

# Synthesis and Structure-Property Study of Polyarylates Derived from Bisphenols with Different Connector Groups

Sheng-Huei Hsiao\* and Hsien-Wei Chiang

Department of Chemical Engineering, Tatung University, 40 Chungshan North Road, 3rd Section, 10451 Taipei, Taiwan, Republic of China

(\*Author for correspondence; Tel.: +886-2-25925252 ext. 2977; Fax: +886-2-25861939; E-mail: shhsiao@ttu.edu.tw)

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## Abstract

Several polyarylates were synthesized from the interfacial or high-temperature solution polycondensation reactions of four bisphenols including 2,2-bis(4-hydroxyphenyl)propane (BPA), 2,2-bis(4-hydroxyphenyl)hexafluoropropane (BPAF), 1,1-bis(4-hydroxyphenyl)-1-phenylethane (BPAP), and 1,1-bis(4-hydroxyphenyl)-1-phenyl-2,2,2-trifluoroethane (BPAPF) with various aromatic diacyl chlorides. The basic properties such as inherent viscosity, film-forming capability, crystallinity, and mechanical and thermal properties and the “structure-property” relationships of these polyarylates were investigated. The polyarylates derived from asymmetric BPAP and BPAPF had lower crystallinity and higher solubility and glass-transition temperature ( $T_g$ ) than the corresponding counterparts based on symmetric BPA and BPAPF. The fluorinated polyarylates revealed higher  $T_g$  values and decomposition temperatures than the corresponding nonfluorinated polyarylates.

## Introduction

Polyarylates are generally defined as highly aromatic polyesters consisting of aromatic diols and aromatic dicarboxylic acids [1, 2]. Numerous efforts have been made to clarify the structure-property relationships in polyarylates [3], and some polyarylates are commercially available as high performance engineering plastics [4]. The primary commercial polyarylates available today are the copolyesters based on 2,2-bis(4-hydroxyphenyl)propane (bisphenol A; BPA) and a mixture of isophthalic and terephthalic acids, that have a glass-transition temperature ( $T_g$ ) around 190 °C. There is still a need for the polyarylates having high  $T_g$ 's while retaining good solubility in organic solvents, thereby keeping the feasible processing. One of the approaches to obtain the polymer of this type is by the introduction of a bulky pendent group along the polymer backbone [5, 6]. In our previous publications we demonstrated that triphenylethane and triphenyltrifluoroethane units-containing aromatic polyamides and polyimides exhibited moderately high  $T_g$ 's and enhanced thermal stability and solubility [7–9]. Therefore, the triphenylethane and triphenyltrifluoroethane units might be a promising segment for the preparation of polyarylates having high  $T_g$  and good solubility. This article deals with the preparation and basic characterization of several polyarylates from the polycondensation reactions of bisphenols BPA, BPAF, BPAP, and BPAPF with various aromatic diacid chlorides. The effects of different bisphenol linking groups and the structures of diacyl chlorides on the properties of polyarylates are also discussed.

## Experimental

### Materials

2,2-Bis(4-hydroxyphenyl)propane (bisphenol of acetone; BPA) (from Wako), 2,2-bis(4-hydroxyphenyl)hexafluoropropane (BPAF) (from Chriskev), and 1,1-bis(4-hydroxyphenyl)-1-phenylethane (biphenol of acetophenone; BPAP) (from Honshu Chemical Co. Ltd.) were used as received. 1,1-Bis(4-hydroxyphenyl)-1-phenyl-2,2,2-trifluoroethane (BPAPF) was prepared by the acid-catalyzed condensation from  $\alpha,\alpha,\alpha$ -trifluoroacetophenone and phenol according to a reported method [10, 11]. Terephthaloyl chloride (TPC) and isophthaloyl chloride (IPC) were purified by vacuum distillation. The other diacyl chloride monomers including 4,4'-biphenyldicarbonyl chloride (BPDC) (mp = 186 °C), 1,4-naphthalenedicarbonyl chloride (NDC) (mp = 99 °C), 4,4'-oxydibenzoyl chloride (ODBC) (mp = 90 °C), and 4,4'-sulfonyldibenzoyl chloride (SDBC) (mp = 162 °C) were prepared by the chlorination of the corresponding commercially available dicarboxylic acids with thionyl chloride ( $\text{SOCl}_2$ ) in the presence of a trace amount of *N,N*-dimethylformamide. Benzyltriethylammonium chloride (BTEAC) (from TCI) used as the phase transfer catalyst in the interfacial polymerizations was used as received.

### Polymer Synthesis

#### Interfacial Polycondensation

A typical procedure for the synthesis of polyarylate **O-3F** is given below. A solution of 4,4'-oxydibenzoyl chloride

(ODBC) (0.5843 g; 2 mmol) in dichloromethane (6 mL) was added all at once to a solution of bisphenol BPAPF (0.6886 g; 2 mmol) and BTEAC 30 mg in 1 M aqueous sodium hydroxide (5 mL). The reaction mixture was stirred vigorously at room temperature for 1 h. The polymer mixture was poured into boiling water (about 300 mL) to evaporate dichloromethane. The precipitated polymer was collected by filtration, washed with hot water thoroughly, and dried. The inherent viscosity of the resulting polyarylate coded as **O-3F** was 1.37 dL/g, measured at a concentration of 0.5 g/dL in 1,1,2,2-tetrachloroethane (TCE)/phenol (40/60 by weight) at 30 °C. The other polyarylates based on diacyl chlorides TPC, IPC, NDC, ODBC, and SDBC were prepared by a similar procedure described as above.

#### High-temperature Solution Polycondensation

Because diacid chloride BPDC was insoluble in dichloromethane, the polycondensation reactions of BPDC and all bisphenols were carried out in *o*-dichlorobenzene at an elevated temperature. As a typical example, the synthetic procedure for polyarylate **B-3F** is described as follows. A mixture of 0.5582 g (2 mmol) of BPDC and 0.6886 g (2 mmol) of BPAPF in 10 mL of *o*-dichlorobenzene was heated to 200 °C and stirred at that temperature for 3 h. After cooling, the viscous solution was poured into 200 mL of methanol. The precipitated white fibrous polymer (**B-3F**) was washed thoroughly by methanol, collected by filtration, and dried. The inherent viscosity of polyarylate **B-3F** was 0.47 dL/g, measured at a concentration of 0.5 g/dL in TCE/phenol (40/60 by weight) at 30 °C.

#### Preparation of the Polyarylate Films

A polymer solution of approximately 10 wt% was made by the dissolution of about 1.0 g of polyarylate in 10 mL of hot TCE. The clear solution was poured into a 11-cm-diameter glass culture dish, which was placed in a 90 °C oven overnight for slow release of the solvent. Then the obtained semidried polymer film was stripped from the glass substrate and further dried in vacuum at 160 °C for 6 h. The obtained films were about 0.08 mm thick and used for X-ray diffraction studies, tensile tests, solubility tests, and thermal analyses.

#### Measurements

Elemental analysis was made on a PerkinElmer 2400 CHN analyzer. Infrared (IR) spectra were recorded on a Horiba FT-720 Fourier transform infrared (FTIR) spectrometer. <sup>1</sup>H and <sup>13</sup>C NMR spectra were measured on a JEOL EX 400 spectrometer. Wide-angle X-ray diffraction (WAXD) measurements were performed at room temperature (ca. 25 °C) on a Siemens Kristalloflex D5000 X-ray diffractometer (operating at 40 kV and 20 mA) with Ni-filtered Cu K $\alpha$  radiation ( $\lambda = 1.5418 \text{ \AA}$ ). The scanning rate was 2°/min over a range of  $2\theta = 5\text{--}45^\circ$ . The inherent viscosities of the polyarylates were determined with a Cannon-Fenske viscometer at 30 °C. Differential scanning calorimetry (DSC) analyses were performed on a PerkinElmer Pyris 1 DSC at a heating rate of 20 °C/min under nitrogen.  $T_g$ 's 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. Thermomechanical analysis (TMA) was conducted with a PerkinElmer TMA 7 instrument. The TMA experiments were conducted from 40 °C to 300 °C at a scan rate of 10 °C/min using a penetration probe 1.0 mm in diameter under an applied constant load of 10 mN. Softening temperatures ( $T_s$ 's) were taken as the onset temperature of probe displacement on the TMA traces. Thermogravimetric analysis (TGA) was conducted with a PerkinElmer Pyris 1 TGA. Experiments were carried out on approximately 3–5 mg of samples in flowing nitrogen or air (flow rate 40 cm<sup>3</sup>/min) at a heating rate of 20 °C/min. An Instron universal tester (model 4400R) with a load cell of 5 kg was used to study the stress-strain behavior of the polyarylate film samples. A gauge length of 2 cm and a crosshead speed of 5 mm/min were used for this study. Measurements were performed at room temperature with film specimens (0.5 cm wide, 6 cm long, and about 0.08 mm thick), and an average of at least five individual determinations were used.

## Results and Discussion

### Polymer Synthesis

The structures and codes of diacyl chlorides and bisphenols employed in the synthesis of polyarylates are shown in Figures 1 and 2, respectively. Because the diacyl chloride BPDC

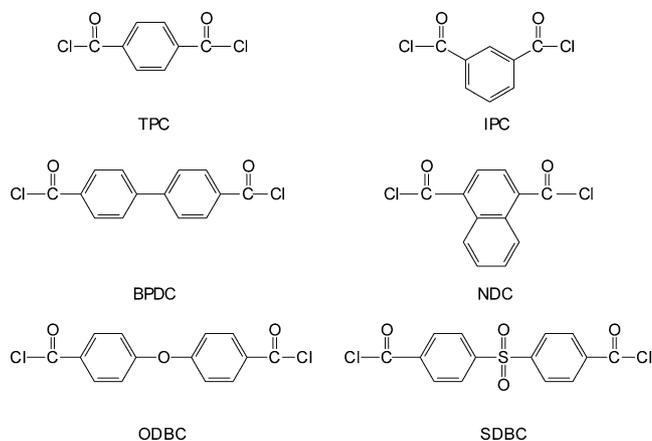


Figure 1. Structure and codes of diacyl chloride monomers.

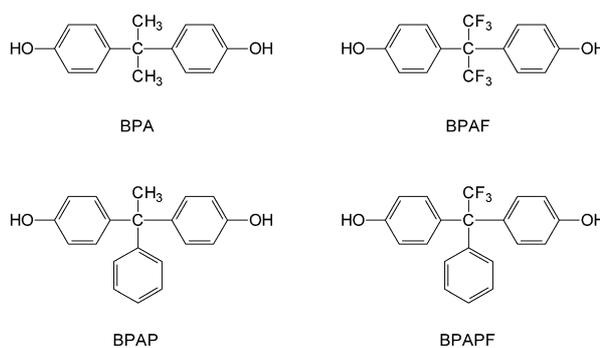


Figure 2. Structure and codes of bisphenol monomers.

is insoluble in dichloromethane, its derived polyarylates were prepared by a high-temperature solution polymerization reaction in *o*-dichlorobenzene. The other polyarylates were prepared by the phase-transfer catalyzed (or interfacial) polycondensation of diacyl chlorides with bisphenols, a well-established technique [12, 13]. The results of polycondensation are summarized in Table 1. Polymer precipitation occurred during the polycondensation reactions of TPC and BPDC with BPA and BPAF due to insolubility of the resulting polyarylates. Insolubility of polymers from these monomer combinations, such as **T-6H**, **T-6F**, **B-6H** and **B-6F**, may be caused by their high level of crystallinity, as evidenced by WAXD and DSC analysis discussed subsequently. These semicrystalline polyarylates could not be dissolved in suitable organic solvents for film casting. As indicated by GPC measurements, the other organosoluble

polyarylates had moderately high molecular weights, with Mw of 35,800–72,400 relative to polystyrene standards. Except for polyarylate **I-6H** (from IPC and BPA), all the organosoluble polyarylates could afford flexible and tough films. The brittle nature of the cast film of **I-6H** also can be attributed to its semicrystalline structure, as shown by WAXD and DSC.

The structural feature of the polyarylates was confirmed by IR spectroscopy. The IR spectra of some BPAPF polyarylates are shown in Figure 3. The characteristic absorptions of ester group appear around 1750 cm<sup>-1</sup> (C=O stretching) and in the region of 1200–1300 cm<sup>-1</sup> (C–O stretching). The strong absorptions at about 1150 cm<sup>-1</sup> are due to the C–F stretching.

### Polymer Properties

#### Crystallinity

The crystallinity of the obtained polyarylates was evaluated by wide-angle X-ray diffraction (WAXD) measurements. The diffraction patterns of the polyarylates on the basis of BPA and BPAF are presented in Figures 4 and 5, respectively. As can be seen from these WAXD patterns, the polymers derived from rigid diacyl chlorides and more symmetric bisphenols such as **T-6H**, **B-6H**, **T-6F**, and **B-6F** showed some stronger reflection peaks, indicative of moderate to high degree of crystallinity. The opaque film obtained from **I-6H** also revealed a crystalline signal around  $2\theta = 20^\circ$ . All the polyarylates derived from diacyl chlorides NDC, ODBC, and SDBC exhibited almost completely amorphous patterns. The effect of bisphenol connecting groups on the crystallinity of polyarylates also can be understood from Figure 6. Polyterephthalates **T-6F** and **T-6H** revealed some stronger diffraction signals, indicating a semicrystalline nature. However, polyarylates **T-3F** and **T-3H** showed almost completely amorphous diffraction patterns. The other polyarylates derived from bisphenols BPAP and BPAPF also showed essentially amorphous patterns as those of **T-3F** and **T-3H**. Apparently, the bulky, non-symmetric triphenyltrifluoroethane and triphenylethane cores interfere with the close chain packing, thus leading to amorphous polyarylates.

#### Organo-Solubility

The organo-solubility of the polyarylates was determined qualitatively, and the results are listed in Table 2. Most of the polyarylates derived from bisphenols BPAP and BPAPF were readily soluble at room temperature in polar solvents, such as NMP, DMSO, THF, and several chlorinated solvents. Their high solubility could be attributed to in part the introduction of the bulky, packing-disruptive triphenylethane and triphenyltrifluoroethane units. Polymers **T-6H** and **T-6F** revealed a relatively poor solubility because of their crystalline structures.

#### Tensile Properties

Most polyarylates could afford good-quality and flexible films, and their tensile properties are summarized in Table 3. The polymer films had tensile strengths of 60–75 MPa,

Table 1. Synthesis and film quality of polyarylates

Diacid chloride	Bisphenol	Polymer			
		Code	$\eta_{inh}^a$ (dL/g)	Remarks <sup>b</sup>	Film quality <sup>c</sup>
TPC	BPA	<b>T-6H</b>	0.26	P	— <sup>d</sup>
	BPAF	<b>T-6F</b>	0.35	P	—
	BPAP	<b>T-3H</b>	1.16	S	Flexible
	BPAPF	<b>T-3F</b>	1.37	S	Flexible
IPC	BPA	<b>I-6H</b>	0.84	S	Brittle, opaque
	BPAF	<b>I-6F</b>	0.98	S	Flexible
	BPAP	<b>I-3H</b>	1.48	S	Flexible
	BPAPF	<b>I-3F</b>	1.20	S	Flexible
BPDC	BPA	<b>B-6H</b>	0.29	P	—
	BPAF	<b>B-6F</b>	0.44	P	—
	BPAP	<b>B-3H</b>	0.49	S	Flexible
	BPAPF	<b>B-3F</b>	0.47	S	Flexible
NDC	BPA	<b>N-6H</b>	0.89	S	Flexible
	BPAF	<b>N-6F</b>	0.71	S	Flexible
	BPAP	<b>N-3H</b>	0.77	S	Flexible
	BPAPF	<b>N-3F</b>	1.19	S	Flexible
ODBC	BPA	<b>O-6H</b>	0.93	S	Flexible
	BPAF	<b>O-6F</b>	0.55	S	Flexible
	BPAP	<b>O-3H</b>	1.16	S	Flexible
	BPAPF	<b>O-3F</b>	1.33	S	Flexible
SDBC	BPA	<b>S-6H</b>	1.21	S	Flexible
	BPAF	<b>S-6F</b>	1.10	S	Flexible
	BPAP	<b>S-3H</b>	1.33	S	Flexible
	BPAPF	<b>S-3F</b>	1.10	S	Flexible

<sup>a</sup>Measured at a concentration of 0.5 g/dL in 1,1,2,2-tetrachloroethane/phenol (40 : 60 by weight) at 30 °C.

<sup>b</sup>Appearance of the polymerization: P, polymer precipitation occurred during the reaction; S, homogeneous organic solution (well-defined two phases).

<sup>c</sup>Films were cast by slow evaporation of the polymer solutions in 1,1,2,2-tetrachloroethane.

<sup>d</sup>Insoluble.

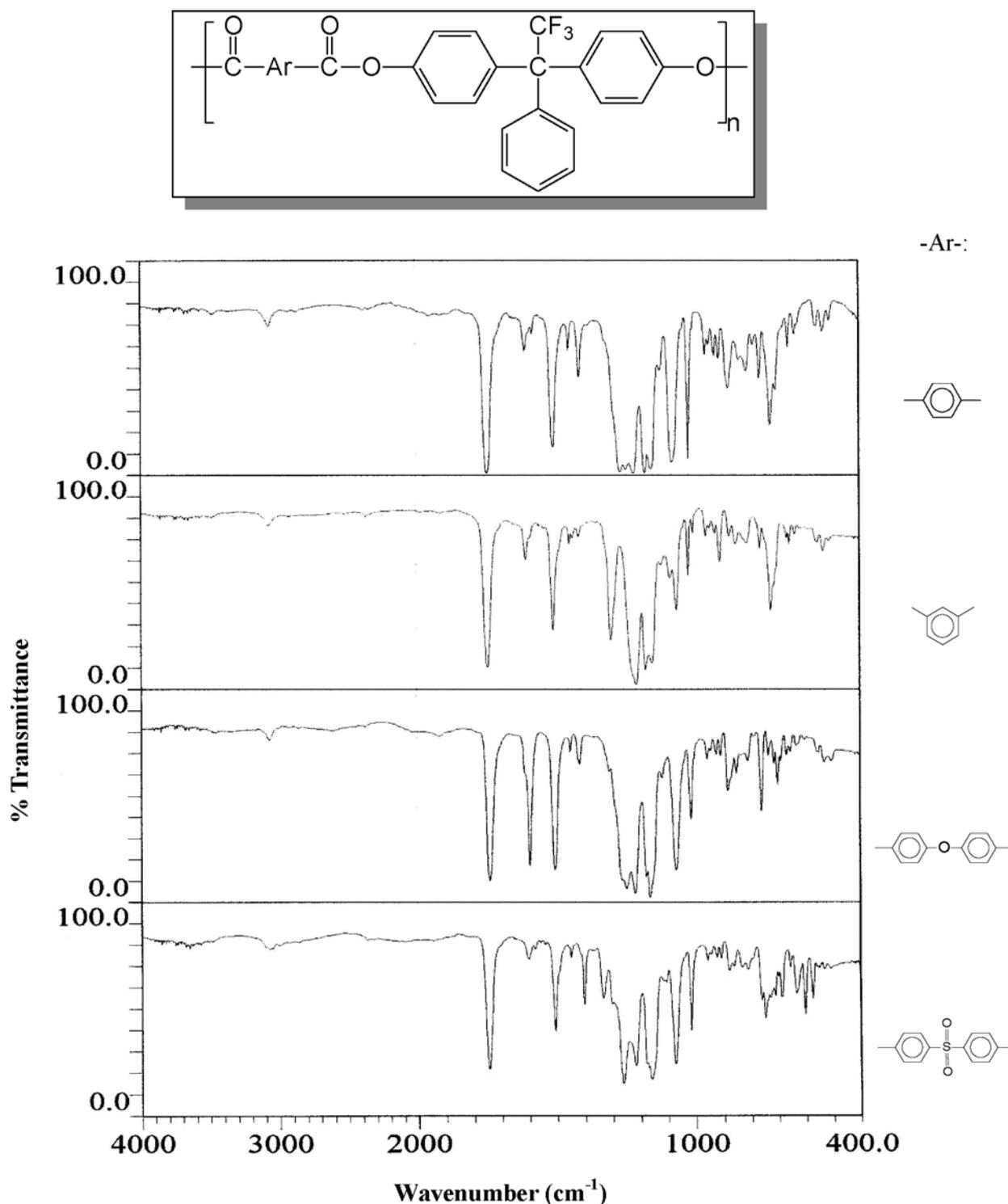


Figure 3. FTIR spectra of some BPAPF polyarylates.

elongations to break of 7–20%, and tensile moduli of 1.36–1.82 GPa. Most polymers behaved as ductile materials with good tensile strengths and moderate elongations to break.

#### Thermal Properties

DSC, TMA, and TGA were used to evaluate thermal properties of all polymers. The thermal behavior data of the polyarylates are summarized in Table 4. The semicrystalline polyarylates **T-6F**, **I-6H**, **T-6H**, and **B-6F** exhibited an obvi-

ous melting endotherm in the first DSC heating traces around 314, 299, 356 and 368  $^{\circ}\text{C}$ , respectively. Although polyarylate **B-6H** revealed a crystalline WAXD pattern, the well-defined endothermal peak was not observed on DSC curve before 400  $^{\circ}\text{C}$ , possibly because of a high melting point. All the other polyarylates did not show melting endotherms up to the decomposition temperatures on the DSC thermograms. This result also supports the amorphous nature of these polyarylates. The  $T_g$  values of these polyarylates were

Table 2. Solubility behavior of polyarylates<sup>a</sup>

Polymer	Solvent <sup>b</sup>						
	NMP	DMSO	THF	Chloroform	TCE	<i>o</i> -CP	<i>o</i> -DCB
T-6H	-	-	-	-	-	-	-
T-6F	-	-	-	-	-	-	-
T-3H	+	+	+	+	+	+	+
T-3F	+	+	+	+	+	+	+
I-6H	-	-	-	-	+	+	+
I-6F	+	+	+	+	+	+	+
I-3H	+	-	+	+	+	+	+
I-3F	+	-	+	+	+	+	+
B-6H	+	-	-	-	+	+	-
B-6F	+	-	+	+	+	+h	-
B-3H	+	+	+	+	+	+	+
B-3F	+	+	+	+	+	+	+
N-6H	+	-	-	-	+	+	-
N-6F	+	+h	-	-	+	+	+h
N-3H	+	+	+	+	+	+	+
N-3F	+	+	+	+	+	+	+
O-6H	+	-	+	+	+	+	+
O-6F	+	-	+	+	+	+	+
O-3H	+	-	+	+	+	+	+
O-3F	+	+h	+	+	+	+	+
S-6H	+	-	+	-	+	+	-
S-6F	+	-	+	+	+	+	-
S-3H	+	+	+	+	+	+	+
S-3F	+	+	+	+	+	+	+

<sup>a</sup>Qualitative solubility tested with 10 mg of sample in 1 mL of solvent. +: soluble at room temperature; +h: soluble on heating at 100 °C; -: insoluble even on heating.

<sup>b</sup>NMP: *N*-methyl-2-pyrrolidone; DMSO: dimethyl sulfoxide; THF: tetrahydrofuran; TCE: 1,1,2,2-tetrachloroethane; *o*-CP: *o*-chlorophenol; *o*-DCB: *o*-dichlorobenzene.

Table 3. Tensile properties of polyarylates films<sup>a</sup>

Polymer	Tensile strength (MPa)	Elongation to break (%)	Initial modulus (GPa)
T-3H	71	20	1.68
T-3F	68	14	1.61
I-6F	65	7	1.47
I-3H	64	8	1.36
I-3F	71	10	1.52
B-3H	65	12	1.53
B-3F	67	18	1.44
N-6H	65	14	1.50
N-6F	59	8	1.50
N-3H	65	9	1.45
N-3F	66	8	1.66
O-6H	67	10	1.57
O-6F	64	7	1.60
O-3H	67	12	1.39
O-3F	75	14	1.82
S-6H	57	12	1.47
S-6F	61	10	1.48
S-3H	60	12	1.44
S-3F	66	11	1.61

<sup>a</sup>Films were cast from slow evaporation of polymer solutions in 1,1,2,2-tetrachloroethane. The cast films were dried under vacuum at 150 °C for 6 h prior to the tensile test.

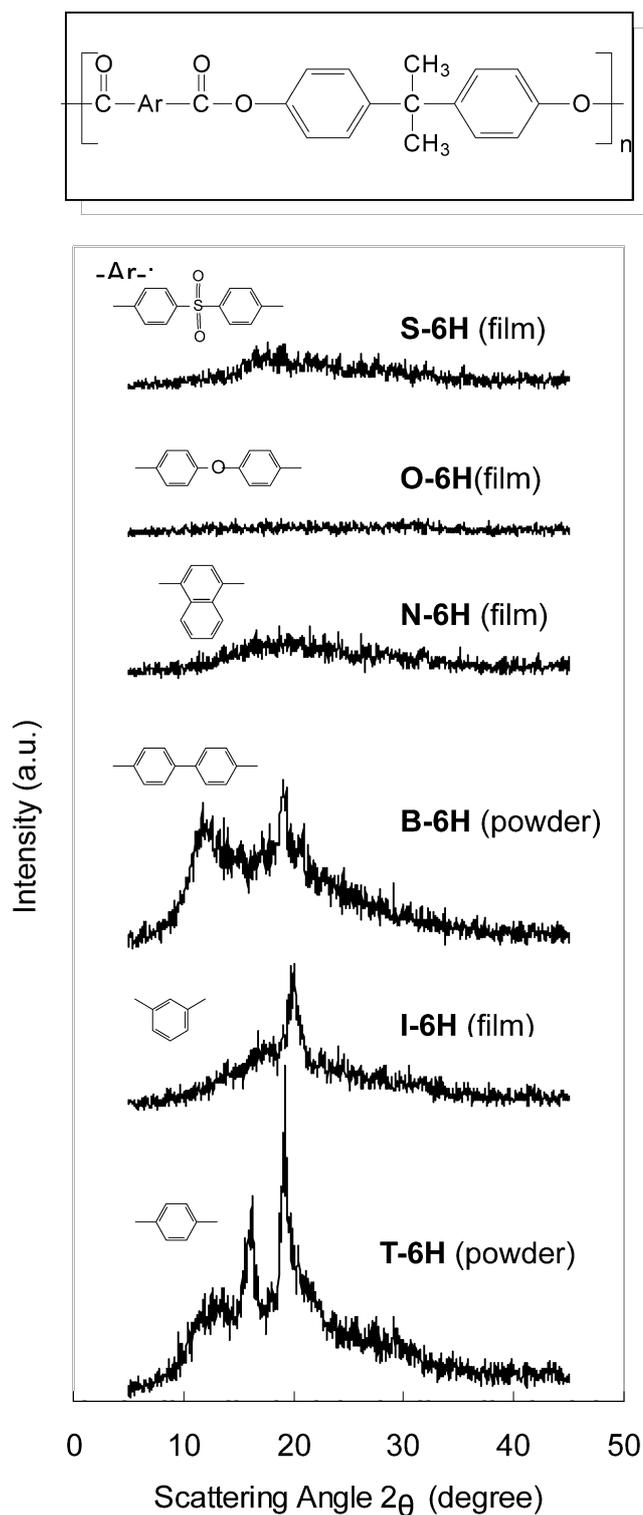


Figure 4. WAXD patterns of BPA-derived polyarylates.

recorded in the range of 166–271 °C. The  $T_s$  values of the polyarylate film samples were also determined with TMA by the penetration method. A typical TMA thermogram of polyarylate T-3F is illustrated in Figure 7. The  $T_s$  values of these polyarylates were between 162–288 °C. As expected, the IPC and ODBC polyarylates showed relatively lower  $T_g$  and  $T_s$  values because of the more flexible backbones. Due to an increased hindrance effect caused by the bulky

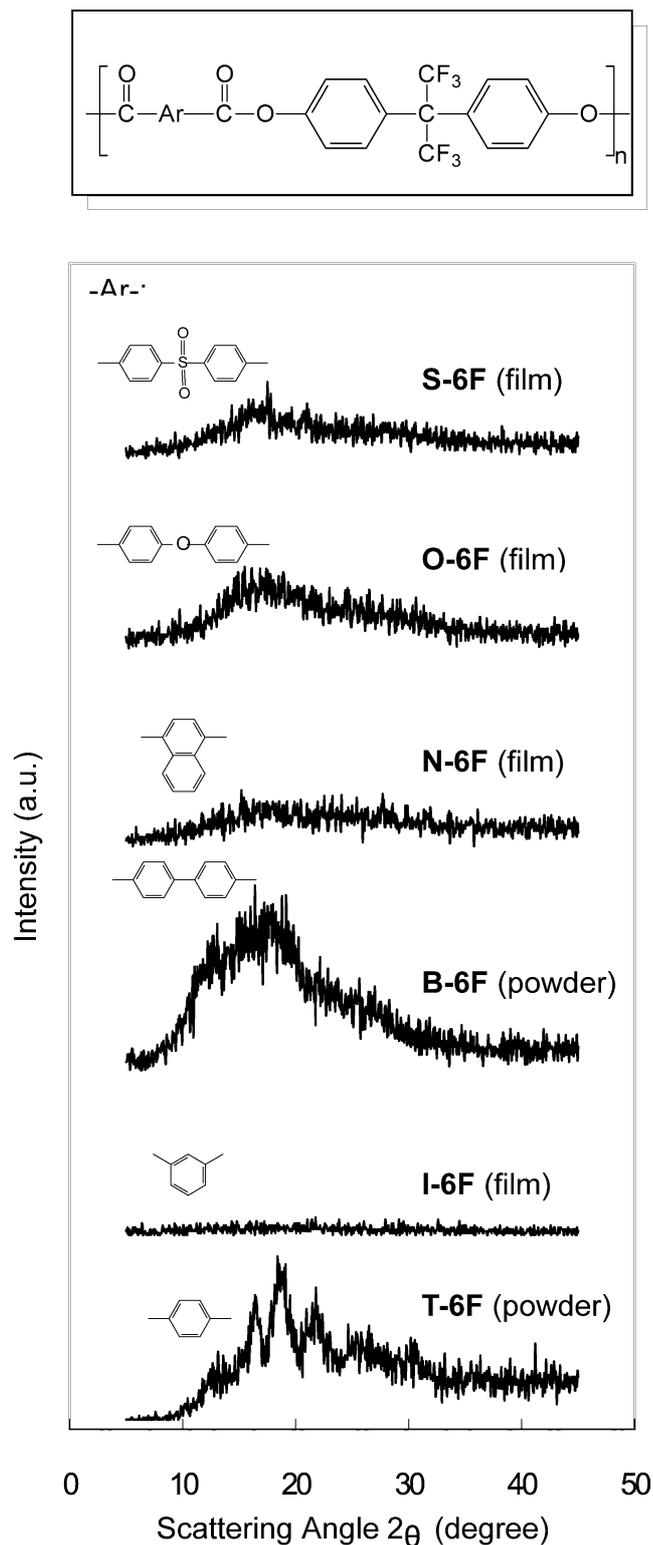


Figure 5. WAXD patterns of BPAF-derived polyarylates.

connector group, the polyarylates derived from bisphenols BPAP and BPAPF exhibited higher  $T_g$  and  $T_s$  values than the corresponding counterparts from BPA and BPAF. The fluorinated polyarylates also seem to reveal a slightly higher  $T_g$  and  $T_s$  value as compared to its nonfluorinated analogs, probably due to the increased structural hindrance caused by the bulkier  $-\text{CF}_3$  groups.

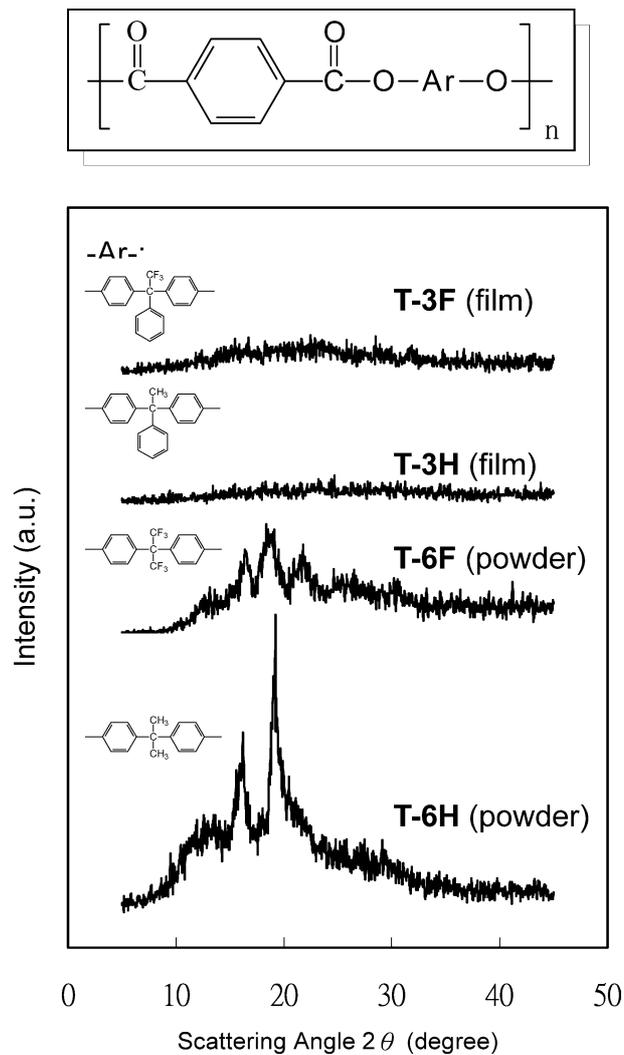


Figure 6. WAXD patterns of TPC-derived polyarylates.

Thermal and thermo-oxidative stabilities of these polyarylates were evaluated by TGA under both nitrogen and air atmospheres using a 5 or 10 wt% loss temperature for comparison. Typical TGA curves in nitrogen and air atmospheres of the representative TPC-derived polyarylates are shown in Figure 8. The TGA data of the polyarylates are also listed in Table 4. The decomposition temperatures ( $T_d$ ) of these polyarylates at 10% weight loss were all above 470 °C. In all cases, the fluorinated polyarylates exhibited a higher  $T_d$  value in comparison to their non-fluorinated counterparts, because of the higher C–F bond strength.

## Conclusions

A series of 24 polyarylates were synthesized from four bisphenols containing different connector groups with six aromatic diacid chlorides by the conventional techniques. The polyarylates derived from symmetric bisphenol BPA with diacid chlorides TPC, IPC, and BPDC and from bisphenol BPAF with diacid chlorides TPC and BPDC were semi-crystalline and showed lower solubilities. These polyarylates precipitated during the polymerization reactions and could

Table 4. Thermal behavior data of polyarylates

Polymer	$T_g^a$ (°C)	$T_s^b$ (°C)	$T_5$ wt% loss <sup>c</sup> (°C)		$T_{10}$ wt% loss <sup>c</sup> (°C)		Char yield <sup>d</sup> (wt%)
			In N <sub>2</sub>	In air	In N <sub>2</sub>	In air	
T-6H	202 (356)	–	501	440	519	472	29
T-6F	240 (314)	–	505	487	527	509	43
T-3H	244	248	516	472	533	493	41
T-3F	246	254	526	495	546	513	53
I-6H	184 (299)	190	515	452	525	474	29
I-6F	188	224	524	493	543	507	51
I-3H	213	215	513	473	525	493	35
I-3F	209	221	529	480	549	497	52
B-6H	–	–	480	423	508	450	32
B-6F	– (368)	–	519	490	545	507	50
B-3H	–	257	516	477	535	502	46
B-3F	269	277	530	492	551	515	56
N-6H	185	193	478	455	491	471	30
N-6F	189	205	478	469	504	492	44
N-3H	214	224	498	469	510	482	36
N-3F	221	231	517	493	538	510	54
O-6H	166	162	506	466	518	489	33
O-6F	180	192	498	487	522	515	44
O-3H	200	232	504	479	517	495	37
O-3F	216	239	507	480	526	496	51
S-6H	252	262	490	452	504	475	31
S-6F	254	219	505	491	521	512	46
S-3H	271	288	507	476	518	490	37
S-3F	271	282	507	488	526	507	47

<sup>a</sup>The samples were heated from 50 to 400 °C at a heating rate of 20 °C/min; this was followed by rapid cooling to 50 °C at 200 °C/min. The midpoint temperature of the baseline shift on the subsequent DSC heating trace (20 °C/min) was defined as  $T_g$ .

<sup>b</sup>Softening temperature, taken as the onset temperature of the probe displacement on the TMA trace. The film samples were heated at 300 °C for 30 min prior to the TMA trace.

<sup>c</sup>Decomposition temperature determined by TGA at a heating rate of 20 °C/min and a gas flow rate of 40 cm<sup>3</sup>/min.

<sup>d</sup>Residual weight percentage at 800 °C in nitrogen.

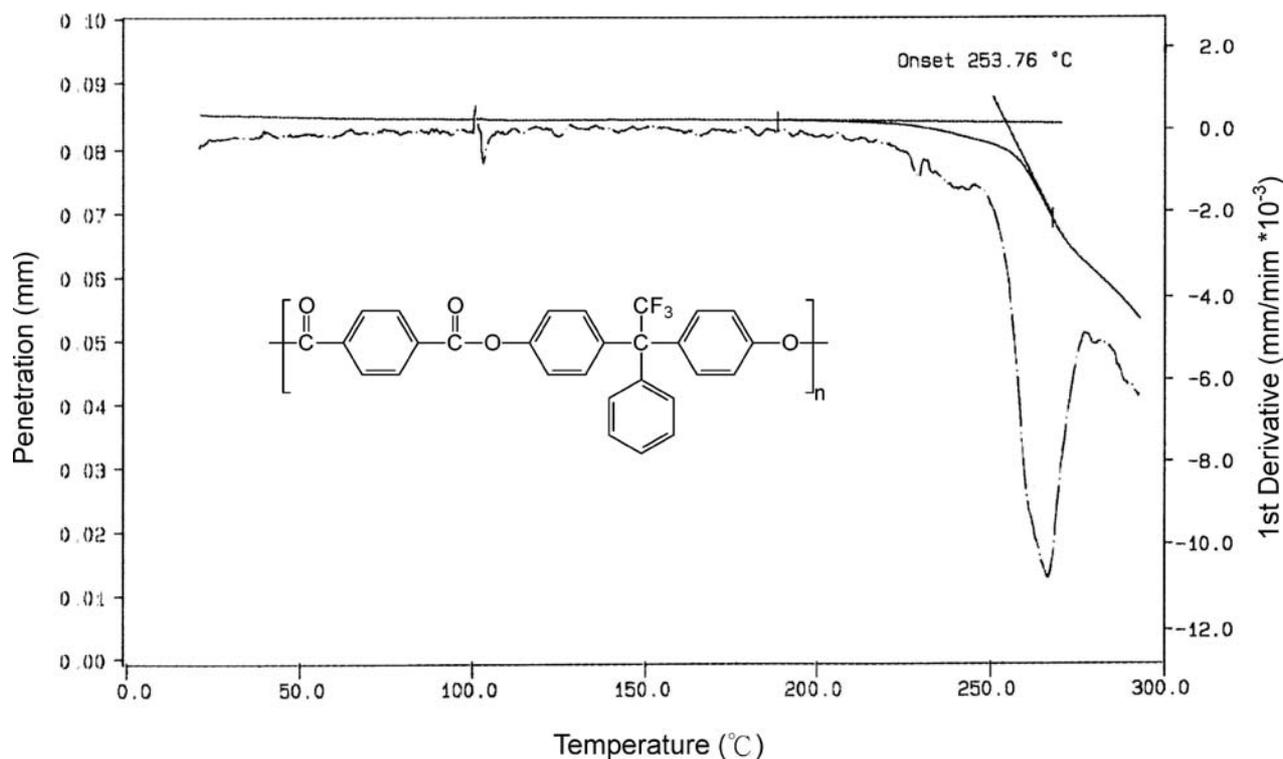


Figure 7. The TMA curve of polyarylate T-3F, at heating rate of 10 °C/min.

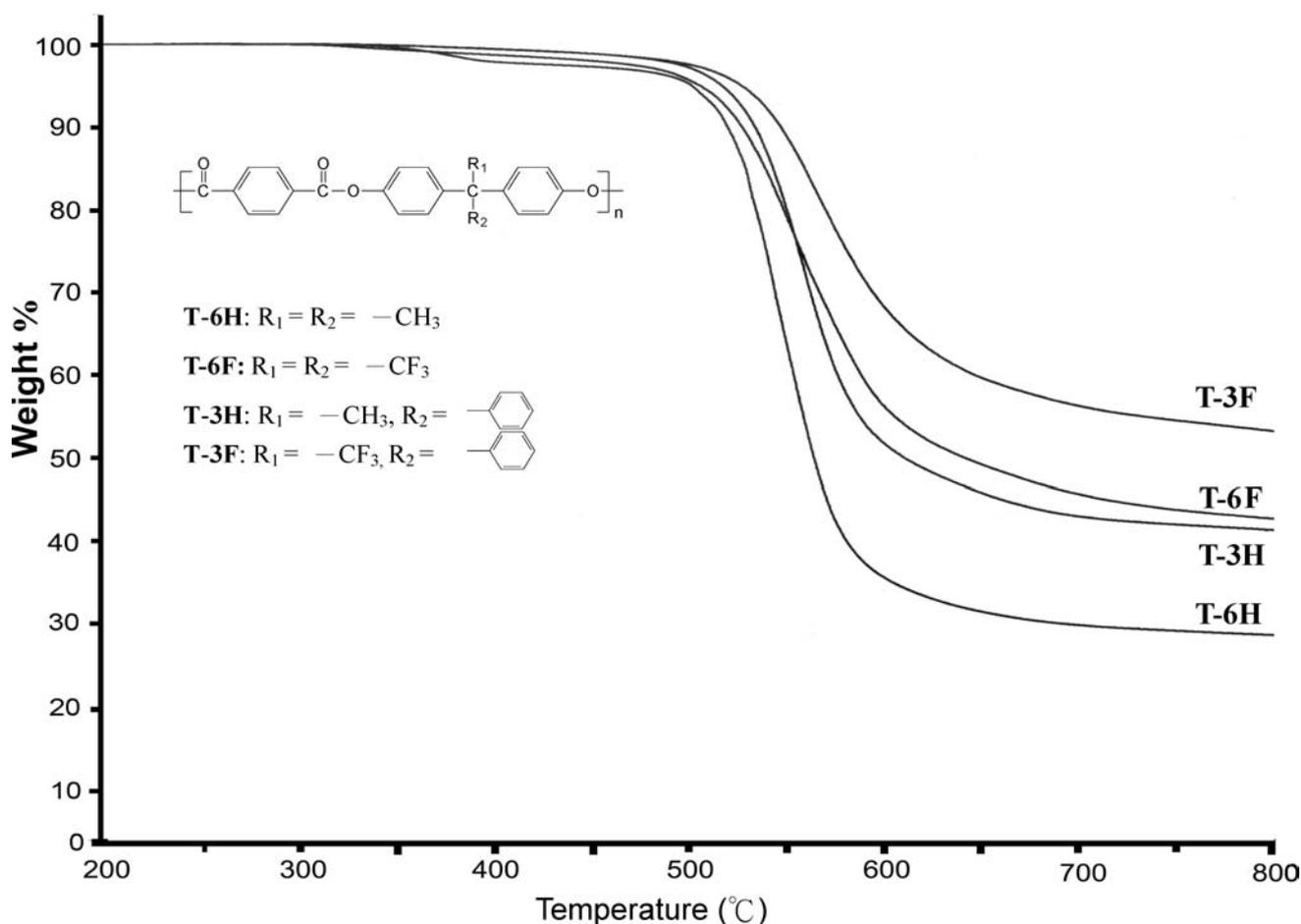


Figure 8. TGA curves of TPC-derived polyarylates, with a heating rate of 20 °C/min in nitrogen.

not afford flexible films. Other polyarylates were readily soluble in many organic solvents and could be solution cast into amorphous and tough films. These polyarylates also showed moderately high thermal stability. Thus, these polyarylates can be considered as candidates of high-performance polymeric materials. The introduction of bulky linking groups such as that of BPAP and BPAPF in the polymer backbone resulted in a decreased crystallinity and an enhanced solubility and  $T_g$ . In general, the fluorinated polyarylates revealed a slightly higher  $T_g$  and thermal stability as compared to nonfluorinated analogs.

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