Synthesis and Characterization of Novel Polyaspartimides Derived from 2,2'-Dimethyl-4,4'-bis(4-maleimidophenoxy)biphenyl and Various Diamines

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ABSTRACT: A novel bismaleimide, 2,2'-dimethyl-4,4'-bis(4-maleimidophenoxy)biphenyl, containing noncoplanar 2,2'-dimethylbiphenylene and flexible ether units in the polymer backbone was synthesized from 2,2'-dimethyl-4,4'-bis(4-aminophenoxy)biphenyl with maleic anhydride. The bismaleimide was reacted with 11 diamines using *m*-cresol as a solvent and glacial acetic acid as a catalyst to produce novel polyaspartimides. Polymers were identified by elemental analysis and infrared spectroscopy, and characterized by solubility test, X-ray diffraction, and thermal analysis (differential scanning calorimetry and thermogravimetric analysis). The inherent viscosities of the polymers varied from 0.22 to 0.48 dL g⁻¹ in concentration of 1.0 g dL⁻¹ of *N*,*N*-dimethylformamide. All polymers are soluble in *N*-methyl-2-pyrrolidone, *N*,*N*-dimethylformamide, dimethylsulfoxide, pyridine, *m*-cresol, and tetrahydrofuran. The polymers, except **PASI-4**, had moderate glass transition temperature in the range of 375° -426°C in air and 357° -415°C in nitrogen. © 1999 John Wiley & Sons, Inc. J Appl Polym Sci 73: 279–286, 1999

Key words: 2,2'-dimethyl-4,4'-bis(4-maleimidophenoxy)biphenyl; noncoplanar; bis-maleimides; diamines; polyaspartimides

INTRODUCTION

Bismaleimides (BMIs) are a leading class of thermosetting polyimides. Their excellent thermal and mechanical properties have made them extremely popular for advanced composites and electronics.¹⁻³ The need for BMI resins arose because of the poor hot/wet performance of epoxies, and the reason for BMI systems is that they are capable of performing at temperatures up to 230° C.⁴⁻⁶ BMIs can be self-polymerized through their active maleic double bonds to give highly crosslinked, brittle polyimides. However, the disadvantages of the cured resin are poor solubility in ordinary solvents, high processing temperature, and brittleness. Recently, modification of BMI has been an active research field, with substantial success.⁷⁻¹⁰ In particular, diamines have been used to extend BMIs, resulting in polyaspartimides that possess better processing character-

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istics than linear, high-molecular-weight condensation polyimides.^{5,11,12} However, nucleophilic or Michael addition of diaminoarenes (H₂NArNH₂) to N,N'-bismaleimide or to N,N'-bismaleimidoarenes provides linear polyimides that are typified by rigid backbones, high glass transition temperatures ($T_g = 210^{\circ}-300^{\circ}$ C) and limited solubility.^{12,13} Many efforts have been made to chemically modify the structure of these polymers with the aim of improving their solubility and lowering their glass transition temperature to a range that facilitates their processing in melt.^{14–16}

This article presents the synthesis and characterization of a new BMI, containing 2,2'-dimethvl-biphenvlene and flexible ether units in the backbone, and polyaspartimides resulting from the BMI with several diamines via a Michael addition reaction. The incorporation of 2,2'-disubstituted biphenylene in a para-linked polymer chain does not initially change the polymer backbone's rod-like structure, but it does reduce the interchain interaction. The phenyl rings are forced by the 2,2'-disubstitution into a noncoplanar conformation, decreasing the intermolecular forces between the polymer chain.^{17–19} Thus, the crystallization tendency and transition temperatures are markedly reduced, and the solubilities are significantly enhanced. In addition, aryl ether linkages inserted in aromatic polymer main chains could reduce the chain rigidity and further increase the tractabilities without greatly sacrificing thermal stability of these polymers. Therefore, the introduction of both noncoplanar 2,2'disubstituted biphenylene and aryl ether units into the polymer backbone would be expected to prepare novel processable polyaspartimides with good thermal properties.

EXPERIMENTAL

Materials

Maleic anhydride was purchased from Acros and recrystallized from chloroform. N,N-Dimethylformamide (DMF) was dehydrated by CaH₂ overnight and then distilled under reduced pressure. Glacial acetic acid, sodium acetate, and *m*-cresol were purchased from Merck and used as received. 2,2-Bis[4-(4-aminophenoxy)phenyl]hexafluoropropane (**DA-1**), 1,4-bis(4-aminophenoxy)benzene (**DA-5**), 1,3-bis(4-aminophenoxy)benzene (**DA-6**), and 4,4'-bis(4-aminophenoxy)biphenyl (**DA-9**) were supplied from Wakayama Seika Co., Ltd. and used without further purification. 3,3',5,5'-Tetramethyl-2,2-bis[4-(4-aminophenoxy)phenyl]propane²⁰ (**DA-2**), 3,3',5,5'-tetramethyl-bis[4-(4-aminophenoxy)phenyl]sulfone²¹ (**DA-3**), 2,2-bis[4-[2-(4-aminophenoxy)ethoxy]phenyl]propane²² (**DA-4**), 1,4-bis(4aminophenoxy)-2-*tert*-butylbenzene²³ (**DA-7**), 1,4-bis(4-aminophenoxy)-2,5-di-*tert*-butylbenzene²⁴ (**DA-8**) and 5,5-bis[4-(4-aminophenoxy)phenyl]-4,7-methanohexahydroindan²⁵ (**DA-11**) were synthesized and purified according to the method reported elsewhere.

Monomer Synthesis

Scheme 1 illustrates the synthesis of 2,2'-dimethylbiphenyl-4,4'-diol (I), 2,2'-dimethyl-4,4'-bis(4nitrophenoxy)biphenyl (II), 2,2'-dimethyl-4,4'bis(4-aminophenoxy)biphenyl (III), and 2,2'-dimethyl-4,4'-bis(4-maleimidophenoxy)biphenyl (BMI).

2,2' -Dimethylbiphenyl-4,4' -diol (1)¹⁹

To a flask (1 L) equipped with a mechanic stirrer, addition funnel and reflux condenser were charged with 2,2'-dimethyl-4,4'-diamino-biphenyl dihydrochloride (28.5 g, 0.1 mol), 20 mL of hydrochloride (37% wt) solution, and water (250 mL) below 5°C. The aqueous solution of NaNO₂ (17.3 g, 0.25 mol) in 40 mL water was added dropwise into the flask. After the whole quantity was added to the flask, the reaction mixture was allowed to warm to room temperature, and then toluene (400 mL) and water (400 mL) were subsequently added. After addition of toluene, the reaction mixture was heated to 50°C for 4 h. The oil layer was separated and collected. Then, the solution was concentrated by rotary evaporator. The crude red product was obtained and recrystallized from benzene twice. M.p. 121°-123°C. Yield = 35%. IR (KBr): 3280 cm^{-1} (O—H). ¹H NMR [dimethylsulfoxide (DMSO)-d₆, ppm]: 9.2 (s, H_e, 2H), 6.76 (d, H_b, 2H), 6.71 (s, H_d, 2H), 6.59 (d, H_c, 2H), and 1.88 (s, H_a, 6H). ¹³C NMR (DMSO- d_6 , ppm): 156.2 (C₅), 136.9 (C₃), 132.1 (C₂), 130.5 (C₇), 116.5 (C₄), 112.6 (C₆), 19.9 (C₁).













Anal. Calcd for $C_{14}H_{14}O_2$: C, 78.50%; H, 6.54%; found: C, 79.10%; H, 6.78%.

2,2' -Dimethyl-4,4' -bis(4-nitrophenoxy)biphenyl (II)¹⁹

This compound was synthesized by the reaction of I (30 g, 0.14 mol) and *p*-chloronitrobenzene (47.2 g, 0.3 mol) in the presence of potassium carbonate (48.7 g, 0.35 mol) and 250 mL DMF at 160°C for 8 h. The mixture was then cooled and poured into methanol. The crude product was recrystallized from glacial acetic acid to provide brown needle (m.p. 142°–144°C) in 83% yield. The IR spectrum (KBr) exhibited absorptions at 1580 and 1339 cm⁻¹ (NO₂), 1238 cm⁻¹ (C—O—C). ¹H NMR (CDCl₃): δ (ppm) = 2.09 (s, H_a, 6H), 6.96 (d, H_e,

4H), 7.01 (s, H_d, 2H), 7.07 (d, H_c, 2H), 7.17 (d, H_b, 2H), 8.22 (d, H_f, 4H). ¹³C NMR (CDCl₃): δ (ppm) = 163.27 (C₅), 154.00 (C₈), 142.72 (C₁₁), 138.62 (C₂), 137.68 (C₇), 131.15 (C₃), 125.9 (C₁₀), 121.61 (C₄), 117.53 (C₆), 117.24 (C₉), 19.98 (C₁).



Anal. Calcd for $C_{26}H_{20}O_4N_2$: C, 68.42%; H, 4.39%; N, 6.14%; found: C, 67.98%; H, 4.59%; N, 6.22%.

2,2' -Dimethyl-4,4' -bis(4-aminophenoxy)biphenyl (III)¹⁹

The obtained dinitro compound II (45.6 g, 0.1 mol), 0.3 g of 10% Pd-C, and 300 mL ethanol were introduced into a three-necked flask to which hydrazine monohydrate (100 mL) was added dropwise over a period of 0.5 h at 85°C. After the addition was complete, the reaction was continued at reflux temperature for another 24 h. The mixture was then filtered to remove Pd-C. After cooling, the precipitated crystals were isolated by filtration and recrystallized from ethanol and dried in vacuo (m.p. 138°-139°C). The yield was 80%. The IR spectrum (KBr) exhibited absorptions at 3324 and 3406 cm⁻¹ (N-H), 1226 cm⁻¹ (C—O—C). ¹H NMR (DMSO- d_6): δ (ppm) = 6.95 (d, H_b , 2H), 6.82–6.78 (m, H_e and H_c , 6H), 6.70– $6.67 \,(dd, H_d, 2H), 6.63 \,(d, H_f, 4H), 4.99 \,(s, H_g, 4H),$ and 1.90 (s, H_a, 6H). ¹³C NMR (DMSO- d_6): δ $(ppm) = 158.01 (C_5), 145.46 (C_8), 145.43 (C_{11}),$ 137.13 (C₇), 134.09 (C₂), 130.57 (C₃), 121.10 (C₉), 117.36 (C₄), 114.89 (C₁₀), 113.45 (C₆), 19.76 (C₁).



Anal. Calcd for $C_{26}H_{24}O_2N_2$: C, 78.79%; H, 6.06%; N, 7.07%; found: C, 78.41%; H, 6.34%; N, 7.10%.

2,2' -Dimethyl-4,4' -bis(4-maleimidophenoxy)biphenyl (BMI)

To a 100-mL three-necked flask equipped with a magnetic stirrer, reflux condenser, argon inlet, and an addition funnel were added 3.96 g (10 mmol) of **III** and 10 mL of DMF. When **III** was completely dissolved, the solution of 2.16 g (22 mmol) of maleic anhydride dissolved in 5 mL of DMF was added dropwise into the above mixture. After a few minutes, precipitate was observed, and the mixture became opaque and a brown color. Maleic solution was continually added for 15 min, and then the reaction mixture was heated to 55°C for 2 h. After this period, an excess of fused sodium acetate and acetic anhydride was added into the mixture. The reaction was maintained while stirring at 55°C for an additional 2 h. The mixture was cooled to room temperature and

poured into ice-water. White precipitates were obtained and collected by filtration. The precipitate was washed several times with cooled water and subsequently with an aqueous dilute solution of sodium carbonate to remove the residual acetic acid. Then BMI was washed with hot methanol using a Soxhlet extractor. The solid was dissolved in benzene and freezed, subsequently dried in a vacuum by sublimation process, and a pale-yellow powder product was obtained. The yield was nearly 70%. M.p. 161°-163°C. The IR spectrum (KBr) exhibited absorptions at 1772 and 1710 cm^{-1} (imide ring C=O), 1383 and 1163 cm^{-1} (C-N-C), 666 cm⁻¹ (C=C, maleimide ring). ¹H NMR (DMSO- d_6): δ (ppm) = 7.37-7.10 (m, H_a, H_d, H_{e} and H_{f} , 14H), 7.02 (s, H_{c} , 2H), 6.90 (d, H_{b} , 2H), 2.03 (s, H_g , 6H). ¹³C NMR (DMSO- d_6): δ (ppm) = 169.92 (C₁₁), 155.97 (C₇), 155.31 (C₄), 137.74 $(C_6), 135.85 (C_1), 134.58 (C_{12}), 130.83 (C_2), 128.56$ (C₉), 126.57 (C₁₀), 119.92 (C₅), 118.72 (C₈), 115.94 (C_3) , 19.58 (C_{13}) .



Anal. Calcd for $C_{34}H_{24}O_6N_2$: C, 73.37%; H, 4.35%; N, 5.03%; found: C, 73.12%; H, 4.49%; N, 4.83%.

Polyaspartimides Synthesis

The general procedure for polyaddition was as follows: to a 100-mL three-necked flask equipped with a magnetic stirrer, a reflux condenser, thermometer, and nitrogen inlet was added 0.5 g (0.9 mmol) of BMI and 10 mL of m-cresol, when all BMI was dissolved; under a nitrogen atmosphere 0.46 g (0.9 mmol) of diamine DA-1 was added. Then, 0.1 mL of glacial acetic acid used as a catalyst was added into the mixture when **DA-1** was completely dissolved. The reaction mixture was heated to 120°C with constant stirring for 10 days. During this time, an increase in the solution viscosity and a darkening in color were noticed. The reaction mixture was trickled into excess methanol with vigorous stirring. The dark brown precipitate was collected by filtration and washed thoroughly with ethanol and subsequently dried. The polymer was ground to powder, extracted with hot ethanol using a Soxhlet extractor, and subsequently dried in a vacuum oven at 60°C. The yield was nearly 93%. The polymer had an inherent viscosity of 0.48 dL g⁻¹, measured at a concentration of 1.0 g dL⁻¹ in DMF at 30°C. The IR spectrum (KBr) exhibited absorptions at 3338 and 1592 cm⁻¹ (N—H), 1708 cm⁻¹ (C=O). Other polymers were synthesized by an analogous procedure.

Measurements

IR spectra were recorded in the range of 4000- 400 cm^{-1} for the polymers on a JASCO IR-700 spectrometer. The inherent viscosities of all polymers were measured using Ubbelohde viscometer. Elemental analysis was made on a Perkin-Elmer 2400 instrument. Wide-angle X-ray diffraction patterns were performed at room temperature on an X-ray diffractometer (Philips model PW 1710) using Ni-filtered CuK_{α} radiation (35 kV, 25 mA). The scanning DMSO- d_6 using a Joel EX-400 operating at 100.40 MHz for carbon and 399.65 MHz for proton. Thermogravimetric data were obtained on a Du Pont 2200 in flowing nitrogen (60 $\rm cm^3\,min^{-1})$ at a heating rate of 20°C min⁻¹. Differential scanning calorimetry analysis was performed on differential scanning calorimeter (Du Pont 2100) at a heating rate of 20°C \min^{-1} .

RESULTS AND DISCUSSION

Synthesis of Polymers

The linear polyaspartimides (PASIs) were obtained via Michael addition reaction (Scheme 2). This reaction occurs between equimolars of diamine and BMI in the presence of the catalyst: glacial acetic acid. Table I shows the inherent viscosity, elemental analysis, and IR spectra data of the polymers. The inherent viscosity of the polymers in DMF was in the range of 0.22-0.48 $dL g^{-1}$, measured at a concentration of 1.0 g dL^{-1} at 30°C. Results of elemental analysis were in good agreement with calculated values. Some of the characteristic differences observed in the IR spectra of the Michael addition product, compared with the spectrum of BMI, are as follows: there are medium intensity bands at 1590-1655 cm^{-1} due to N—H bending and 3290–3390 cm^{-1} from N—H stretching, suggesting the formation of a succinimide group. Furthermore, the disappearance of the bands ~ 666 cm⁻¹ due to the maleimide ring (C=C stretching) confirmed the addition reaction. The C=O stretching of the imide group of the polymers is in the range of 1707–1713 cm⁻¹. The dark brown and brittle polymer films could be obtained by casting from their tetrahydrofuran solutions.

Characterization of Polymers

The solubility of polyaspartimides in various solvents is shown in Table II. All the polymers were easily soluble at room temperature in polar aprotic solvents such as N-methyl-2-pyrrolidone, N,N-dimethylacetamide, DMF, DMSO, and pyridine; in phenolic solvents, such as m-cresol; and also in common organic solvents such as tetrahydrofuran.

The crystallinity of the **PASIs** was evaluated by wide-angle X-ray diffraction measurement with 2θ from 8° to 40°. All the polymers showed amorphous diffraction patterns. The good solubility and highly amorphous nature of the polymers could be attributed to the structural modification through the incorporation of the flexible units, such as hexafluoroisopropylidene (PASI-1), isopropylidene (PASI-2 and PASI-4), sulfone (PASI-3), ether (PASI-1-PASI-11), and methylsubstituted arylene ether (PASI-2 and PASI-3) groups. It could also be attributed to the incorporation into the polymer structure of bulky substituent and pendant groups like the *tert*-butyl group (PASI-7 and PASI-8) and the cardo group (PASI-11), as well as the 2,2'-dimethyl-biphenylene unit (PASI-1-PASI-11). There was a considerable decrease in the rigidity and a lowering of energy of internal rotation for the polymer chain due to the presence of the flexible units, thus reducing the crystallinity and improving polymer solubility. Incorporating a bulky tert-butyl substituent, the pendant cardo group and noncoplanar conformation of 2,2'-dimethyl-biphenylene in a *para*-linked polymer chain, decreases the intermolecular forces between the polymer chains, due to a looser packing of polymer chain as compared with the unsubstituted polymers. Therefore, the crystallization tendency is markedly reduced, and solubilities are significantly enhanced (Table II).

The thermal behavior of the polymers was evaluated by differential scanning calorimetry and thermogravimetric analysis. Table III presents the thermal properties of the polyaspar-



DA-1-DA-11









Scheme 2 Synthesis of various polyaspartimides (PASIs).

timides. It can be seen that the T_g 's of polymers, except **PASI-4**, are reasonably high, varying between 188° and 226°C. **PASI-4** containing the

most flexible unit, the oxyethylene group, in the polymer chain, exhibited the lowest T_g of 139°C.

			Elemental Ar	Infrared Spectra (cm^{-1})				
Polymer Code			С	Н	Ν	$\nu_{\rm NH}$	$\delta_{ m NH}$	$\nu_{\rm CO}$
PASI-1	0.48	Calcd	69.69	4.90	4.34	3338	1592	1708
		Found	68.81	4.51	4.01			
PASI-2	0.28	Calcd	74.29	5.96	3.47	3390	1596	1709
		Found	73.42	5.36	3.33			
PASI-3	0.28	Calcd	71.50	5.34	3.61	3320	1651	1713
		Found	70.86	5.02	3.28			
PASI-4	0.22	Calcd	73.97	5.79	3.65	3332	1599	1708
		Found	73.09	5.37	3.47			
PASI-5	0.30	Calcd	70.58	5.13	5.14	3332	1593	1708
		Found	69.04	4.81	4.75			
PASI-6	0.36	Calcd	74.48	5.13	5.14	3364	1587	1707
		Found	72.94	5.08	4.93			
PASI-7	0.28	Calcd	73.98	5.65	3.66	3290	1655	1708
		Found	72.84	5.13	3.30			
PASI-8	0.32	Calcd	74.98	5.99	3.35	3300	1654	1713
		Found	73.00	5.58	3.24			
PASI-9	0.28	Calcd	75.02	5.15	3.81	3330	1596	1712
		Found	73.17	4.83	3.53			
PASI-10	0.24	Calcd	72.86	5.44	4.30	3306	1595	1710
		Found	71.91	5.11	3.94			
PASI-11	0.36	Calcd	76.07	5.87	3.63	3338	1652	1713
		Found	74.94	5.57	3.11			

Table I Inherent Viscosity, Elemental Analysis, and IR Data for PASIs

^a Measured in DMF at a concentration of 1.0 g dL⁻¹ at 30°C.

Thermogravimetric analyses revealed that these polymers were stable up to a temperature of \sim 300°C. Their decomposition temperatures of 10% mass loss were in the range of 375°-426°C in air and 357°-415°C in nitrogen. It was observed that the 10% mass loss temperatures of the polymers in air atmosphere are similar to or higher than the loss in nitrogen, indicating that these

	$\operatorname{Solvent}^{\operatorname{b}}$									
Polymer Code	NMP	DMAc	DMF	DMSO	<i>m</i> -Cresol	Pyridine	THF	Chlorobenzene	Acetone	Dichloromethane
PASI-1	+	+	+	+	+	+	+	+-	+-	_
PASI-2	+	+	+	+	+	+	+	+-	+-	_
PASI-3	+	+	+	+	+	+	+	+-	+-	_
PASI-4	+	+	+	+	+	+	+	+-	+-	_
PASI-5	+	+	+	+	+	+	+	+-	+-	_
PASI-6	+	+	+	+	+	+	+	+-	+-	_
PASI-7	+	+	+	+	+	+	+	+-	+-	_
PASI-8	+	+	+	+	+	+	+	+-	+-	_
PASI-9	+	+	+	+	+	+	+	+-	+-	_
PASI-10	+	+	+	+	+	+	+	+-	+-	_
PASI-11	+	+	+	+	+	+	+	+-	+-	_

Table II Solubility of PASIS	Fable II	Solubility	of PASIs ^a
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 $^{\rm a}$ Solubility: +, soluble at room temperature; +–, partial soluble; –, insoluble. $^{\rm b}$ NMP, N-methyl-2-pyrrolidinone; DMAc, N,N-dimethylacetamide; THF, tetrahydrofuran.

		Decomy Tempe (°	position rature ^b C)	
Polymer	T_g^{a}	L. N	T. A.'	Char Yield ^c
Code	(-C)	$\ln N_2$	In Air	(%)
PASI-1	226	411	405	43
PASI-2	186	394	391	38
PASI-3	193	390	400	40
PASI-4	139	372	390	34
PASI-5	226	415	400	38
PASI-6	212	387	400	43
PASI-7	192	381	394	30
PASI-8	189	360	375	49
PASI-9	206	391	398	50
PASI-10	211	375	390	34
PASI-11	188	357	426	49

Table IIIThermal Properties of PASIs

^a From differential scanning calorimetry measurements conducted at a heating rate of 20° C min⁻¹.

 $^{\rm b}$ Decomposition temperature at 10% mass loss was determined by thermogravimetric analysis in nitrogen at a heating rate of 20°C min⁻¹.

° Char yield at 800°C in nitrogen.

PASIs had thermo-oxidative stability in air. Among these polymers, **PASI-11** with the pendant cardo group had the highest thermo-oxidative stability, and exhibited a relatively high mass loss at 10% mass at temperatures up to 426°C. The char yields of these polymers at 800°C in nitrogen atmosphere were above 34%. The polymer **PASI-9** containing the biphenylene unit had the highest char yield of up to 50%.

CONCLUSIONS

The novel polyaspartimides resulted from the Michael addition reaction of BMI, which contains noncoplanar 2,2'-dimethyl-biphenylene units in the backbone, and various diamines were successfully obtained. These polymers exhibited amorphous behavior and excellent solubility. The polymers had moderate T_g values in the range of 139°–226°C and good thermo-oxidative stability, losing 10% mass in the range of 375°–426°C in air and 357°–415°C in nitrogen.

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