

Synthesis and Characterization of New Aromatic Polyesters Containing Pendent Naphthyl Units

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ABSTRACT: Two bisphenols, viz., 4,4'-[1-(2-naphthalenyl)ethylidene]bisphenol and 4,4'-[1-(2-naphthalenyl) ethylidene]bis-3-methylphenol were prepared by condensation of commercially available 2-acetonaphthanone with phenol and *o*-cresol, respectively. A series of new aromatic polyesters containing pendent naphthyl units was synthesized by phase-transfer-catalyzed interfacial polycondensation of these bisphenols with isophthaloyl chloride, terephthaloyl chloride, and a mixture of isophthaloyl chloride/terephthaloyl chloride (50 : 50 mol %). Inherent viscosities of polyesters were in the range 0.83–1.76 dL g⁻¹, while number average molecular weights (M_n) were in the range 61,000–235,000 g mol⁻¹. Polyesters were readily soluble in organic solvents such as dichloromethane, chloroform, tetrahydro-

furan, *m*-cresol, pyridine, *N,N*-dimethylformamide, *N,N*-dimethylacetamide, and 1-methyl-2-pyrrolidinone at room temperature. Tough, transparent, and flexible films were cast from a solution of polyesters in chloroform. X-Ray diffraction measurements displayed a broad halo at $2\theta \cong 19^\circ$ indicating the amorphous nature of polyesters. Glass transition temperatures of polyesters were in the range 209–259°C. The temperature at 10% weight loss (T_{10}), determined by TGA in nitrogen atmosphere, of polyesters was in the range 435–500°C indicating their good thermal stability. © 2010 Wiley Periodicals, Inc. *J Appl Polym Sci* 117: 2545–2552, 2010

Key words: aromatic polyesters; pendent naphthyl units; bisphenol; interfacial polycondensation

INTRODUCTION

Aromatic polyesters are an important class of high performance thermoplastics which possess high thermal stability, chemical resistance, and excellent mechanical properties and find applications in aviation, automobile, and electronic industries.^{1,2} However, most aromatic polyesters are generally difficult to process because of their limited solubility in organic solvents and their high melting or high glass transition temperature by virtue of their rigid structure.^{3,4} Therefore, development of aromatic polyesters with improved solubility and processability and that too without sacrificing thermal properties is an important goal. A lot of efforts have been expended to improve the processing characteristics of aromatic polyesters. One of the approaches to obtain high T_g and yet soluble polyesters is the introduction of bulky pendent groups along the polymer backbone.^{5–26}

In our earlier work, we demonstrated that polyesters containing biphenyl as pendent groups exhibited high glass transition temperatures (232–256°C) and improved solubility.⁵ In this work, we wish to report synthesis of two bisphenols, viz., 4,4'-[1-(2-naphthalenyl) ethylidene]bisphenol (NABP) and 4,4'-[1-(2-naphthalenyl)ethylidene]bis-3-methylphenol (*o*-NABP). A series of new aromatic polyesters was prepared by phase-transfer-catalyzed interfacial polycondensation of these bisphenols with isophthaloyl chloride (IPC), terephthaloyl chloride (TPC), and a mixture of IPC/TPC (50 : 50 mol %). Polyesters were characterized by inherent viscosity measurements, solubility tests, FTIR, ¹H-NMR spectroscopy, gel permeation chromatography (GPC), X-ray diffraction analysis (WAXD), thermogravimetric analysis (TGA), and differential scanning calorimetry (DSC). The effect of pendent naphthyl units on properties of polyesters such as solubility behavior and thermal transitions is discussed.

EXPERIMENTAL

Materials

3-Mercaptopropionic acid, benzyltriethylammonium-chloride (BTEAC), and calcium hydride (Aldrich, USA) were used as received. 2-Acetonaphthanone,

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phenol, *o*-cresol, concentrated hydrochloric acid, glacial acetic acid, sodium bicarbonate, and sodium sulfate (Merck, India) were used as received. Terephthalic acid chloride (TPC) and isophthalic acid chloride (IPC) were synthesized from terephthalic acid and isophthalic acid (both, received from Aldrich, USA), respectively using excess thionyl chloride in the presence of *N,N*-dimethylformamide as a catalyst and were purified by distillation under reduced pressure.²⁷

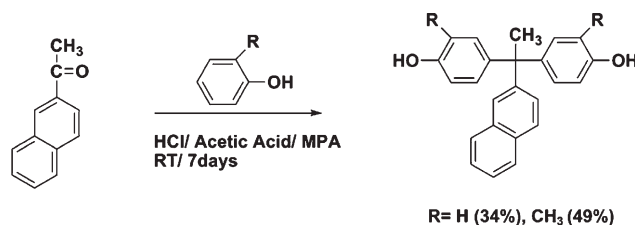
Measurements

Inherent viscosities of polyesters were measured on 0.5% (w/v) solutions of aromatic polyesters in chloroform at $30 \pm 0.1^\circ\text{C}$ using an Ubbelohde suspended level viscometer. Solubility of polyesters was determined at 3 wt % concentration in various solvents at room temperature or upon heating. FTIR spectra were recorded as KBr pellets for bisphenols and as films for polyesters on a Perkin-Elmer 883 spectrophotometer. ^1H - and ^{13}C -NMR spectra of monomers and polyesters were recorded on a Bruker 200 or 500 MHz spectrophotometer using $\text{DMSO-}d_6$ or CDCl_3 as a solvent. Melting points were determined by open capillary method and are uncorrected. HPLC analysis was performed on Waters Modular System consisting of three 515 pumps, 717 auto sampler, 996-photodiode array detector, 2410 RI detector controlled by *Millenium 32* software. Column used was *Zorbax RP-C8*. Molecular weights of aromatic polyesters were measured on ThermoFinnigan make gel permeation chromatograph (GPC), using the following conditions: Column—polystyrene-divinylbenzene (10^5 Å to 50 Å), Detector—RI, room temperature. Polystyrene was used as the reference standard. Polymer sample (5 mg) was dissolved in 5 mL chloroform and filtered through 0.2 μ SS-filter. X-ray diffraction patterns of polymers were obtained on a Rigaku-Dmax 2500 X-Ray diffractometer at a tilting rate of 2° min^{-1} . Thermogravimetric analysis was performed on Perkin-Elmer TGA-7 system at a heating rate of $10^\circ\text{C min}^{-1}$ under nitrogen atmosphere. Sample weight taken was ~ 5 mg. Glass transition temperatures (T_g) of polyesters were determined on Perkin-Elmer DSC-7 at a heating rate of $20^\circ\text{C min}^{-1}$ in nitrogen atmosphere.

Synthesis of monomers

Preparation NABP

Into a 500 mL round bottom flask equipped with a magnetic stirring bar were charged 2-acetonaphthone (40 g, 0.24 mol), phenol (132 g, 1.41 mol), 3-mercaptopropionic acid (2.5 g, 0.02 mol), concen-



Scheme 1 Synthesis of bisphenols containing naphthyl unit.

trated hydrochloric acid (100 mL), and glacial acetic acid (50 mL). The flask was stoppered, and the reaction mixture was stirred at room temperature for 7 days. The reaction mixture was dissolved in ethyl acetate (800 mL) and washed with aqueous sodium bicarbonate solution (3×200 mL) followed by washing with water (200 mL). Ethyl acetate was removed on a rotary evaporator. Excess phenol was removed by distillation under reduced pressure, and the crude product was purified by recrystallization from benzene.

Yield = 27 g (34%), M.P. = 190°C .

Purity (HPLC): $> 99.9\%$.

FTIR (KBr disk): 3336 cm^{-1} (O—H).

^1H -NMR ($\text{DMSO-}d_6$, ppm): 9.44 (s, 2H), 7.18–7.82 (m, 7H), 6.86 (d, 2H), 6.66 (d, 2H), 2.08 (s, 3H).

^{13}C -NMR ($\text{DMSO-}d_6$, ppm): 155.0, 147.6, 139.7, 133.0, 131.6, 129.7, 128.2, 127.4, 126.3, 126.0, 115.0, 51.3, 30.7.

Preparation *o*-NABP

Into a 500 mL round bottom flask equipped with a magnetic stirring bar were charged 2-acetonaphthone (30 g, 0.18 mol), *o*-cresol (114 g, 1.05 mol), 3-mercaptopropionic acid (1.9 g, 0.02 mol), concentrated hydrochloric acid (80 mL), and glacial acetic acid (40 mL). The flask was stoppered and the reaction mixture was stirred at room temperature for 7 days. The reaction mixture was dissolved in ethyl acetate (500 mL) and was washed with aqueous sodium bicarbonate solution (3×200 mL) followed by washing with water (200 mL). Excess *o*-cresol was removed by distillation under reduced pressure, and the crude product was purified by recrystallization from toluene.

Yield: 32 g (49%), M.P. = 170°C .

Purity (HPLC): $> 99.9\%$.

FTIR (KBr disk): 3336 cm^{-1} (O—H).

^1H -NMR ($\text{DMSO-}d_6$, ppm): 9.24 (s, 2H), 7.25–7.82 (m, 7H), 6.85 (s, 2H), 6.71 (d, 4H), 2.08 (s, 3H), 2.05 (s, 6H).

^{13}C -NMR ($\text{DMSO-}d_6$, ppm): 153.5, 147.9, 139.5, 132.8, 131.4, 130.7, 128.0, 127.9, 127.3, 127.1, 127.0, 126.1, 125.7, 123.1, 114.1, 51.0, 30.7.

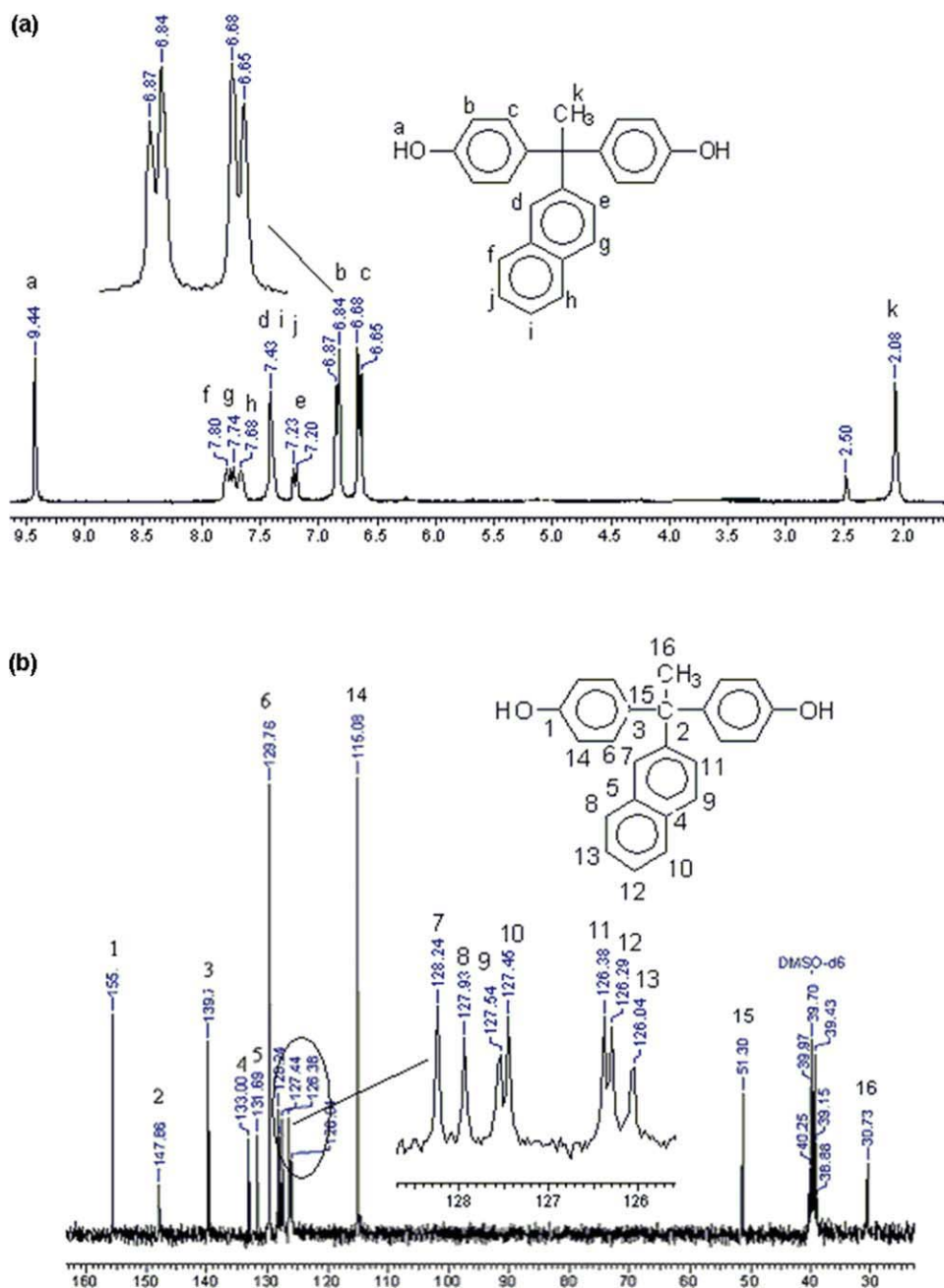


Figure 1 (a) ^1H -NMR (top) and (b) ^{13}C -NMR (bottom) spectrum in ($\text{DMSO}-d_6$) of NABP. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

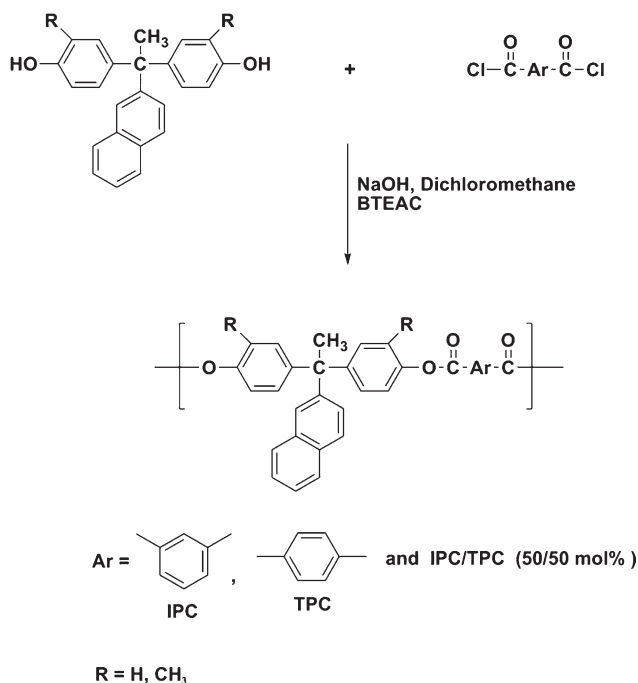
Synthesis of polyesters

A representative procedure for synthesis of polyesters is described below.

Polymerizations were carried out in a 100 mL two-necked round bottom flask equipped with a mechanical stirrer. In a typical polymerization reaction, NABP (1.70 g, 5.0 mmol) was dissolved in 10 mL of 10 mmol aqueous solution of sodium hydroxide. The mixture was stirred for 1 h at 10°C . Next, BTEAC (30 mg) was added to the reaction mixture, and stirring was continued. After 30 min, a solution of IPC (1.015 g, 5 mmol) in 20 mL of

dichloromethane was added to the reaction mixture, and the mixture was stirred vigorously at 2000 rpm for 1 h. The reaction mixture was poured into hot water; the precipitated polymer was filtered and washed several times with water. The polymer was dissolved in chloroform and reprecipitated into methanol. The fibrous polymer obtained was filtered, washed with methanol, and dried under reduced pressure at 80°C for 24 h. The yield of polyester was almost quantitative.

A similar procedure was followed for the synthesis of other polyesters and copolyesters.



Scheme 2 Synthesis of polyesters containing pendent naphthyl units.

RESULTS AND DISCUSSION

Monomer synthesis and characterization

Scheme 1 depicts route for synthesis of NABP and *o*-NABP. NABP and *o*-NABP were synthesized by the reaction of 2-acetonaphthanone with excess phenol and *o*-cresol, respectively, in the presence of a mixture of concentrated hydrochloric acid and glacial acetic acid (2 : 1, v/v) and 3-mercaptopropionic acid as a catalyst system. Both the bisphenol monomers NABP and *o*-NABP have been claimed in the patent.²⁸ However, structural characterization data has not been provided.

The structure of NABP and *o*-NABP was confirmed by FTIR and NMR spectroscopy. In the FTIR

spectrum of NABP, a band at 3336 cm⁻¹ was observed which corresponds to phenolic hydroxyl group. Figure 1(a,b) shows ¹H- and ¹³C-NMR spectra, respectively of NABP along with the assignments. In ¹H-NMR spectrum of NABP, a singlet observed at 9.44 ppm is assignable to proton of hydroxyl group. The seven protons of naphthyl ring exhibited a multiplet in the range 7.20–7.84 ppm. The doublets at 6.66 and 6.86 ppm correspond to aromatic protons *ortho* and *meta* to hydroxyl group, respectively. A singlet corresponding to methyl group attached to bridge carbon atom appeared at 2.08 ppm. The peak assignments in ¹³C-NMR spectrum were confirmed by DEPT spectrum. Mass spectra of NABP and *o*-NABP showed molecular ion peak at 340 and 368, respectively. The purity of NABP and *o*-NABP were determined by HPLC and was found to be >99.9%.

Polymer synthesis

A series of aromatic polyesters was synthesized by polycondensation of bisphenols containing pendent naphthyl groups, viz., NABP and *o*-NABP with diacid chlorides, viz., IPC and TPC and a (50 : 50 mol %) mixture of IPC/TPC in dichloromethane-aqueous sodium hydroxide system using BTEAC as a phase-transfer catalyst (Scheme 2). Inherent viscosities of polyesters were in the range 0.83–1.76 dL g⁻¹ indicating the formation of medium to high molecular weight polymers. Molecular weights of aromatic polyesters were measured by GPC (Table I). Number average molecular weight (M_n) and polydispersity index of polyesters were in the range 61,000–235,000 g mol⁻¹ and 2.0–2.3, respectively. GPC data indicate the formation of medium to high molecular weight polyesters. However, the molecular weight values provided by GPC should not be taken as absolute as the calibration of GPC was carried out using polystyrene standards.

TABLE I
Synthesis of Aromatic Polyesters Containing Pendent Naphthyl Units

Polyester	Bisphenol	Diacid chloride	η_{inh} dL/g ^a	Molecular weight g/mol		
				M_n	M_w	M_w/M_n ^b
PES-1	NABP	TPC	0.97	61,000	137,000	2.2
PES-2	NABP	IPC	1.0	103,000	214,000	2.1
PES-3	NABP	TPC-IPC (50:50)	1.76	235,000	475,000	2.0
PES-4	<i>o</i> -NABP	TPC	1.10	67,000	152,000	2.3
PES-5	<i>o</i> -NABP	IPC	0.83	82,000	172,000	2.1
PES-6	<i>o</i> -NABP	TPC-IPC (50:50)	1.47	133,000	257,000	2.0

^a η_{inh} of polyester was measured with 0.5% (w/v) solution in chloroform at 30 ± 0.1°C.

^b Measured on GPC in chloroform; polystyrene was used as the calibration standard.

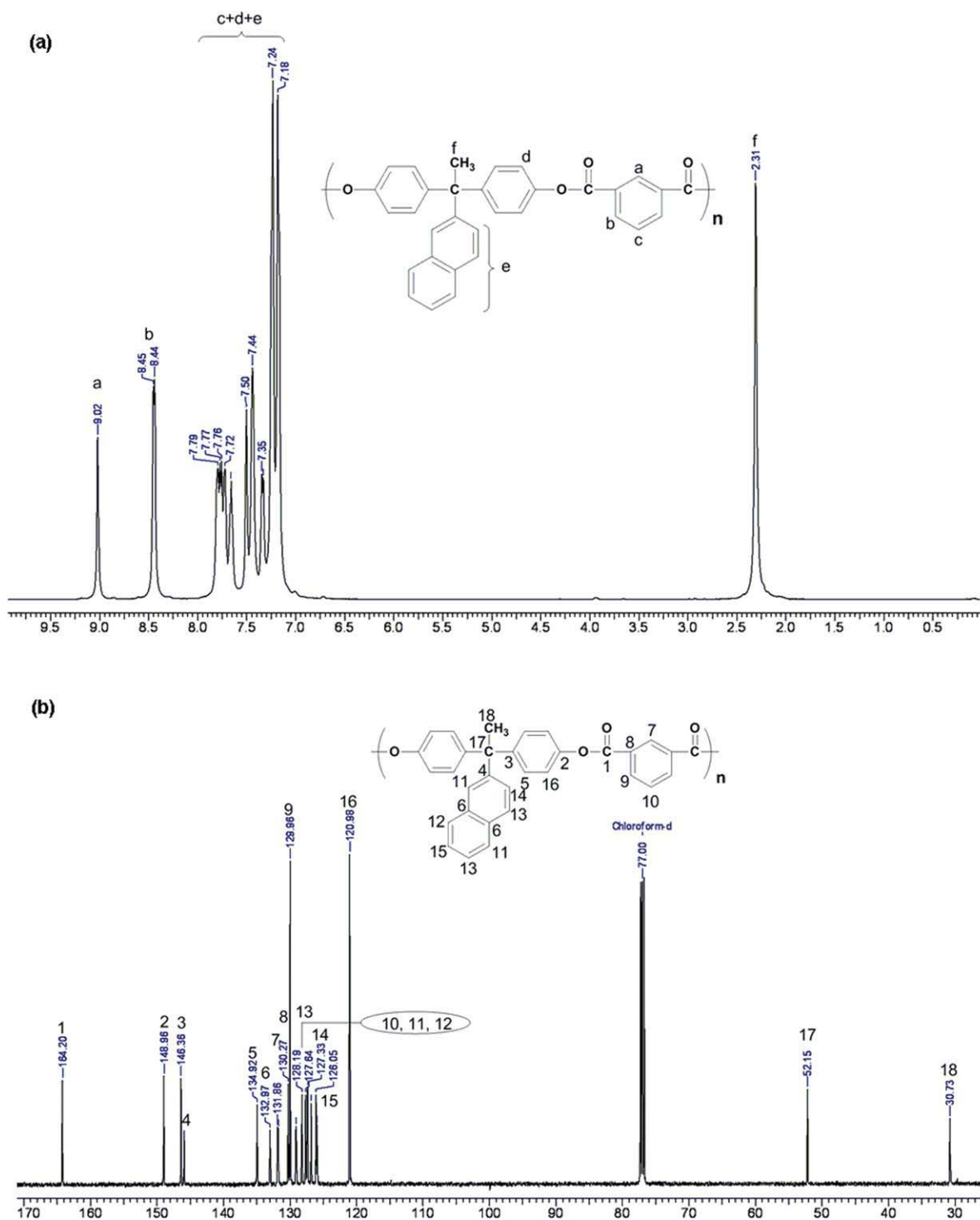


Figure 2 (a) ^1H -NMR (top) and (b) ^{13}C -NMR (bottom) spectrum in (CDCl_3) of polyester derived from NABP and IPC. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

Tough, transparent, and flexible films of polyesters containing pendent naphthyl units could be cast from their chloroform solutions.

Polymer characterization

The formation of polyesters was confirmed by FTIR, ^1H -NMR, and ^{13}C -NMR spectroscopy. FTIR spec-

trum of polyester showed band of ester carbonyl group at 1743 cm^{-1} . A representative ^1H - and ^{13}C -NMR spectra of polyester derived from NABP and IPC along with assignments is shown in Figure 2(a,b). Three different types of protons "a," "b," and "c" can be distinguished for isophthalic moiety in ^1H -NMR spectrum. The most deshielded proton "a" is located at 9.02 ppm as a singlet. The magnetically

TABLE II
Solubility of Aromatic Polyesters Containing Pendent Naphthyl Units

Polyester	DCM	CHCl ₃	THF	<i>m</i> -Cresol	Pyridine	DMF	NMP	DMAc	DMSO
PES-1	++	++	++	++	++	++	++	++	-+
PES-2	++	++	++	++	++	++	++	++	-+
PES-3	++	++	++	++	++	++	++	++	-+
PES-4	++	++	++	++	++	++	++	++	-+
PES-5	++	++	++	++	++	++	++	++	-+
PES-6	++	++	++	++	++	++	++	++	-+

Solubility: ++ = soluble at room temperature; -+ = partially soluble on heating at 70°C.

DCM = dichloromethane, CHCl₃ = chloroform, THF = tetrahydrofuran, DMF = *N,N*-dimethylformamide, DMAc = *N,N*-dimethylacetamide, NMP = 1-methyl-2-pyrrolidinone, DMSO = dimethyl sulfoxide.

equivalent protons "b" exhibited a doublet at 8.45 ppm. A multiplet in the range 6.58–8.0 ppm is due to the proton "c," protons *ortho* to oxygen "d" and naphthyl group protons "e." The protons of methyl group attached to bridge carbon exhibited a singlet at 2.31 ppm. The peak assignments in ¹³C-NMR spectrum [Fig. 2(b)] were confirmed by DEPT spectrum.

The solubility behavior of aromatic polyesters was examined in different solvents. Solubility data of polyesters in several organic solvents at 3% (w/v) is given in Table II. Aromatic polyesters dissolved readily in solvents such as dichloromethane, chloroform, tetrahydrofuran, *m*-cresol, pyridine, *N,N*-dimethylformamide, *N,N*-dimethylacetamide, and 1-methyl-2-pyrrolidinone at room temperature and were spar-

ingly soluble in dimethyl sulfoxide even upon heating at 70°C. In contrast to these results, aromatic polyesters synthesized from bisphenol-A (BPA) and TPC and BPA and IPC are insoluble in these organic solvents.²⁹ This result indicates that the incorporation of pendent naphthyl units into the polyester backbone improved the solubility of aromatic polyesters.

X-Ray diffraction patterns of aromatic polyesters showed an amorphous halo at $2\theta = 19^\circ$ (Fig. 3). This could be attributed to the presence of naphthyl pendent units into the polymer backbone which hinders the dense chain packing of polymer chains resulting in amorphous nature of these polyesters, which is also reflected in their improved solubility.

Glass transition temperature (T_g) of the aromatic polyesters was measured by DSC. The results are summarized in Table III and representative DSC curves are reproduced in Figure 4. DSC thermograms of NABP-based polyesters with TPC and IPC obtained from second heating scan at a heating rate of 20°C min⁻¹ showed T_g at 259°C and 234°C, respectively. The T_g value for copolyester from NABP with IPC/TPC (50 : 50 mol %) was observed at 245°C. It was observed that the T_g values for NABP-IPC and NABP-TPC were higher than those of the corresponding polyesters based on BPA. The T_g values for polyesters based on BPA-IPC and BPA-TPC are 181°C and 210°C, respectively.⁵ It was observed that the T_g values for NABP-based polyesters with IPC, TPC and IPC/TPC were higher than those of the corresponding polyesters based on *o*-NABP. The T_g values for polyesters obtained from *o*-NABP with IPC and TPC were 209°C and 238°C, respectively. The copolyester derived from *o*-NABP with IPC/TPC (50 : 50 mol %) showed T_g at 216°C. This behavior is attributed to the presence of methyl group on bisphenol moiety *ortho* to ester linkage, which makes unsymmetrical structure. It is reported that an unsymmetrical molecule decreases the T_g of the polymers.^{30–32} Dimethyl substitutions on bisphenols results into an asymmetric segment,

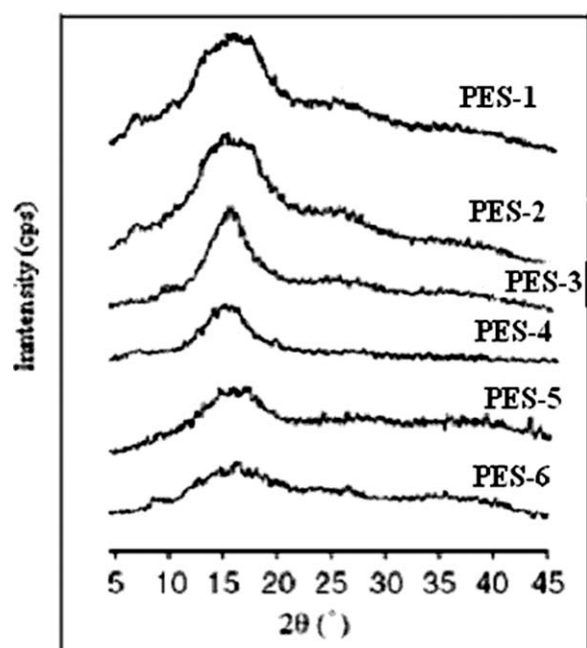
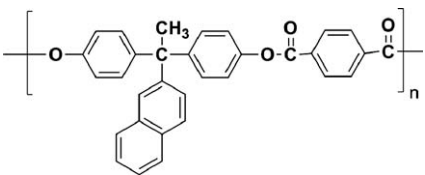
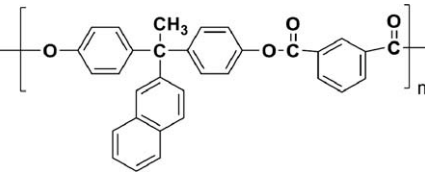
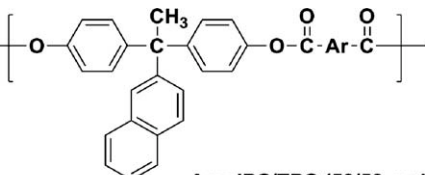
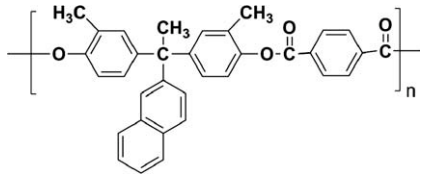
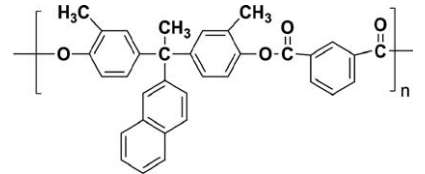
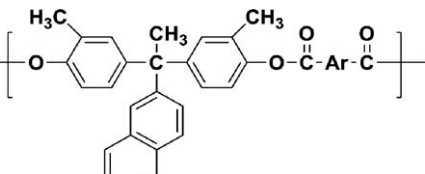


Figure 3 X-Ray diffractograms of aromatic polyesters containing pendent naphthyl units.

TABLE III
Thermal Properties of Aromatic Polyesters Containing Pendent Naphthyl Units

Entry	Polyester	T_g (°C) ^a	IDT (°C) ^b	T_{10} (°C) ^b
PES-1		259	485	500
PES-2		234	475	490
PES-3	 Ar = IPC/TPC (50/50 mol%)	245	485	495
PES-4		238	420	445
PES-5		209	410	445
PES-6	 Ar = IPC/TPC (50/50 mol%)	216	410	435

^a Measured on DSC at a heating rate of 20°C min⁻¹ in nitrogen.

^b Measured on TGA at a heating rate of 10°C min⁻¹ in nitrogen.

which could have resulted in less efficient packing than unsubstituted bisphenols. The T_g values of polyesters obtained in the present study were comparable with T_g values (232–256°C) of corresponding polyesters containing biphenyl pendent groups.⁵

The thermal stability of polyesters was studied by TGA in nitrogen atmosphere at a heating rate of

10°C min⁻¹. From TG traces, the initial decomposition temperature (IDT) and temperature at 10% weight loss (T_{10}) were determined and the values are reported in Table III. T_{10} values of the polyesters were in the range 435–500°C, indicating their good thermal stability. A comparison of T_{10} values of polyesters indicated that *o*-NABP-based polyesters

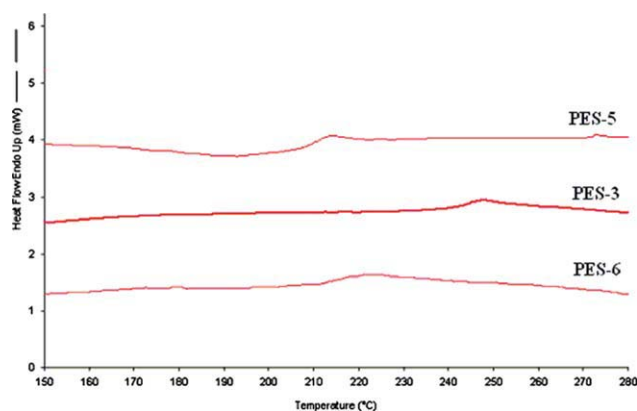


Figure 4 DSC curves of representative polyesters containing pendent naphthyl units. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

were less stable than corresponding polyesters based on NABP. This could be attributed to the presence of methyl substitution on aryl rings in case of *o*-NABP-based polyesters.

CONCLUSIONS

Two bisphenols, viz., 4,4'-[1-(2-naphthalenyl)ethylidene]bisphenol and 4,4'-[1-(2-naphthalenyl)ethylidene]bis-3-methylphenol containing pendent naphthyl group were synthesized. A series of new aromatic polyesters was synthesized by phase-transfer-catalyzed interfacial polycondensation of these bisphenols with aromatic diacid chlorides. Aromatic polyesters containing naphthyl pendent units showed excellent solubility and could be cast into tough, transparent and flexible films from their chloroform solutions. The introduction of pendent naphthyl units in the polymer backbone resulted in enhanced thermal properties when compared with BPA-based polyesters.

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