, III_{a-j}) was synthesized by the direct polycondensation of 1,4-bis(4-aminophenoxy)naphthalene (1,4-BAPON) with various aromatic diimide–diacids. These polymers were obtained in quantitative yields with inherent viscosities of 0.71–1.03 dL/g. Except for III_a, most of the polymers were soluble in aprotic polar solvents such as NMP, DMAc, DMF, and DMSO and could be solution-cast into transparent, flexible, and tough films. The glass transition temperatures of these PAIs were in the range of 235–280°C. Thermogravimetric analyses established that these polymers were fairly stable up to 450°C, and 10% weight loss temperatures were recorded in the range of 520–569°C under nitrogen and 506–566°C under an air atmosphere. Compared with the PAIs with the 1,4-bis(4aminophenoxy)benzene structure (series IV), the solubility of series III was better than that of series IV. © 2000 John Wiley & Sons, Inc. J Appl Polym Sci 77: 217–225, 2000

Key words: 1,4-bis(4-aminophenoxy)naphthalene; aromatic poly(amide–imide); direct polycondensation

INTRODUCTION

Aromatic poly(amide–imide)s (PAIs) are useful high-performance materials that exhibit better processing characteristics when compared with polyimides of analogous structures.^{1,2} Conventionally, PAIs can be prepared in several ways, starting from trimellitic anhydride (TMA): First, two-step polycondensation from acyl chloride of TMA with aromatic diamines involving polycondensation and subsequent cyclodehydration³; second, low-temperature-solution polycondensation of a TMA-derived imide ring-performed diacyl chlorides and aromatic diamines³; and, third, polycondensation of a TMA or TMA-derived imide ring-containing dicarboxylic acids with diisocyanates.^{5–7} Recently, we successfully applied a direct phosphorylation reaction⁸ to synthesize high molecular weight PAIs by direct polycondensation of imide-containing dicarboxylic acids with aromatic diamines.^{9–14} This direct polycondensation route avoids using moisture-sensitive acyl chloride or isocyanates and provides significant advantages in the laboratory preparation of PAIs.

Acceding to recent publications,^{15–19} the introduction of a naphthalene ring into the polymer backbone has some advantages for structure modification such as increasing the solubility of polymers, lowering the phase-transfer temperature,

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Polymer	NMP (mL)	$\operatorname{CaCl}_{2}\left(\mathbf{g}\right)$	Py (mL)	$\eta_{\rm inh}{}^{\rm a}(\rm dL/g)$
IIIa	5.5	0.55	1.5	0.73^{b}
III	4.0	0.41	1.2	0.86
ĨIJ	4.0	0.35	1.1	0.71
III _d	5.0	0.46	1.2	0.78
III	5.0	0.45	1.2	0.75
III _f	4.0	0.35	1.0	0.83
III	4.5	0.42	1.0	0.87
III _b	3.5 + 1	0.32	0.8	1.03
III _i	3.5	0.33	0.8	0.76
III _i	4.0	0.32	1.0	0.93
IV	6.0	0.65	1.5	$0.11^{ m c}$
IV _b	5.6 + 1	0.5	1.2	0.87
IVe	6.0 + 0.5	0.4	1.2	1.13
IVd	5.0 + 1.5	0.5 + 0.2	1.2	$0.45^{ m b}$
IV	6.0 + 1.0	0.6 + 0.1	1.2	$0.71^{ m b}$
IV _f	5.0 + 3.5	0.5	1.2	$0.79^{ m b}$
IV "	6.0	0.7	1.5	$0.14^{ m c}$
IVh	6.0 + 2	0.7	1.5	$0.19^{ m c}$
IV,	5.0 + 1.5	0.5	1.2	0.84^{b}
IV _j	5.0	0.45	1.2	$0.95^{ m b}$

Table I Reaction Conditions for the Preparation of PAIs

In each polymerization, 1 mmol of monomer and 0.6 mL of TPP was used. Reaction temperature = 100° C; reaction time = 3 h. ^aMeasured at a polymer concentration of 0.5 g/dL in DMAc at 30°C.

^bMeasured at a polymer concentration of 0.5 g/dL in DMAc + 0.5 wt % LiCl at 30°C.

^cMeasured at a polymer concentration of 0.5 g/dL in concentrated H_2SO_4 at 30°C.

and retaining the thermal stability. 1,4-Bis(4aminophenoxy)naphthalene (1,4-BAPON), containing a structure similar to 1,4-bis(4-aminophenoxy)benzene, has a substituted phenyl ring in the 2,3-position that forms an asymmetric structure in the 2,3- and 5,6-positions of the benzene ring. Because the synthesis of **1,4-BAPON** is more difficult than that of the other bis(aminophenoxy)naphthalenes,²⁰ the properties of PAIs having 1.4-BAPON units in the main chains have not been reported. In this work, a series of new 1,4-BAPON-containing PAIs was prepared by the direct polycondensation of 1,4-BAPON with various diimide-diacids using triphenvl phosphite (TPP) and pyridine as a condensing agent. Physical properties of these new PAIs were reported and also compared with the PAIs (IV_{a-i}) derived from 1,4-bis(4-aminophenoxy)benzene (**TPE-Q**). Although the diamine **TPE-Q** is not a new monomer, the PAIs IV_{a-i} have not been systematically reported on concerning their preparations and physical properties.

EXPERIMENTAL

Materials

1,4-Dihydroxynaphthalene (from TCI), p-fluoronitrobenzene (from Janssen), 10% palladium on activated carbon (Pd/C; from Fluka), TMA (from Wako), TPP (from Fluka), and TPE-Q (Ig from TCI) were used as received. Aromatic diamines including p-phenylenediamine (I_a), m-phenylenediamine (I_b) , 2,4-diaminotoluene (I_c) , 4,4'-diaminophenyl methane (I_d) , 4,4'-diaminophenyl ether $(\mathbf{I}_{\mathbf{e}})$, and 3,4'-diaminophenyl ether $(\mathbf{I}_{\mathbf{f}})$ were used directly as received from TCI. 1,3-Bis(4aminophenoxy)benzene (I_h), 2,2-bis[4-(aminophenoxy)phenyl]propane (Ii), and 2,2-bis[4-(aminophenoxy)phenyl]hexafluoropropane (I_i) were obtained from Chriskev and used without further purification. Reagent-grade calcium chloride was dried under a vacuum at 180°C before use. N-Methyl-2-pyrrolidone (NMP), N,N-dimethylformamide (DMF), and pyridine were purified by





distillation under reduced pressure over calcium hydride and stored over 4 Å molecular sieves.

Monomer Synthesis

According to the method reported in a previous article,²⁰ **1,4-BAPON** was synthesized starting from the nucleophilic displacement of 1,4-dihydroxynaphthalene and *p*-fluoronitrobenzene in the presence of potassium carbonate, giving 1,4-bis(4-nitrophenoxy)naphthalene (**1,4-BNPON**), and followed by reduction with a hydrazine and Pd/C system (mp 113–115°C). Diimide–diacids $(\mathbf{II}_{\mathbf{a}-\mathbf{j}})$ were prepared according to a previous method 9,10 by condensing diamines $(\mathbf{I}_{\mathbf{a}-\mathbf{j}})$ with TMA followed by dehydration in a toluene–water azeotropic distillation.

Polymerization

The PAIs (series **III**) were prepared by direct polycondensation using the following general procedure: Taking **III_h** as an example, a mixture of 0.342 g (1.0 mmol) of diamine **1,4-BAPON**, 0.640 g (1.0 mmol) of diimide–diacid **II_h**, 0.32 g of calcium chloride, 3.5 mL of NMP, 0.8 mL of pyridine,

		Elemental Analaysis (%) ^a				
Polymer	Formula (Molecular Weight)		С	Н	Ν	Moisture Intake (%) ^b
III。	$(C_{46}H_{26}N_4O_8)_n$	Calcd	72.77	3.44	7.34	
a	$(762.71)_n$	Found	69.55	3.68	7.21	4.19
		Corrected	72.59	3.53	7.52	
III _b	$(C_{46}H_{26}N_4O_8)_n$	Calcd	72.77	3.44	7.34	
b	$(762.71)_n$	Found	69.86	4.06	7.18	3.72
		Corrected	72.55	3.91	7.45	
III	$(C_{47}H_{28}N_4O_8)_n$	Calcd	72.67	3.63	7.21	
e	(776.76),	Found	69.95	3.86	7.05	3.99
		Corrected	72.80	3.70	7.34	
III_d	$(C_{53}H_{32}N_4O_8)_n$	Calcd	74.64	3.78	6.57	
u	$(852.84)_n$	Found	72.14	3.82	6.49	3.38
		Corrected	74.65	3.67	6.71	
III	$(C_{52}H_{30}N_4O_9)_n$	Calcd	73.10	3.54	6.55	
e	$(854.84)_n$	Found	71.48	3.67	6.37	2.09
		Corrected	73.01	3.60	6.51	
III _f	$(C_{52}H_{30}N_4O_9)_n$	Calcd	73.10	3.54	6.55	
1	$(854.84)_n$	Found	71.62	3.64	6.27	2.02
		Corrected	73.11	3.54	6.55	
IIIa	$(C_{58}H_{34}N_4O_{10})_n$	Calcd	73.57	3.62	5.92	
8	(946.93),	Found	70.59	3.71	6.34	3.45
		Corrected	73.10	3.58	6.55	
III _b	$(C_{58}H_{34}N_4O_{10})_n$	Calcd	73.57	3.62	5.92	
п	(946.93),	Found	71.63	3.56	5.99	2.62
		Corrected	73.66	3.56	5.91	
III,	$(C_{67}H_{44}N_4O_{10})_n$	Calcd	75.55	4.16	5.26	
1	$(1065.11)_{n}$	Found	73.42	4.30	5.13	2.81
		Corrected	75.54	4.18	5.27	
III,	$(C_{67}H_{38}F_6N_4O_{10})_{10}$	Calcd	68.60	3.26	4.77	
J	(1173.06),	Found	66.95	3.37	4.68	2.38
	16	Corrected	68.58	3.29	4.79	

Table II Elemental Analysis of PAIs

^aFor C and N: corrected value = found value \times (100% + moisture intake %). For H: corrected value = found value \times (100% - moisture intake %).

^bMoisture intake (%) = (W – W₀)/W₀ × 100%. W is the weight of polymer sample after standing at room temperature for 3 days. W_0 is the weight of polymer sample after dried in vacuum at 100°C for 12 h.

and 0.6 mL of TPP was heated with stirring at 100°C. The polymer solution was too viscous to stir after 1 h and was diluted gradually with an additional 1 mL of NMP to maintain smooth stirring. After 3 h of stirring at 100°C, the viscous polymer solution was slowly poured into 400 mL of stirred methanol to give rise to a stringy precipitate which was washed thoroughly with methanol and hot water, collected by filtration, and dried at 100°C under a vacuum. The yield was quantitative. The inherent viscosity of polymer III_h was 1.03 dL g⁻¹, as measured at a concentration of 0.5 g/dL in DMAc at 30°C. Other PAIs in this series (III_{a-j}) were synthesized analogously.

PAIs **IV** were prepared by a similar process as above, but the diamine monomer **TPE-Q** was used to replace **1,4-BAPON**. The amounts of solvents and calcium chloride were larger to synthesize PAIs. Synthetic conditions and inherent viscosities of the obtained polymers are summarized in Table I.

Measurements

Melting points were measured in capillaries on a Yamato mp apparatus (Model MP-21) without correction. Elemental analyses were run on a Perkin–Elmer Model 2400 C, H, N analyzer. The inherent viscosities of PAIs were measured at a 0.5 g/dL concentration with a Cannon–Fenske

		Solvent							
Polymer	DMAc	NMP	DMF	DMAc + 5% LiCl	DMSO	Ру	<i>m</i> -Cresol		
III。	_	+-	_	+	_	_	_		
III	+	+	+	+	+-	+	_		
III	+	+	+	+	+	+	+		
III _d	+	+	+	+	+	+	+		
III	+	+	+	+	+-	+	+-		
III	+	+	+	+	+	+	+		
III,	+	+	+	+	+	+-	+-		
III _b	+	+	+	+	+	+	+		
III	+	+	+	+	+	+	+		
III	+	+	+	+	+	+	+		
IV _a	_	—	_	+-	_	_	_		
IV _b	+	+	+	+	+	+	+		
$\overline{IV_{e}}$	+	+	+	+	+	+	+		
IV _d	+	+	+	+	+	+	+		
IV _e	+-	+-	_	+	_	_	_		
IV_{f}	+-	+-	_	+	_	_	—		
IV_{g}	_	_	_	+-	—	—	—		
IV_{h}	_	—	_	—	_	_	_		
IV _i	+-	+	_	+	_	_	_		
IV_j	-	+	-	+	—	-	-		

Table III Solubility of PAIs

Solubility: (+) soluble at room temperature, (+-) partially soluble or swelling, (-) insoluble.

viscometer at 30°C. Thermogravimetric analysis (TGA) was conducted with a Rigaku thermoflex TG 8110 coupled to a Rigaku TAS-100 thermal analysis station. Experiments were carried out on 10 ± 2 -mg samples heated in flowing nitrogen or air (50 cm³/min) at a heating rate of 20°C /min. Differential scanning calorimetry (DSC) traces were performed on a Sinku-Riko DSC-7000 differential scanning calorimeter coupled to a TA 7000 thermal analyzer in flowing nitrogen $(30 \text{ cm}^3/$ min) at a heating rate of 20°C/min. Wide-angle X-ray diffraction patterns were obtained on a Rigaku Geiger D-Max IIIa X-ray diffractometer, using Ni-filtered CuK α radiation (40 kV, 15 mA) with film specimens of about 0.1 mm in thickness. The scanning rate was 2°/min. An Instron Universal Test Model 1130 with a load cell of 5 kg was used to study the stress-strain curves of the samples. A gauge of 2 cm and a strain rate of 5 cm/min were used. Measurements were performed at room temperature with film specimens (0.5 cm wide, 6 cm long, and 0.1 mm thick), and the average of at least six individual determinations was used.

RESULTS AND DISCUSSION

Synthesis

A series of novel poly(amide-imide)s (PAIs, III_{a-i}) was synthesized by the direct polycondensation of 1,4-BAPON with various aromatic diimidediacids (II_{a-i}) , as shown in Scheme 1. As reported recently,²⁰ the diamine **1,4-BAPON** was prepared from the displacement reaction between 1,4-dihydroxynaphthalene and p-fluoronitrobenzene followed by catalytic reduction. Diimide-diacids (II_{a-i}) were synthesized from the condensation of diamines (I_{a-i}) with TMA followed by dehydration. Generally, to obtain PAIs of high molecular weight in a direct polycondensation, the solubility of the polymer seems to be an important factor; although the optimum condition may vary with different kinds of monomers. Under homogeneous conditions, higher polymer concentrations and a suitable amount of salt (CaCl₂) were favorable for polymerization.

The synthesis conditions and inherent viscosities of PAIs $\mathbf{III}_{\mathbf{a}-\mathbf{j}}$ and $\mathbf{IV}_{\mathbf{a}-\mathbf{j}}$ are listed in Table I. The inherent viscosities of $\mathbf{III}_{\mathbf{a}-\mathbf{j}}$ were in the



SCATTER ANGLE (2 θ)

Figure 1 Wide-angle X-ray diffractograms of polymers.

range of 0.71–1.03 dL/g. In series **III**, the diamine **1,4-BAPON** exhibited good solubility in organic solvents, making reaction conditions dependent

on the kind of diimide-diacid. Larger amounts of solvents and salt were needed to prevent the precipitation from occurring and to allow the direct polycondensation reaction to process smoothly for PAIs III_{a,d,e} derived from rigid diimide-diacid moieties. For polymers III_{g.h.i} containing flexible diacid moieties, solvent and salts could be reduced to a smaller amount than those of $III_{a,d,e}$. With the polymer solution of III_h too viscous to stir during the reaction period, an additional 1 mL of NMP was added to maintain stirring and to provide a high molecular weight for the polymer. PAIs IV_{a-i}, synthesized from TPE-Q and aromatic diimide-diacids using a similar procedure, were prepared for physical property comparisons with series III. The amounts of solvent and salts needed for preparation of PAIs IV were larger than the corresponding polymers III, because the diamine monomer TPE-Q was more rigid than 1,4-BAPON. Polymers IV_{a,g,h} precipitated during the polymerization, even though larger amounts of solvent and salt were added to the solution, so lower molecular weights of the polymer were obtained.

The results of the elemental analyses of all the PAIs are shown in Table II. The carbon values were found to be lower than the calculated values for the proposed structures in all cases. This is attributed to the hygroscopic nature of amide groups of these PAIs. The intake of water was in the range of 2.09-4.19%, which were calculated from the weight change of the vacuum-dried polymer samples after being exposed in air at room temperature. When the found values were cor-

Polymer ^a	Yield Strength (MPa)	Tensile Strength (MPa)	Elongation at Break (%)	Initial Modulus (GPa)
III _b	_	90	10.8	2.1
III	86	78	10.4	2.0
III _d	_	82	8.9	2.3
III	_	93	9.3	2.1
III _f	_	96	9.5	2.2
III,	108	103	12.9	2.4
III	_	99	12.5	2.3
III,	97	86	10.5	2.0
III,	112	105	12.3	2.5
IV	82	81	17.0	2.4
IV _c	89	87	15.4	2.1
IV_d	—	85	10.3	2.2

Table IVTensile Properties of PAIs

^aFilms were cast from polymer solution of DMAc.

	DSC	TGA			
		Decomposition	Temperature ^b (°C)		
Polymer	$T_g^{\rm \ a} (^{\rm o}{\rm C})$	In $\rm N_2$	In Air	Wt % Residue at 800°C in $\rm N_2$	
IIIa	_	569	566	65.3	
III	280	565	560	64.3	
III	273	534	515	62.5	
III_d	_	529	522	58.3	
III	263	533	523	63.6	
III_{f}	250	531	514	63.4	
III _g	257	545	545	60.1	
III_{h}	240	520	506	60.8	
III _i	235	533	534	61.5	
III_i	250	563	550	59.5	
IV_{a}	_	534	527	62.3	
IV _b	285	562	558	63.2	

Table VThermal Properties of PAIs

^aFrom the second-heating DSC traces conducted at a heating of 20°C/min in nitrogen.

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

rected by eliminating the amount of water absorbed, the correction values were in good agreement with the calculated ones.

Properties of Polymers

The qualitative solubility of PAIs was tested in various solvents at room temperature as a concentration of approximately 1% (w/v). The results are shown in Table III. Except for III_a, PAIs III were readily soluble in polar solvents like NMP, DMAc, DMF, and DMSO. Polymer III_b containing *meta*-oriented benzene in a diimide-diacid residue was easily soluble in test solvents when compared with III_a. On the contrary, most PAIs IV showed poor solubility in organic solvents. The solubility of series III was better than that of series **IV**, especially for the PAIs containing an ether linkage in diimidediacid moieties such as III_{e,f,g,h}. This may be attributed to the introduction of the naphthalene unit into the polymer backbone, a move that reduces the polymer packing density and interchain interaction.

The crystallinity of polymers was evaluated by an X-ray diffraction measurement with scattering angles 2θ from 5°–40°, as shown in Figure 1. All polymers **III** showed amorphous characteristics and exhibited no observable reflection peak in the test range. But in series **IV**, **IV**_{a,g,h} showed crystalline characteristics and exhibited stronger reflection peaks. From this result, the lower crystallinity of series **III** could be confirmed when compared with the related polymers **IV**.

Table IV summarizes the tensile properties of the polymers. Most polymers afforded transparent, tough, and flexible films by casting from their DMAc solutions. The PAI films had yield strengths of 86-112 MPa, tensile strengths of 78-105 MPa, elongations at break of 8.9-12.9%, and initial moduli of 2.0–2.5 GPa. III_{c,g,i,j} showed yield points on their stress-strain curves, revealing the tough characters of polymer films. The films III_{g,j} exhibited the best tensile properties, because their tensile strengths were higher than 100 MPa and their initial moduli higher than 2.4 GPa. The tensile strengths of series **III** were higher than those of series IV, suggesting that the incorporation of the 1,4-BAPON unit into the polymer chain was better for the improvement of processability than that of other related units.

The thermal stability and glass transition temperatures $(T_g$'s) of PAIs were evaluated by thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC), respectively. The 10% decomposition temperatures $(T_d$'s) in air and nitrogen atmospheres and the char yields at 800°C in nitrogen are summarized in Table V. The T_d 's of PAIs **III** were recorded in



Figure 2 TGA curves of III_a and IV_a at a heating rate of 20°C/min under nitrogen.

the range of 520-569°C in nitrogen and 506-566°C in air. The PAIs III_{a.b} containing *p*-phenylene or *m*-phenylene units in the diimidediacids showed the best thermal stability, due to their high aromaticity. The fluorine-containing PAI III_i showed better thermal stability than its nonfluoro analogous III_i due to that the C—F bond of the CF₃ group is stronger than the C—H bond of the CH_3 group. Comparisons of thermal properties of related polymers $\mathbf{IV}_{\mathbf{a},\mathbf{b}}$ are also shown in Table V and Figure 2. The TGA curves of PAIs III_a and IV_a showed that both of them had good thermal stability below 450°C and the T_d of III_a was 35°C higher, a characteristic that may result from the low degree of polymerization of IV_a .

The T_g 's of PAIs were recorded in the range of 235–280°C. However, no discernible T_g was detected for polymers $\mathbf{III}_{\mathbf{a},\mathbf{d}}$ from DSC traces in the test range. Higher T_g 's of $\mathbf{III}_{\mathbf{b},\mathbf{c}}$ in polymers \mathbf{III} may be caused by the rigid structures of diimide-diacids that contain no ether linkages. Polymer $\mathbf{III}_{\mathbf{i}}$ exhibits the lowest T_g because its diacid moiety has a flexible isopropyl group. Polymer $\mathbf{III}_{\mathbf{i}}$

containing bulk CF_3 groups in the diacid moiety have increased steric hindrances, leading to a higher T_g than that of **III**_i.

CONCLUSIONS

A series of novel aromatic PAIs containing 1,4-bis-(phenoxy)naphthalene units was prepared by the direct polycondensation of 1,4-bis(aminophenoxy)naphthalene and various diimide-diacids. Most of the PAIs are characterized by good solubility in organic solvents, excellent mechanical properties, and high thermal stability. Compared with the related isomers, this series of polymers exhibits some advantages such as lower crystallinity, better solubility, and higher thermal stability.

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