Synthesis and Characterization of New Fluorinated Aromatic Polyesters Containing Trifluoromethylphenoxy Pendant Groups

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ABSTRACT: A series of novel fluorinated aromatic polyesters containing trifluoromethylphenoxy pendant groups was synthesized by interfacial polycondensation of 2-(4-trifluoromethylphenoxy)terephthalyl chloride with various bisphenols in dichloromethane. The polyesters obtained in good yields had weight-average molecular weights of 70,600–29,800 g/mol, polydispersities of 1.81–2.08, and were all amorphous. All polyesters were easily soluble in organic solvents such as *N,N*-dimethylformamide, tetrahydrofuran, *o*-chlorophenol, pyridine, and dichloromethane. These fluorinated polyesters showed glass transition temperature of 133–210°C, and good thermal stability with almost no weight loss up to 378°C, the 10% weight loss temperature of 472–523°C as well as char yield of 32–63% at 600°C in nitrogen. These polyester films cast from chloroform solutions exhibited tensile strengths ranging from 102 to 126 MPa, elongation at break from 6.3% to 11.7%, and tensile moduli from 2.1 to 3.3 GPa. The resulting polyester films also displayed low dielectric constants between 2.18 and 2.49 (1 MHz), high transparency with an ultraviolet-visible absorption cut-off wavelengths in the 332–355 nm range, and excellent electric strengths (50.4–65.6 kV/mm) and volume resistivity (2.51–6.03 × 10^{16} Ω cm). © 2010 Wiley Periodicals, Inc. J Appl Polym Sci 119: 702–708, 2011

Key words: fluorinated polyester; 2-(4-trifluoromethylphenoxy)terephthalyl chloride; interfacial polymerization; synthesis; property

INTRODUCTION

Aromatic polyesters exhibit good thermal stability, solvent resistance, and good mechanical properties and are, therefore, applied widely in the aviation, automobile, and electronic industries.[1–3] Despite their outstanding properties, most of them have no or high glass transition temperatures and poor solubility in organic solvents by virtue of their rigid structures, leading to difficulties in processing. Therefore, many efforts have been made to improve the processing characteristics of the relatively intractable polymers. Several approaches to improve the solubility of aromatic polyesters without much loss of their high thermal stability were the introduction of bulky pendant groups,[4–9] flexible groups,[10–13] or cardo groups[5,9,14–17] into the polymer backbone. The introduction of fluorine-containing groups in backbone or side chain of the polyesters is another efficient method to improve polymer solubility as well as electrical and dielectric performance. In general, most of the fluorinated polyesters were prepared by condensation polymerization of hydroxyl-terminated fluorinated diols or fluorinated polyethers with nonfluorinated diacids (derivatives),[18–24] or the condensation polymerization of fluorinated acid (derivatives) with diols or hydroxyl-terminated polyethers.[25,26] To our knowledge, comparatively little research has now been devoted to introduce trifluoromethyl pendant groups unsymmetrically into the polyester backbone. In this study, a series of new fluorinated aromatic polyesters based on a diacid chloride monomer, 2-(4-trifluoromethylphenoxy)terephthaloyl chloride (TFTPC)[27] was prepared. The solubility, crystallinity, thermal stability, and mechanical property as well as electrical insulating, optical and dielectric behaviors of the newly fluorinated polyesters were investigated.

EXPERIMENTAL

Materials

TFTPC[27] and 9,9-bis(4-hydroxyphenyl)xanthene (BHPX) (**2g**)[28] were prepared according to our reported methods. Bisphenol A (**2a**) and 4,4′-(1-phenylethylidene)bisphenol (**2d**) were obtained from Aldrich and used as received. 2,2-Bis-(4-hydroxy-3,5-dimethylphenyl)propane (**2b**),[29] 2,2′-dimethylbiphenyl-4,4′-diol (**2c**),[30] bis(4-hydroxyphenyl)diphenylmethane (**2e**),[31] and 1,1′-bis(4-hydroxyphenyl)cyclohexane (**2f**)[15] were

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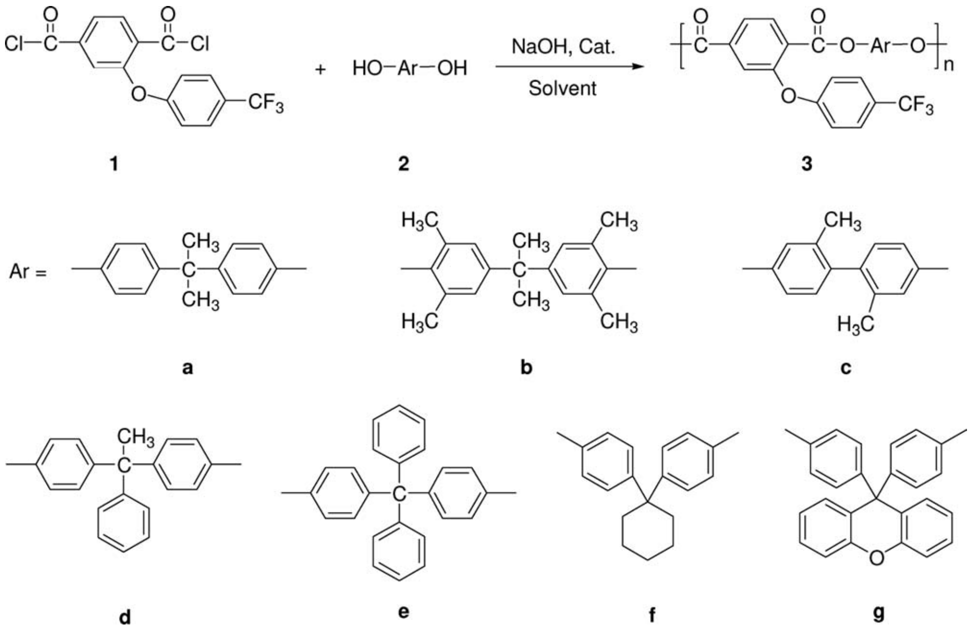

Scheme 1 Preparation of various new fluorinated polyesters.

prepared and purified by procedures reported in the Refs. 15 and 29–31. The solvents were purified according to standard methods.

Polymer synthesis

In a typical experiment, polyester (**3a**), which derived from TFTPC and bisphenol A (**2a**), was prepared as follows: In a 100-mL three-necked flask equipped with a mechanical stirrer, **2a** (1.1415 g, 5 mmol) and benzyltriethylammonium chloride (BTEAC) (0.11 g, 0.5 mmol) were dissolved in 10.0 mL of 1 *M* aqueous sodium hydroxide under nitrogen atmosphere, and the mixture was stirred for 30 min at room temperature. To this solution, TFTPC (1.8156 g, 5 mmol) in 20 mL of dichloromethane was added rapidly, and the mixture was stirred vigorously at room temperature for 3 h. The solution was poured into methanol with rapid stirring to induce the polymer precipitation, and the white precipitate was collected by suction filtration and dried *in vacuo*. The polymer was then redissolved in dichloromethane, reprecipitated using methanol, filtered, and dried at 120°C under reduced pressure. The white polyester (**3a**) was obtained in 2.38 g (92 %) with inherent viscosity of 0.43 dL/g. IR (KBr): ν = 1743 (C=O stretch), 1237, 1230 (C—F stretch) and 1282 cm^{-1} (C—O—C stretch); ^{1}H-NMR ($CDCl_3$): δ = 8.24 (d, *J* = 8.4 Hz, 1 H), 8.15 (d, *J* = 8.0 Hz, 1 H), 7.92 (s, 1 H), 7.60 (d, *J* = 8.4 Hz, 2 H), 7.31–7.19 (m, 4 H), 7.13–7.05 (m, 4 H), 6.94 (d, *J* = 8.4 Hz, 2 H), 1.68 (s, 6 H); ^{13}C-NMR ($CDCl_3$): δ = 163.5, 163.1, 160.3, 154.9, 148.5, 148.3, 148.2, 135.3, 132.7, 128.0, 127.9, 127.4, 127.3, 126.2, 123.7, 120.9, 120.8, 120.7, 120.6, 117.3, 42.6, 30.9.

All other polyesters (**3b–3g**) as white solid were prepared using a similar procedure.

Measurements

The ^{1}H-NMR (400 MHz) and ^{13}C-NMR (100 MHz) spectra were recorded on Bruker AVANCE 400-MHz spectrometer using $CDCl_3$ as the solvent and with tetramethylsilane as internal standard. The FTIR spectra of polymers in KBr pellets were determined on a Perkin-Elmer SP One FTIR spectrophotometer. Microanalyses were performed with a Carlo Erba 1106 Elemental Analyzer. Melting points were determined on X_4 melting point apparatus and were uncorrected. The glass-transition temperatures (T_g) were performed on a Perkin-Elmer DSC-7 instrument at a heating rate of 20°C/min under nitrogen protection. The second scan was immediately initiated after the sample was cooled to room temperature. The T_g values were reported from the second scan after the first heating and quenching, and taken from the midpoint of the change in the slope of the baseline. The thermal degradation temperature (T_d) of the polymers from 50°C to 600°C was determined with a Seiko SSC-5200 thermogravimetric analysis (TGA) at a heating rate of 10°C/min in nitrogen atmosphere (120 mL/min). The inherent viscosities were measured at 0.5g/dL concentration in NMP with an Ubbelohde viscometer at 30°C, in which the polyesters were pretreated by drying in oven at 120°C for 1 h to remove the adsorbed moisture. Weight-average (M_w) and number-average molecular weights (M_n) were determined by a gel permeation chromatography (GPC) on a Waters 510 HPLC equipped with 5-μm phenogel columns (linear, 3 × 500 Å) arranged in

TABLE I
Synthesis of Polyester 3a from TFTPC with 2a

Entry	Organic solvent	Catalyst[a]	Reaction time (h)	η_{inh} (dL/g)[b]
1	Toluene	BTEAC	3	0.28
2	Nitrobenzene	BTEAC	3	0.48
3	Dichloromethane	BTEAC	3	0.43
4	Dichloromethane	BTEAC	2	0.40
5	Dichloromethane	BTEAC	1	0.34
6	Dichloromethane	BTEAC	4	0.44
7	Dichloromethane	BTEAC	8	0.46
8	Dichloromethane	TBAB	3	0.35
9	Dichloromethane	BTEAB	3	0.40

[a] 10 mol % of catalyst based on TCTPC was used.
[b] Measured in NMP at a concentration of 0.5 g dL^{-1} at 30°C.
BTEAC, benzyltriethylammonium chloride; TBAB, tetrabutylammonium bromide; BTEAB, benzyltriethylammonium bromide.

series and a UV detector at 254 nm using tetrahydrofuran (1 mL/min) as the eluent, the polystyrene was used as the standard. The wide-angle X–ray diffraction measurements were conducted at room temperature (ca. 25°C) with a Rigaku Geiger Flex D-Max III X-ray diffractometer, using Ni-filtered CuK_α radiation (operating at 40 kV and 15 mA); the scanning rate was 2°/min over a range of 2θ = 5–40°). Mechanical properties of the thin films (cast from chloroform solutions onto the glass plates, and followed by heating at 100°C *in vacuo*) were evaluated at room temperature using an Instron 1121 instrument at a strain rate of 10 mm/min. The Ultraviolet-visible (UV–vis) spectra (200–600 nm) were recorded on a V-550 spectrophotometer. The electrical properties were measured on a Hewlett–Packard 4284A Presion LCR meter. The dielectric constants were determined by the bridge method with an LKI-1 capacitance meter at 25°C.

RESULTS AND DISCUSSION

Polymer synthesis

Several polyesters with trifluoromethylphenoxy pendant groups (**3a–3g**) were synthesized from TFTPC (**1**) with various bisphenols (**2a–2g**) according to Scheme 1 by the phase transfer-catalyzed interfacial polycondensation technique as described by Morgan.[32] The commercial bisphenol A (**2a**) was used to obtain the bisphenol A-based polyester for comparison in this study. The polymerization condition was investigated through several options, i.e., different solvents (toluene, dichloromethane, and nitrobenzene), different catalysts such as benzyltriethylammonium chloride (BTEAC), tetrabutylammonium bromide (TBAB), and benzyltriethylammonium bromide (BTEAB), and different reaction times (1, 2, 3, 4, and 8 h). As shown in Table I, nitrobenzene seemed to give the highest inherent viscosity under the same reaction conditions, but this solvent is much more toxic. After several experiments, the better result was observed when the alkaline solution of **2a** was added in one portion into the dichloromethane solution of a TFTPC using BTEAC as a phase transfer catalyst at room temperature for 3 h. Using the optimized conditions, the polyesters **3a–3g** were obtained with a high yield, above 90%, and inherent viscosities ranging from 0.36 to 0.70 dL/g (Table II). The number-average molecular weights (M_n), and weight-average molecular weights (M_w) of the

TABLE II
Polymerization Results, IR Spectra, and Elemental Analysis of the Polyesters

							Elemental analysis[d]	
Polymer	Yield (%)	η_{inh}[a] (dL/g)	M_w[b]	M_n[b]	PDI [c]	IR (C=O)	C (%)	H (%)
3a	92	0.43	37,700	20,800	1.81	1743	69.30 (69.50)	3.90 (4.08)
3b	91	0.36	29,800	16,100	1.85	1741	71.60 (71.83)	3.87 (4.08)
3c	93	0.40	38,400	20,400	1.88	1740	68.83 (69.05)	3.63 (3.80)
3d	90	0.69	64,500	33,300	1.94	1744	72.27 (72.41)	3.84 (4.00)
3e	94	0.70	70,600	35,700	1.98	1745	74.53 (74.76)	5.24 (5.45)
3f	96	0.45	42,000	20,200	2.08	1744	70.75 (70.96)	4.28 (4.51)
3g	96	0.67	56,200	28,800	1.95	1746	72.09 (72.28)	4.44 (4.70)

[a] Measured in NMP at a concentration of 0.5 g dL^{-1} at 30°C.
[b] Measured by GPC in THF, polystyrene was used as standard.
[c] The polydispersity index (PDI) was obtained by M_w/M_n.
[d] Theoretical percentages are in parentheses.

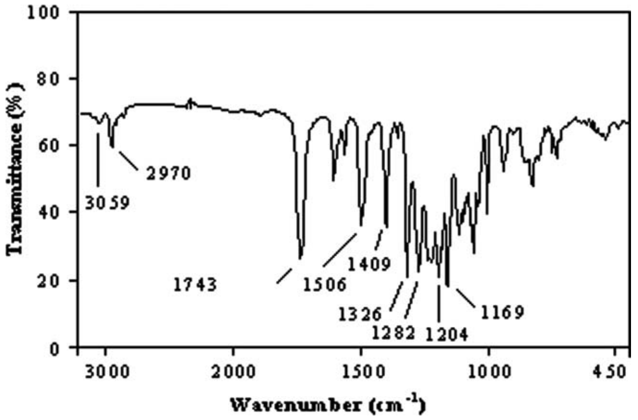

Figure 1 IR spectrum of the polyester (**3a**).

obtained polyesters with polydispersity values between 1.81 and 2.08 were in the range of 16,100–35,700 g/mol and 29,800–70,600 g/mol, respectively.

The structures of the polymers were confirmed to be aromatic polyesters by means of IR, NMR spectra, and elemental analysis. A typical IR spectrum of polymer **3a** was shown in Figure 1. The formation of the ester linkage was detected by the presence of characteristic ester carbonyl absorptions at 1743 and 1282 cm^{-1}. The other absorption bands such as a C—H asymmetric stretching of the methyl group at 2970 cm^{-1}, an aromatic C—H stretching absorption at 3059 cm^{-1}, O—C—O stretching at 1204 and 1169 cm^{-1}, and C—F stretching at 1326 cm^{-1} were also presented. Moreover, there were two absorption bands around 1506 and 1409 cm^{-1} due to C=C stretching of phenyl groups. Figure 2 showed the typical ^{1}H-NMR spectrum of the polyester **3a**, in which the resonance signals of aromatic protons appeared in the region of 6.92–8.24 ppm, and the absorption of CH_3 protons of bisphenol A appeared at 1.68 ppm as a single peak. The ^{13}C-NMR spectra of the obtained polyesters revealed that the carbon of the ester group resonated at downfield around 163.4 ppm. The elemental analysis values of these polyesters, as listed in Table II generally agreed with the calculated ones for the proposed structures.

Thermal property

The thermal behavior of these novel polyesters was evaluated by differential scanning calorimetry (DSC) as well as TGA. The typical DSC and TG curves of the polyester (**3a**) were shown in Figure 3, indicating its T_g and onset decomposition temperature (T_d) were 133 and 394°C in nitrogen, respectively. The thermal property of all the polymers was reported in Table III. The T_g of the polyesters were in the

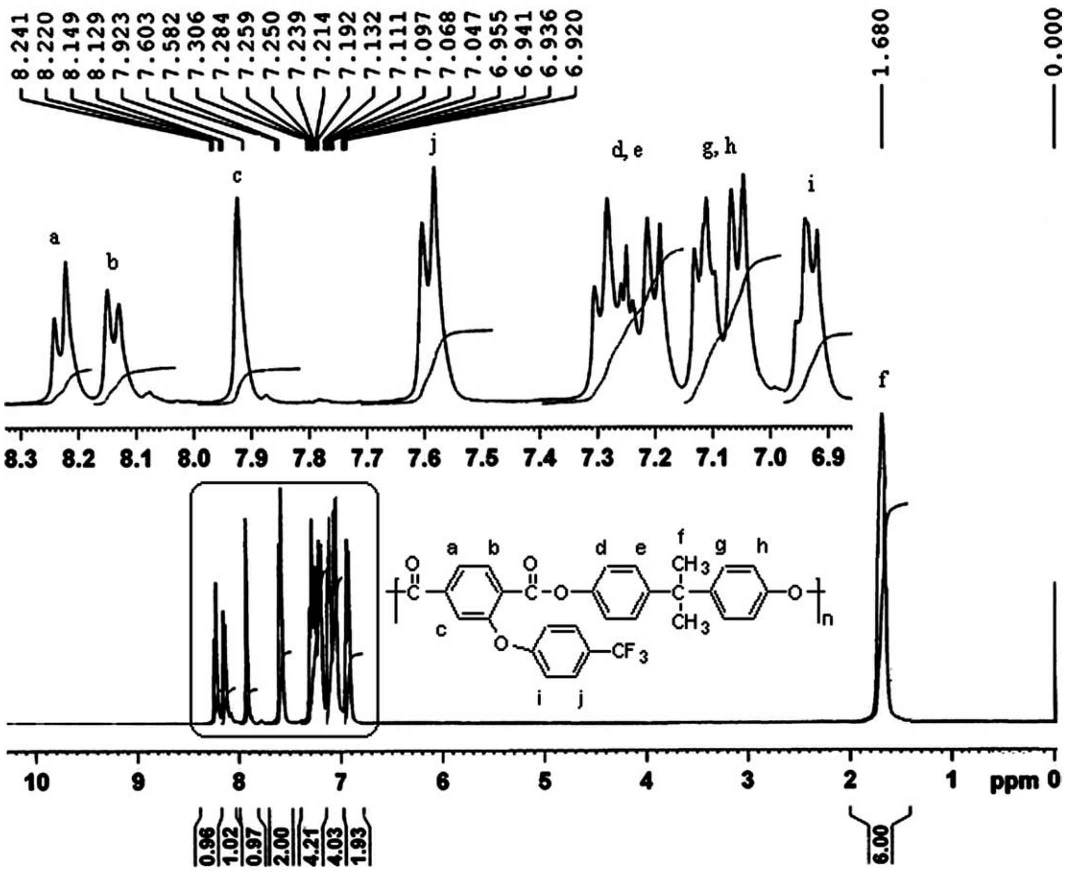

Figure 2 1H-NMR spectrum of the polyester (**3a**) in $CDCl_3$ solution.

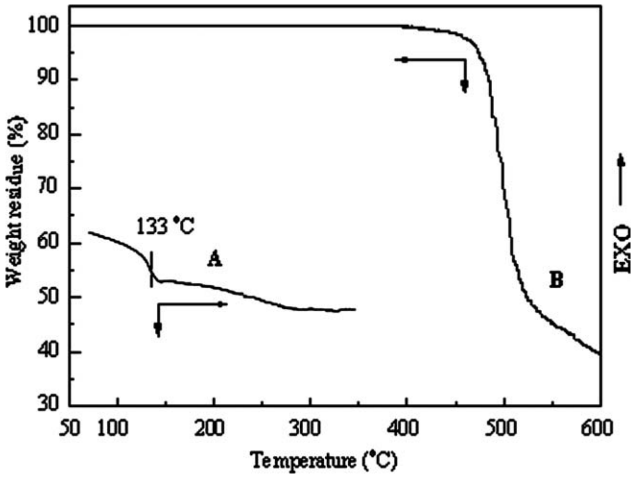

Figure 3 DSC (A) and TGA (B) curves for the polyester (**3a**) in nitrogen.

TABLE III
Thermal Properties of the Polyesters

Polymer	T_g (°C) [a]	T_d (°C) [b]	T_{d5} (°C) [c]	T_{d10} (°C) [d]	R_w (%) [e]
3a	133	394	473	482	40
3b	155	388	465	476	41
3c	143	402	480	490	45
3d	177	386	465	477	42
3e	180	428	488	503	60
3f	150	378	460	470	32
3g	210	397	493	510	63

[a] From the second heating trace of DSC measurements.
[b] Onset decomposition temperature in nitrogen.
[c] 5 % Weight loss temperatures measured by TGA in nitrogen.
[d] 10 % Weight loss temperatures measured by TGA in nitrogen.
[e] Residual weight (%) when heated to 600°C in nitrogen.

range of 133–210°C. Obviously, the T_g order of these polyesters was comparable to the decreasing order of stiffness and bulkiness of the polymer backbones. Among the resulted polyesters, polyester (**3a**) derived from bisphenol A showed the lowest T_g (133°C) value, attributed to the presence of isopropylidene, a kink unit, in the polymer backbone. The incorporation of the kink unit into the polymer backbone lowered the rigidity of the polymer backbone and reduced the T_g value of the polymers.[33] In contrast, polyester (**3b**) containing both kink isopropylidene and methyl-substituted phenylene displayed a higher T_g value than that of **3f**, which might be attributed to the fact that the methyl group on the phenylene unit inhibited the free rotation of the polymer chains leading to an enhanced T_g value. In addition, the polyester (**3e**) having diphenylmethylene unit in its backbone had a higher T_g (180°C) than those of the analogs containing isopropylidene (**3a** and **3b**), and triphenylmethylmethane (**3d**) units. For the cyclic side cardo polyesters, polyester (**3g**) containing xanthene group presented a higher T_g (210°C) than that of the analog with a bulky cyclohexyl pendant group (**3f**). On the other hand, polyester (**3g**) exhibited the highest T_g value in the series, which might reflect by the fact that the incorporating both rigid and bulky xanthene pendant group into the polymer backbone more effectively enhanced T_g value.

The thermal stability of these polyesters in a nitrogen atmosphere, as measured by TGA, was also summarized in Table III. All the polyesters exhibited good thermal stability being stable up to 378°C, 5%, and 10% weight loss temperatures (T_{d5} and T_{d10}) in the range of 460–493°C and 470–510°C in nitrogen, respectively. Among these polymers, polyester (**3g**), derived from BHPX, had the highest T_{d10} value, showing a higher thermal stability than that of the other polyesters. As expected, polyester (**3f**) containing the alicyclic hexyl group showed the lowest T_{d10} value, which might be attributed to its somewhat higher methylene contents of the cyclohexane groups in the main chain, leading to its lower thermal stability.[17]

Solubility of polymer

The solubilities of the polyesters in several organic solvents at 3.0% (w/v) were summarized in Table IV. Almost of all the polyesters exhibited good solubility in a variety of solvents such as *N*,*N*-dimethylformamide, *N*-methyl-2-pyrrolidinone, tetrahydrofuran,

TABLE IV
Solubility Behavior of the Polyesters in Various Organic Solvents

Polymer	DMF	DMSO	NMP	THF	Py	DCM	$CHCl_3$	*o*-Chlorophenol	*m*-Cresol
3a	+ +	−	+ +	+ +	+ +	+ +	+ +	+ +	+ −
3b	+ +	+	+ +	+ +	+ +	+ +	+ +	+ +	+ +
3c	+ +	+ −	+ +	+ +	+ +	+ +	+ +	+ +	+
3d	+ +	+	+ +	+ +	+ +	+ +	+ +	+ +	+
3e	+ +	+	+ −	+ +	+ +	+ +	+ +	+ +	+
3f	+ +	+ −	+ +	+ +	+ +	+ +	+ +	+ +	+ +
3g	+ +	+	+ +	+ +	+ +	+ +	+ +	+ +	+ +

Solubility: + + = soluble at room temperature; + = soluble on heating at 70°C, + − = partially soluble; − = insoluble.
DMF, *N*,*N*-dimethylformamide; THF, tetrahydrofuran; DMSO, dimethyl sulfoxide; NMP, *N*-methyl-2-pyrrolidinone; Py, pyridine; DCM, dichloromethane.

TABLE V
Mechanical Properties of the Polyesters

Polymer	Thickness (μm)	Tensile strength (MPa)	Tensile modulus (GPa)	Elongation at break (%)
3a	41.5	121 ± 5	2.4 ± 0.02	8.4 ± 0.2
3b	42.0	116 ± 4	2.4 ± 0.02	8.0 ± 0.1
3c	40.3	123 ± 6	2.6 ± 0.03	7.8 ± 0.2
3d	39.5	105 ± 3	2.5 ± 0.04	6.4 ± 0.1
3e	41.0	106 ± 2	2.6 ± 0.07	6.3 ± 0.2
3f	39.0	102 ± 1	2.1 ± 0.02	10.2 ± 0.3
3g	41.6	126 ± 5	3.3 ± 0.10	11.7 ± 0.3

m-cresol, *o*-chlorophenol, pyridine, dichloromethane, and chloroform at room temperature or upon heating at 70°C. For comparison, polyester (**3a**) derived from TFTPC with bisphenol A (**2a**) showed less solubility. The above result demonstrated that introduction of the trifluoromethylphenoxy pendant group, or combined with bulky cardo groups such as diphenylmethylene, cyclohexyl, and xanthene into the polymer backbone enhanced the solubility of the polyesters. The pendant group probably disturbed dense chain packing of the polymer chains; consequently, the solvent molecules could penetrate easily to solubilize the polymer chain.[34]

Mechanical and crystal properties

Colorless and transparent films of the polyesters **3a**–**3g** could readily be prepared by casting their chloroform solutions. Their mechanical properties with tensile strengths of 102–126 MPa, elongations at break of 6.3–11.7%, and tensile modulus of 2.1–3.3 GPa, shown in Table V, were good and could be used as useful engineering materials.

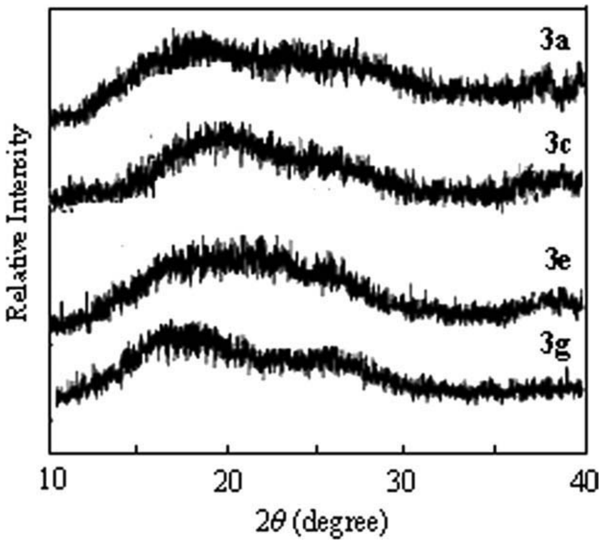

Figure 4 Wide-angle X-ray diffraction patterns of polyesters **3a**, **3c**, **3e**, and **3g**.

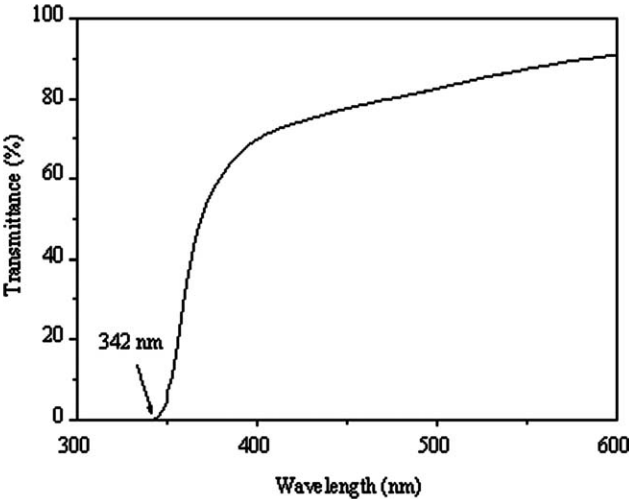

Figure 5 Percent transparency of the polyester film (**3a**).

The crystallinity of these new polyesters was estimated by means of wide-angle X-ray diffractograms, and all the polymers presented amorphous patterns. Some selected WAXD patterns were shown in Figure 4. The results might be attributable to the presence of the bulky, asymmetrical trifluoromethylphenoxy pendant groups, which resulted in poor chain packing. Additionally, among the cardo polyesters in this study, the bulky cardo group also decreased the intermolecular force between the polymer chains, subsequently causing a decrease in crystallinity. Thus, the amorphous structure of these polyesters also reflected in their excellent solubility and good film forming ability.

Optical, electrical, and dielectric properties

The optical properties of these polyester films were evaluated by UV–vis spectroscopy. The typical UV–vis spectrum of polyester film (**3a**) was displayed in Figure 5. All polyesters had good transparency in a visible region with over 80% of the transmittance, and their cutoff wavelengths of these polyesters ranged from 332 to 355 nm (Table VI). Additionally, the polyester films also showed good electrical

TABLE VI
Optical, Electrical, and Dielectric Properties of the Polyesters

Polymer	Cut off wavelength (nm)	Dielectric constant (1 MHz)	Volume resistivity (Ω cm)	Dielectric strength (kV/mm)
3a	342	2.25	2.86×10^{16}	55.5
3b	354	2.18	2.80×10^{16}	54.1
3c	342	2.35	3.45×10^{16}	56.7
3d	350	2.49	2.90×10^{16}	55.0
3e	340	2.21	3.78×10^{16}	57.8
3f	332	2.20	2.51×10^{16}	50.4
3g	355	2.32	6.03×10^{16}	65.6

insulating and dielectric properties, and their volume resistivity, dielectric strength were in the range of 2.51–6.03 × 10^{16} Ω cm and 50.4–65.6 kV/mm, respectively. Moreover, the dielectric constants at 1 MHz were measured in the range of 2.18–2.49. The low dielectric constants of the fluorinated polyesters could be attributed to the low polarizability of the C—F band and the increase in free volume. These results suggested that the CF_3 groups in the polymer chains played an important role in the electrical and dielectric performance.

CONCLUSIONS

Some novel fluorinated polyesters from 2-(4-trifluoromethylphenoxy)terephthaloyl chloride with various bisphenols were successfully synthesized by interfacial polycondensation procedure. These polyesters exhibited good mechanical property, thermal stability, electrical and dielectric properties, as well as excellent solubility, and optical property. These characteristics indicated that these fluorinated polyesters could be considered as new candidates for processable high-performance engineering plastic and photoelectric materials.

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