

Synthesis and Characterization of New Cardio Poly(ether imide)s Derived from 9,9-Bis[4-(4-aminophenoxy)phenyl]xanthene

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Received 24 October 2010; accepted 24 January 2012

DOI 10.1002/app.36877

Published online in Wiley Online Library (wileyonlinelibrary.com).

ABSTRACT: A series of new cardio poly(ether imide)s bearing flexible ether and bulky xanthene pendant groups was prepared from 9,9-bis[4-(4-aminophenoxy)phenyl]xanthene with six commercially available aromatic tetracarboxylic dianhydrides in *N,N*-dimethylacetamide (DMAc) via the poly(amic acid) precursors and subsequent thermal or chemical imidization. The intermediate poly(amic acid)s had inherent viscosities between 0.83 and 1.28 dL/g, could be cast from DMAc solutions and thermally converted into transparent, flexible, and tough poly(ether imide) films which were further characterized by X-ray and mechanical analysis. All of the poly(ether imide)s were amorphous and their films exhibited tensile strengths of 89–108 MPa, elongations at break of 7–9%, and initial moduli of 2.12–2.65 GPa. Three poly(ether imide)s derived from 4,4'-

oxydiphthalic anhydride, 4,4'-sulfonyldiphthalic anhydride, and 2,2-bis(3,4-dicarboxyphenyl)hexafluoropropane anhydride, respectively, exhibited excellent solubility in various solvents such as DMAc, *N,N*-dimethylformamide, *N*-methyl-2-pyrrolidinone, pyridine, and even in tetrahydrofuran at room temperature. The resulting poly(ether imide)s with glass transition temperatures between 286 and 335°C had initial decomposition temperatures above 500°C, 10% weight loss temperatures ranging from 551 to 575°C in nitrogen and 547 to 570°C in air, and char yields of 53–64% at 800°C in nitrogen. © 2012 Wiley Periodicals, Inc. *J Appl Polym Sci* 000: 000–000, 2012

Key words: 9,9-bis[4-(4-aminophenoxy)phenyl]xanthene; cardio poly(ether imide); bulky pendant group

INTRODUCTION

Aromatic polyimides are an important class of high-performance materials and have been widely applied in many fields such as electronics, coatings, composite materials, and membranes owing to their outstanding comprehensive properties such as excellent thermo-oxidative stability, mechanical strength, electrical properties, and high radiation and solvent resistance.^{1,2} However, most of the aromatic polyimides are insoluble in organic solvents and have high softening temperatures, which result in serious processing difficulties. To overcome these problems, several approaches such as introduction of flexible linkages,^{3–5} bulky groups,^{6–9} and molecular asymmetry or noncoplanar^{10–14} into the polymer backbone

via the synthesis of new diamines and dianhydrides have led to considerable success. Another attractive method is the incorporation of pendant loop groups (cyclic side groups) along the polymer backbone, which has been shown to impart greater solubility and enhanced rigidity as well as better mechanical and thermal properties to the resulting “cardo” polymer.¹⁵ It has been reported that the presence of cardio groups such as fluorene, phthalide, norbornane, adamantane, anthrone, etc., in the repeat unit of polyimide results in enhanced solubility as well as high-thermal properties of polymer.^{16–27} It was suggested that the intermolecular chain distance and chain rigidity of the polymer were increased owing to the bulky pendent cardio structure which eliminated the influence of the charge transfer, restricted the dense packing and free rotation of the polymer chain. Hence, the obtained polymers showed good thermal stability as well as enhanced solubility. Very recently, we have successfully synthesized soluble high-temperature aromatic polyamides by using 9,9-bis[4-(4-aminophenoxy)phenyl]xanthene (BAPX)²⁸ as a diamine monomer. In a continuation study with this diamine, the preparation and characterization of new cardio aromatic poly(ether imide)s derived from this diamine with various commercial aromatic

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Contract grant sponsor: The National Natural Science Foundation of China; contract grant numbers: 20664001, 21064003, 51163008.

Contract grant sponsor: The Research Program of Jiangxi Province Department of Education; contract grant numbers: 2007-123, GJJ08166, GJJ09138.

dianhydrides were investigated. So far, the present prepared poly(ether imide)s containing both ether and bulky pendant xanthene units in the polymer backbone have not been reported. The effect of the cardo group on solubility, crystallinity, and thermal properties of these polymers is also discussed in this article.

EXPERIMENTAL

Materials

BAPX²⁸ was prepared according to our previously reported procedures. Commercially available aromatic tetracarboxylic dianhydrides such as pyromellitic dianhydride (PMDA) (2a) (Beijing Chemical Reagent, China), 3,3',4,4'-biphenyltetracarboxylic dianhydride (BPDA) (2b) (Aldrich), 4,4'-oxydiphthalic anhydride (ODPA) (2c) (Aldrich), 3,3',4,4'-benzophenonetetracarboxylic dianhydride (BTDA) (2d) (Aldrich), diphenylsulfone-3,3',4,4'-tetracarboxylic dianhydride (DSDA) (2e) (Aldrich), and 2,2-bis(3,4-dicarboxyphenyl)hexafluoropropane anhydride (6FDA) (2f) (Aldrich) were purified by recrystallization from acetic anhydride and then dried *in vacuo* at 150°C for 10 h. *N,N*-dimethylacetamide (DMAc) and pyridine were purified by distillation under reduced pressure over calcium hydride prior to use. All other solvents were obtained from various commercial sources and used without further purification.

Characterization

NMR spectra were measured on a Bruker AVNCE 400 MHz spectrometer with DMSO-*d*₆ or CDCl₃ as solvent and tetramethylsilane as an internal standard. Fourier transform infrared (FTIR) spectra were recorded on a Perkin-Elmer SP One FTIR spectrophotometer. Elemental analysis was run on a Carlo Erba 1106 Elemental Analyzer. Differential scanning calorimetry (DSC) analysis was performed on a PerkinElmer DSC-7 instrument at a heating rate of 20°C/min in flowing nitrogen (20 cm³/min). The glass-transition temperatures were read at the middle of the transition in the heat capacity and were taken from the second heating scan after quick cooling from 400°C at a cooling rate of 200°C/min. Thermogravimetric analysis (TGA) was carried out on approximately 6–8 mg samples with a PerkinElmer Pyris 1 TGA in nitrogen or air (40 cm³/min) at a heating rate of 10°C/min. Wide-angle X-ray diffraction patterns were recorded at room temperature (ca. 25°C) on a Shimadzu XRD-6000 X-ray diffractometer (operating at 40 kV and 30 mA) with graphite-monochromatized CuK_α radiation ($\lambda = 1.5418 \text{ \AA}$); the scanning rate was 2°/min over a range of $2\theta = 5\text{--}40^\circ$. The inherent viscosities were measured at a concentration of 0.5 g/dL in DMAc, *N*-methyl-2-pyrrolidinone (NMP), or conc.

H₂SO₄ with an Ubbelohde viscometer at 30°C. The mechanical properties of the polymer films were performed on an Instron 1122 testing instrument with a 5-kg load cell at a crosshead speed of 5 mm/min on strips (0.5 cm wide, 2 cm long, and ca. 0.05 mm thick), and an average of at least four individual determinations was used.

Polymer synthesis

The synthesis of poly(ether imide) 4c was used as an example to illustrate the general synthetic route used to produce the present polymers. Diamine (BAPX) (1) (2.195 g, 4.0 mmol) was dissolved in DMAc (16 mL) in a 100-mL three-necked flask fitted with a nitrogen inlet pipet and a mechanical stirrer, to which the aromatic dianhydride ODPA (2c) (1.241 g, 4.0 mmol) was added with stirring. The mixture was stirred at room temperature in nitrogen for 24 h to yield a viscous poly(amic acid) solution. The inherent viscosity of the poly(amic acid) in DMAc was 0.83 dL/g, measured at a concentration of 0.5 g/dL at 30°C. Then, the poly(amic acid) was converted into poly(ether imide) using thermal or chemical imidization methods.

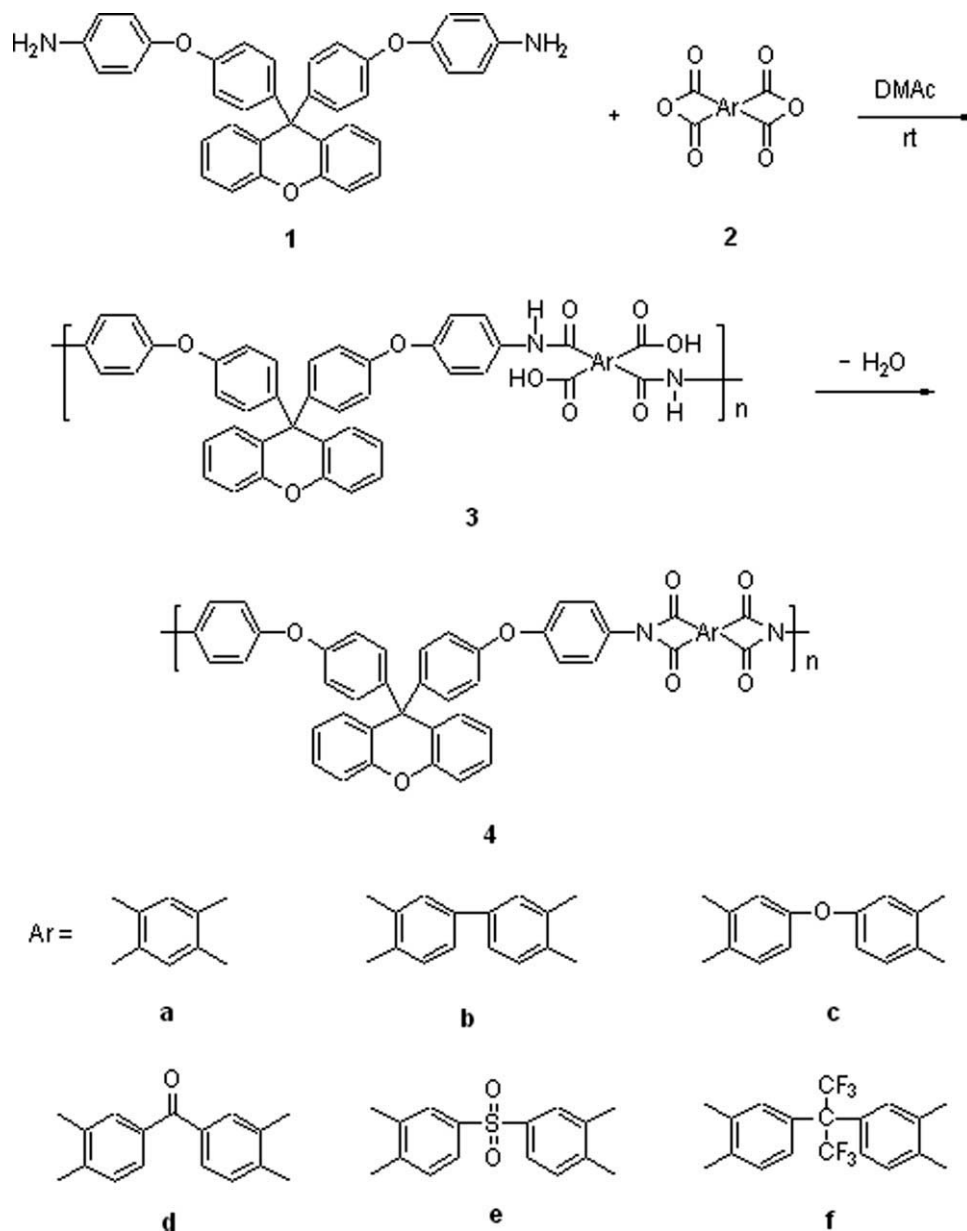
For the thermal imidization method, the poly(amic acid) solution was cast onto a clean glass plate and heated (80°C /6 h, 120°C /1 h, 150°C /1 h, 200°C /2 h, 250°C /2 h, and 300°C /2 h) under vacuum to produce a fully imidized polyimide film.

Chemical imidization was carried out by adding extra DMAc and an equimolar mixture of acetic anhydride and pyridine into the aforementioned poly(amic acid) solution, stirring at room temperature for 1 h and then heating at 80°C for 4 h. The poly(ether imide) solution was then poured into methanol and the precipitate was collected by filtration, washed thoroughly with methanol and hot water, and dried at 80°C in a vacuum to give poly(ether imide) 4c with the yield of 97%. The inherent viscosity was 0.70 dL/g, as measured at a concentration of 0.5 g/dL in NMP at 30°C. ¹H NMR (400 MHz, DMSO-*d*₆): $\delta = 8.15$ (d, $J = 8.4$ Hz, 2 H), 8.01 (d, $J = 8.4$ Hz, 2 H), 7.93 (s, 2 H), 7.45 (d, $J = 8.8$ Hz, 4 H), 7.32 (t, $J = 7.2$ Hz, 2 H), 7.21 (d, $J = 8.0$ Hz, 2 H), 7.13 (t, $J = 7.2$ Hz, 2 H), 7.08 (d, $J = 8.8$ Hz, 4 H), 6.93–6.88 (m, 10 H). IR (film): 1776 (asymmetrical C=O stretch), 1723 (symmetrical imide C=O stretch), 1600–1495 (aromatic C=C stretch), 1373 (C=N stretch), 1252 (C=O stretch), 1026, 743 cm⁻¹ (imide ring deformation). Other polymers were synthesized according to the analogous procedure.

RESULTS AND DISCUSSION

Polymer synthesis

Six new xanthene-containing poly(ether imide)s 4a–4f were prepared from BAPX (1) and commercially



Scheme 1 Synthesis of the poly(ether imide)s (**4a–4f**).

available aromatic tetracarboxylic dianhydrides including **PMDA** (**2a**), **BPDA** (**2b**), **ODPA** (**2c**), **BTDA** (**2d**), (**DSDA**) (**2e**), and **6FDA** (**2f**), via a conventional two-step procedure, as shown in Scheme 1, involving ring-opening polyaddition forming poly(amic acid) and subsequent thermal or chemical cyclodehydration. Adding the dianhydride (**2a–2f**) to the diamine **BAPX** solution at room temperature for 24 h yielded viscous poly(amic acid)s (**3a–3f**). As summarized in Table I, the inherent viscosities of the intermediate **3a–3f** were 0.83–1.28 dL/g, indicating the molecular weights of **3a–3f** were high enough to cast flexible and tough films. The poly(amic acid) films were further heated up to 300°C under vacuum to produce flexible and tough poly(ether imide) films.

Alternatively, chemical imidization of poly(amic acid)s by adding dehydrating agents, the mixture of acetic anhydride and pyridine was also effective in obtaining the corresponding poly(ether imide)s. Before adding dehydrating agent, extra-dry DMAc must be added to the poly(amic acid)s solution to prevent gelation during imidization. By the chemical procedure, the resulting poly(ether imide)s, except for **4a**, had a good solubility in NMP with inherent viscosities of 0.70–0.85 dL/g (Table II). It had been observed that the poly(ether imide) **4c** obtained from **ODPA** (**2c**) showed the lowest inherent viscosity. It might be as a result of the presence of a flexible ether linkage in the main chains, which enhances the flexibility of polymer, resulting in the comparatively low viscosity.

TABLE I
Inherent Viscosity of Poly(amic acid)s and Poly(ether imide)s and Elemental Analysis of the Poly(ether imide)s

Poly(amic acid)		Poly(ether imide) ^a			Elemental analysis (%) of poly(ether imide) ^b			
Code	η_{inh} (dL/g) ^c	Code	η_{inh} (dL/g) ^d	Yield (%)	Formula (molecular weight)	C	H	N
3a	1.28	4a	0.78 ^e	98	(C ₄₇ H ₂₆ N ₂ O ₇) _n (730.73) _n	77.12 (77.25)	3.52 (3.59)	3.99 (3.83)
3b	1.06	4b	0.85	97	(C ₅₃ H ₃₀ N ₂ O ₇) _n (806.83) _n	78.78 (78.90)	3.67 (3.75)	3.40 (3.47)
3c	0.83	4c	0.70	97	(C ₅₃ H ₃₀ N ₂ O ₈) _n (822.83) _n	77.13 (77.37)	3.57 (3.68)	3.28 (3.40)
3d	0.91	4d	0.75	98	(C ₅₄ H ₃₀ N ₂ O ₈) _n (834.84) _n	77.52 (77.69)	3.48 (3.62)	3.24 (3.36)
3e	0.95	4e	0.81	97	(C ₅₃ H ₃₀ N ₂ O ₉ S) _n (870.89) _n	72.82 (73.09)	3.35 (3.47)	3.06 (3.22)
3f	1.02	4f	0.74	96	(C ₅₆ H ₃₀ F ₆ N ₂ O ₇) _n (956.85) _n	70.41 (70.29)	3.02 (3.16)	2.99 (2.93)

^a Obtained by chemical imidization.

^b Theoretical percentages are in parentheses.

^c Determined at a concentration of 0.5 g/dL in DMAc at 30°C.

^d Determined at a concentration of 0.5 g/dL in NMP at 30°C.

^e Determined at a concentration of 0.5 g/dL in conc. H₂SO₄ at 30°C.

The formation of poly(ether imide)s was confirmed by FTIR, HNMR, and elemental analysis. The elemental analysis data of the thermally cured series are summarized in Table I, which generally agreed well with the calculated values for the proposed structures. Figure 1 shows the typical FTIR spectra for thin-film sample of poly(amic acid) **3c** and fully cyclized poly(ether imide) **4c**. The complete conversion of *o*-carboxyl amide into the imide ring was supported by the disappearance of amic acid bands at 1655–1700 and 2500–3500 cm⁻¹, together with the appearance of characteristic imide absorption bands at 1776 (asymmetrical C=O stretch), 1723 (symmetrical imide C=O stretch), 1373 (C=N stretch), 1171, 1026, 743 cm⁻¹ (imide ring deformation). Additionally, the structure of poly(ether imide) **4c** was further verified by its ¹H NMR spectrum (Fig. 2), in which H¹ appears as a singlet at 7.92 ppm and H² and H³ as AB doublets at 7.97 and 8.13 ppm, respectively, owing to their coupling are observed clearly, whereas the overlapping signals (H⁴, H⁵, and H¹) ascribed to the aromatic protons of the diamine

monomer were difficult to distinguish from one by one.

Thermal property

The thermal properties of the poly(ether imide)s obtained by thermal imidization were determined by using DSC and TGA techniques, and the results are summarized in Table II. Typical DSC curve for poly(ether imide) **4c** in nitrogen atmosphere is shown in Figure 3, in which no melting transition temperature was detected by DSC thermogram, supporting the generally amorphous character of the polymer. As summarized in Table II, the *T_g* values of the poly(ether imide)s were in the range of 286–335°C, which generally correlated with the rigidity of polymer backbone. As a result, the poly(ether imide) **4a** derived from BAPX and PMDA (**2a**) exhibited the highest *T_g* value (335°C) probably owing to its most rigid structure as compared with other poly(ether imide)s, whereas the poly(ether imide) **4c** showed the lowest *T_g* of 286°C for its flexible

TABLE II
Thermal Behavior Data of the Poly(ether imide)s (Thermal Imidization)

Polymer	<i>T_g</i> ^a (°C)	<i>T_{d0}</i> ^b (°C)	<i>T_{d0}</i> ^c (°C)	<i>T_{d10}</i> ^d (°C)	<i>T_{d10}</i> ^e (°C)	<i>R_w</i> ^f (%)
4a	335	513	511	575	567	64
4b	315	511	509	573	570	61
4c	286	510	510	570	567	60
4d	290	506	502	565	560	58
4e	311	505	501	551	547	53
4f	304	512	510	567	559	57
	296 ^g	509 ^g	508 ^g	557 ^g	544 ^g	55 ^g

^a Glass transition temperature base on the second heating trace of DSC measurements.

^b Initial decomposition temperature measured by TGA in N₂.

^c Initial decomposition temperature measured by TGA in air.

^d 10% Weight loss temperature measured by TGA in N₂.

^e 10% Weight loss temperature measured by TGA in air.

^f Residual weight % at 800°C in N₂.

^g Poly(ether imide) was prepared by chemical imidization.

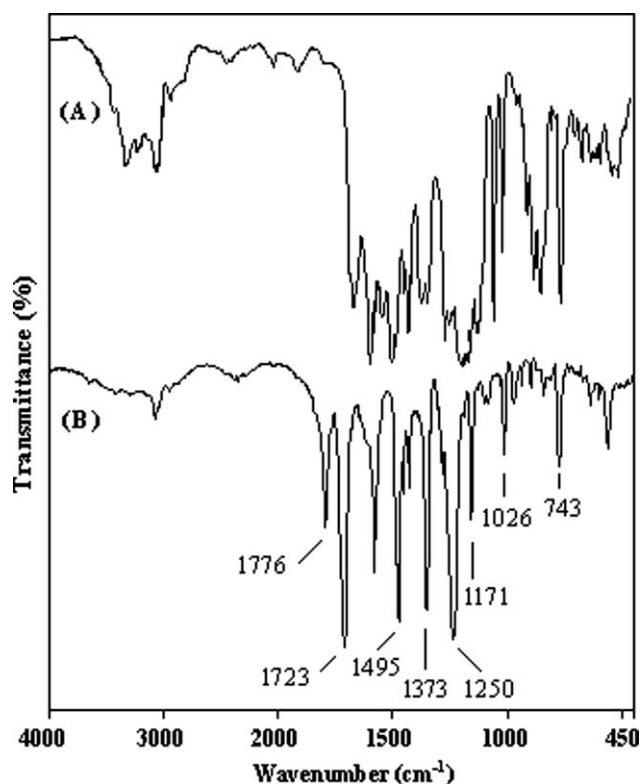


Figure 1 FTIR spectra of (A) poly(amic acid) **3c** and (B) poly(ether imide) **4c**.

polymer chain. However, the poly(ether imide) **4f** obtained from **6-FDA** had a higher T_g value than that of poly(ether imide) **4d** (derived from BTDA), which might be attributed to the hexafluoroisopropylidene group in the backbone which inhibited the free rotation of the polymer chains.

The thermal stability of these poly(ether imide)s was evaluated by TGA. They were stable up to 500°C and their 10% weight loss temperatures were in the range of 551–575 and 547–570°C in nitrogen and air, respectively. Also, the char yields at 800°C in nitrogen for all the polymers were in the range of

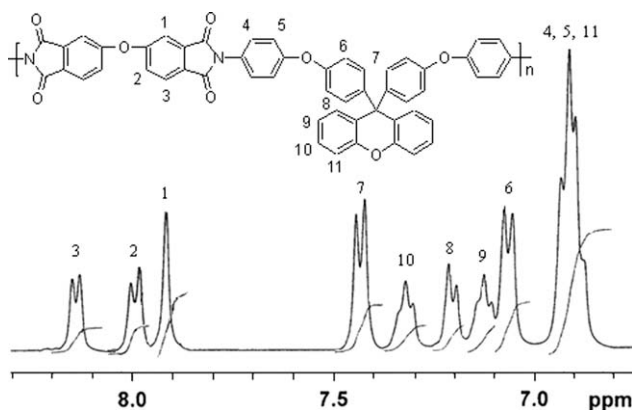


Figure 2 ^1H NMR spectrum of the poly(ether imide) **4c** in $\text{DMSO}-d_6$.

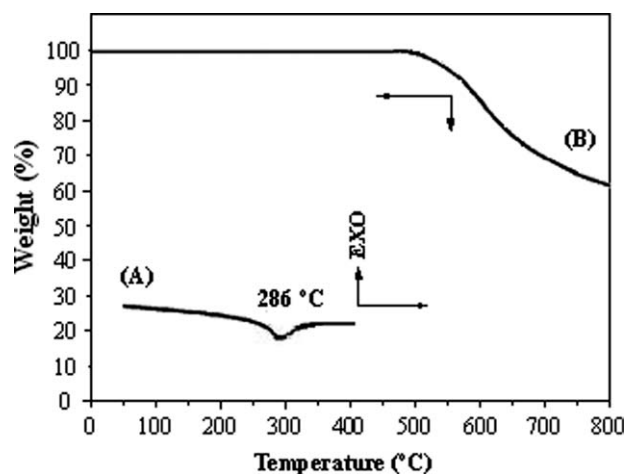


Figure 3 DSC (A) and TGA (B) curves for the poly(ether imide) **4c** in nitrogen.

53–64%. Clearly, the data from thermal analysis showed that the resulting poly(ether imide)s had fairly high thermal stability.

It should be noted that different imidization methods could produce polymers with a slight difference in T_g values. For instance, poly(ether imide) **4f** obtained from **6FDA** showed two T_g values depending on its imidization methods: 304°C (thermal imidization) and 296°C (chemical imidization). This could be explained by the difference in polymer packaging and aggregation in the solid state and partial crosslinking in polymer chains. The hydroxy group of the carboxylic acid and the amino group of amide in the poly(amic acid) (PPA) ether form aromatic imide by further dehydration or form aromatic anhydride and amine groups again by a nucleophilic substitution reaction between each other. The aromatic amine formed, however, could react with the electrophilic carbonyl group presented in PAA chain during the heating process to form an imine structure which results in some crosslinkages between polyimide chains.²⁹ Besides, the similar results of their thermal stabilities from different imidization methods were also observed.

Solubility

The solubility of all poly(ether imide)s obtained by thermal and chemical imidization was tested qualitatively in various solvents, and the results are summarized in Table III. Poly(ether imide)s **4a**, **4b**, and **4d** were almost insoluble in organic solvents, but dissolved in concentrated sulfuric acid. Poly(ether imide) **4c**, **4e**, and **4f**, containing ether, sulfone, and hexafluoroisopropylidene linkages, respectively, exhibit excellent solubility in a variety of solvents such as DMAc, NMP, *N,N*-dimethylformamide, pyridine, and even in tetrahydrofuran at room temperature. Obviously, the better solubility of polymers **4c**, **4e**, and **4f** as compared with the other polymers (**4a**, **4b**,

TABLE III
Solubility of the Poly(ether imide)s^a

Polymer ^{b c}	Solvent ^c								
	DMAc	DMF	NMP	DMSO	Py	<i>m</i> -Cresol	THF	CHCl ₃	H ₂ SO ₄
4a	- (-)	- (-)	- (-)	- (-)	- (-)	- (-)	- (-)	- (-)	+ (+)
4b	+ (-)	+ (-)	+ (+)	- (-)	- (-)	+ (-)	- (-)	- (-)	+ (+)
4c	+ (+)	+ (+)	+ (+)	+ (+)	+ (+)	+ (+)	+ (+)	+ (+)	+ (+)
4d	+ (-)	+ (-)	+ (+)	- (-)	+ (-)	- (-)	- (-)	- (-)	+ (+)
4e	+ (+)	+ (+)	+ (+)	+ (+)	+ (+)	+ (+)	+ (-)	- (-)	+ (+)
4f	+ (+)	+ (+)	+ (+)	+ (+)	+ (+)	+ (+)	+ (+)	+ (+)	+ (+)

^a Qualitative solubility was measured with as 100 mg of polymer in 1 mL of solvent. The symbols represent the following: +, soluble at room temperature; +-, partially soluble at room temperature; -, insoluble even on heating.

^b Data in parentheses are obtained from the thermal cyclization polyimides.

^c NMP, *N*-methyl-2-pyrrolidone; DMAc, *N,N*-dimethylacetamide; DMF, *N,N*-dimethylformamide; DMSO, dimethyl sulfide; Py, pyridine; THF, tetrahydrofuran.

and 4d) is governed possibly by the structural modification through the link groups such as ether, sulfonyl, and hexafluoroisopropylidene groups in the polymer backbone.²

In addition, as summarized in Table III, the most of poly(ether imide)s obtained by chemical imidization exhibited good solubilities, comparing with those obtained by thermal imidization. The difference in solubility of between the poly(ether imide)s from thermal and chemical imidization might be mainly attribute to the formation of intermolecular links of poly (amic acid)s during the thermal cyclodehydration method.²⁹

Crystallinity and tensile property

Figure 4 shows the wide-angle X-ray diffraction curves of the poly(ether imide) films obtained by

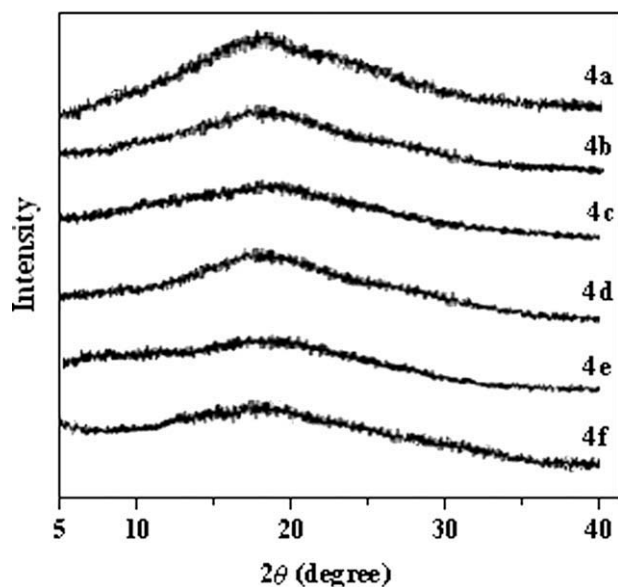


Figure 4 WAXD curves of the poly(ether imide)s (thermal imidization).

thermal imidization. As expected, all the samples displayed fairly amorphous patterns in the regions of $2\theta = 5$ and 40° , which indicated that the polymers were amorphous. This observation is reasonable because the presence of the flexible ether linkages and bulky xanthene cardo groups, which hindered chain packing and reduced intramolecular interactions between the polymer chains, causing a decrease in crystallinity.

The tensile properties of the poly(ether imide) films prepared by thermal imidization are summarized in Table IV. The films exhibited tensile strengths in the range of 89–108 MPa, elongations at break in the range of 7–9% and initial moduli in the range of 2.12–2.65 GPa, indicating they are strong and tough polymeric material. As summarized in Table IV, the flexible structural feature could increase the segmental motion, and hence the tensile strength and modulus of poly(ether imide) films derived from the dianhydrides containing flexible groups such as ODPA (2c) and DSDA (2e) were lower than those of poly(ether imide) films derived from the dianhydride without any flexible groups like as BPDA (2b). However, the elongation at break of poly(ether imide) films derived from ODPA or DSDA were higher than those of poly(ether imide) films derived from BPDA.

TABLE IV
Mechanical Properties of the Poly(ether imide)s (Thermal Imidization)

Polymer	Tensile strength (MPa)	Elongation at break (%)	Initial modulus (GPa)
4a	98	8	2.47
4b	108	7	2.65
4c	94	9	2.27
4d	86	8	2.48
4e	89	9	2.12
4f	102	9	2.34

CONCLUSIONS

In this study, a series of new cardo poly(ether imide)s-containing xanthene derived from BAPX with six different aromatic tetracarboxylic dianhydrides via the poly(amic acid) precursors and thermal or chemical imidization exhibited amorphous nature, good thermal stability, and good mechanical properties. The investigation of the properties of the polymers revealed that the introduction of bulky xanthene pendant groups into the polymer backbone resulted in polyimides with enhanced solubility, reduced crystallinity, as well as satisfactory thermal and mechanical properties.

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