New Poly(arylene ether)s Containing Phthalimidine Group in the Main Chain

Mohit Aggarwal, Samarendra Maji, Suman Kumar Sen, Barnali Dasgupta, Shyambo Chatterjee, Anindita Ghosh, Susanta Banerjee

Materials Science Centre, Indian Institute of Technology, Kharagpur-721302, India

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ABSTRACT: Five new poly(arylene ether)s containing phthalimidine group in the main chain and pendent trifluoromethyl group have been prepared by the reaction of 4,4'-(bis-4-fluoro-3-trifluoromethylphenyl)benzene (BTF) with bisphenols. Different molar ratios of *N*-phenyl-3,3-bis(4-hydroxyphenyl)phthalimidine (PA) and 4,4'-isopropylidenediphenol (BPA) have been used to generate different copolymers. The polymers obtained by step growth polymerization exhibited weight-average molecular weight upto 134,000 g/mol with a polydispersity index of 2.1–2.4. The homopolymer from BTF and PA showed very high

glass transition temperature of 258°C and outstanding thermal stability upto 536°C for 5% weight loss under nitrogen. The polymers were soluble in a wide range of organic solvents. Transparent thin films of these polymers exhibited tensile strengths upto 65 MPa and elongation at break upto 45% depending on the exact repeat unit structures. © 2009 Wiley Periodicals, Inc. J Appl Polym Sci 112: 1226–1233, 2009

Key words: poly(arylene ether)s; phthalimidine; thermal properties; mechanical properties

INTRODUCTION

Poly(arylene ether)s are one of the important highperformance engineering plastics which consist of aromatic rings and ether linkages. Poly(arylene ether)s are generally prepared by a step growth polymerization of activated diaryl dihalo or dinitro monomers with bisphenoxides.^{1,2} These polymers show excellent hydrolytic, thermal, and dimensional stability along with good mechanical properties.^{3,4} In nucleophillic substitution reaction, the electron withdrawing group at the ortho or para position to the leaving group is necessary to stabilize the negative charge developed during the reaction (Meisenheimer complex). Trifluoromethyl group at ortho position is an example of such activating group. The activation by electron-withdrawing perfluoroalkyl group is expected due to the stabilization of the negative charges at the 2- or 4-position by hyperconjugation and by the negative inductive (-I) effect.⁵ The steric hindrance due to a bulky trifluoromethyl group may also facilitate the formation of a stable Meisenheimer complex with release of steric strain.⁶

Fluorinated polymers are of special interest because of their many interesting set of properties.⁷ Insertion of fluorine or fluorine-containing moieties into the macromolecular chain increases the solubility, thermal stability, flame resistance, glass transition temperature, optical transparency, etc., while decreases crystallinity, dielectric constant, water absorption, and color.^{8–10} Several semifluorinated poly(arylene ether)s prepared by several workers¹¹⁻¹³ showed improved properties in comparison to their nonfluorinated analogues. Semifluorinated polymers are possible candidates for interlayer dielectric materials (ILD) and also considered as good materials for membrane-based separation.14-17 Korshak and coworker reported that the polymer chain having cyclic side cardo group (for example; fluorene, phthalide, or phthalimidine) would increase in both glass transition temperature and thermo-oxidative stability and also good solubility.18

Because of all these interesting properties of fluorinated polymers a considerable attention has been devoted to the preparation of new class of fluorine containing polymers or improvement of the properties of the existing polymers by judicious selection of new monomers. In our previous work, we have reported several new poly(arylene ether)s from BTF and different bisphenols.¹² The highest glass transition temperature 234°C was obtained from the following polymer structure in the series when 4,4'-(9-fluorenylidene)diphenol was used as bisphenol.

Correspondence to: S. Banerjee (susanta@matsc.iitkgp. ernet.in).

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In the present investigation, we report the successful synthesis of a new poly(arylene ether) containing pendent trifluoromethyl groups and phthalimidine in the main chain by the reaction of BTF and PA.



The T_g of the above synthesized homopolymer is manipulated through copolymerization approach by using BPA as one of the comonomer and a structure property corelationship is drawn.

EXPERIMENTAL

General considerations

¹H NMR and ¹³C NMR spectra were recorded on a Bruker 400 MHz NMR instrument [reference 0 ppm with TMS (¹H and ¹³C NMR)] in CDCl₃ solvent. DSC measurements were made on NETZSCH DSC 200PC instrument, at a heating rate of 20 K/min under nitrogen. Glass transition temperatures (T_{o}) were taken as the center of the step transition in the second heating run. Thermogravimetry was measured on NETZSCH TG 209 F1 thermal analyzer instrument at a heating rate of 10 K/min to determine the decomposition temperature under nitrogen. The inherent viscosity of the polymer solutions in DMF was measured at 28°C using Ubbelohde Viscometer. Molecular weights and molecular weight distributions of the polymers were determined by gel permeation chromatography using a Viscotek Gel Permeation Instrument; Model 350 High Temperature Triple Detector Array (HT-TDA) (RI, viscometer and light scattering) instrument. Data analysis was collected using OmniSEC 4.2 software. THF was used as the eluent at a flow rate of 1.0 mL/min and calibration was carried out using narrow polystyrene standard. Stress-strain behavior of the thin polymer films was measured at room temperature using Hounsfield, H10KS-0548 instrument at a strain rate of 5% of sample length. Water absorption test of the polymer films were done by

immersing rectangular pieces of samples (30×10 mm) in distilled water after taking the initial weight at room temperature. Final weight was taken after 48 h and %water absorption was calculated by using a Sartorious balance of sensitivity 10^{-6} g. Dielectric constant of the polymer films was measured by parallel plate capacitor method with a HIOKI 3535 LCR Hi Tester from 100 KHz to 1 MHz at a temperature of 30°C. The relative dielectric constant can be then calculated from

and

$$C_0 = \varepsilon_0 \frac{E}{d}$$

 $\varepsilon = \frac{C_p}{C}$

Where, " C_0 " is the capacitance of a test capacitor measured under vacuum between the plates. " C_p " is the capacitance of the polymer sample, which is placed between the plates of the same capacitor and distance. " ε_0 " is the vacuum permittivity, its value is 8.854187 × 10⁻¹² C² N⁻¹ m⁻². "d" is the thickness of polymer sample, and "E" is the electric field applied.

MATERIALS

1,4-Dibromobenzene (Aldrich); Pd(Ph₃P)₄ (Aldrich); phenolphthalein (Loba Chemie Pvt, India); aniline hydrochloride (Loba Chemie Pvt, India); aniline (E. Merck, India); toluene (E. Merck, India); Na₂CO₃ (E. Merck, India); and dichloromethane (DCM) (E. Merck, India) were used as received. Nmethyl-2-pyrrolidone (NMP) (E. Merck, India) was purified from NaOH and distilled from P₂O₅ before 4,4'-isopropylidenediphenol use. (BPA) (Loba Chemie Pvt, India) was recrystallized from toluene. PA was prepared according to the procedure reported in the literature.¹⁹ The compound BTF was prepared according to our previous work.¹²

Polymerization

Polymerization reactions were carried out in a 50 mL, three-necked round-bottomed flask equipped with a nitrogen inlet, a stir bar, and a Dean-Stark trap fitted with condenser. The flask was charged with equimolar amounts of a BTF and PA (1.24 mmol each), K_2CO_3 (2.98 mmol), NMP (10 mL), and toluene (25 mL). The mixture was then heated to reflux (140–150°C, oil bath temperature) for 2–3 h to remove the water azeotropically with toluene. After removal of the toluene from the Dean-Stark trap, the reaction temperature was increased to 180°C and

maintained for 6 h. After cooling to room temperature, the polymer was precipitated from about 500 mL of methanol containing a few mL of HCl. Fibrous solids were isolated. These products were washed several times in boiling water to remove any inorganic impurities and dried in a vacuum at 120°C for overnight.

Poly(arylene ether) a

The reaction mixture composition of poly(arylene ether) "**a**" is 0.5 g (1.24 mmol) of BTF, 0.49 g (1.24 mmol) of PA, 0.41 g (2.98 mmol) of K₂CO₃, 10 mL of NMP, and 25 mL of toluene.



Anal. cal. for; C₄₆H₂₇O₃F₆N (755.717)_n g/mol: C, 73.11; H, 3.60; F, 15.08; O, 6.35; N, 1.85. Found: C,

72.69, H, 3.55, N, 1.78. FTIR (KBr) (cm⁻¹): 3042 (aromatic C-H stretching), 1714 (C=O symmetric stretching), 1610 (C=C ring stretching band), 1414 (C-F stretching), 1350 (C-O-C asymmetric stretching), 1169, 1052 (C-O-C symmetric stretching), 820 (C-N bending). ¹H NMR (CDCl₃) (400 MHz) δ (ppm): 8.03 (d, I = 7.2 Hz, 1H, H¹⁹); 7.91 (s, 2H, H⁹); 7.71 (*d*, J = 8.4 Hz, 2H, H²³); 7.65 (*s*, 4H, H⁸); 7.6–7.5 $(m, 2H, H^5)$; 7.26-7.18 $(m, 8H, H^{12}, H^{16}, H^{17}, H^{18}, H^{25})$; 7.05–6.94 $(m, 8H, H^4, H^{11}, H^{24})$. ¹³C NMR $(CDCl_3)$ (100 MHz) δ (ppm): 168.92 (C²¹); 156.31 (C^{10}) ; 154.18 (C^3) ; 150.85 (C^{22}) ; 138.51 (C^{20}) ; 136.89 (C^7) ; 135.67 (C^{15}) ; 135.53 (C^{19}) ; 132.85 (C^6) ; 131.51 (C^5) ; 130.48 (C^{13}) ; 130.36 (C^{23}) ; 130.05 (C^{16}) ; 128.69 (C^{17}) ; 128.60 (C^{18}) ; 127.66 (C^{12}) ; 127.47 (C^{8}) ; 125.78 (d, d) $I = 4.8 \text{ Hz}, \text{ C}^9$; 124.62 (C²⁵); 124.49 (C²⁴); 123.92 (q, I = 276.1 Hz, C^{1}); 121.95 (q, J = 30.1 Hz, C^{2}); 119.92 (C^4) ; 118.72 (C^{11}) ; 76.65 (C^{14}) .

Poly(arylene ether) b

The reaction mixture composition of poly(arylene ether) "b" is 0.5 g (1.24 mmol) of BTF, 0.39 g (0.994 mmol) of PA, 0.06 g (0.249 mmol) of BPA, 0.41 g (2.98 mmol) of K_2CO_3 , 10 mL of NMP, and 25 mL of toluene.



Anal. cal. For $C_{43.8}H_{26.4}F_6O_{2.8}N_{0.8}$ (722.68)_n g/mol: C, 72.79; H,3.68; O, 6.19; F, 15.77; N, 1.55. Found: C, 72.34, H, 3.53, N, 1.48. FTIR (KBr) (cm⁻¹): 3041 (aromatic C–H stretching), 2970 (aliphatic C–H stretching), 1776 (C=O asymmetric stretching), 1698 (C=O symmetric stretching), 1604 (C=C ring stretching band), 1449 (C–F stretching), 1166, 1062 (C–O–C symmetric stretching), 820 (C–N bending). Poly(arylene ether) c

The reaction mixture composition of poly(arylene ether) "**c**" is 0.5 g (1.24 mmol) of BTF, 0.29 g (0.746 mmol) of PA, 0.11 g (0.497 mmol) of BPA, 0.41 g (2.98 mmol) of K₂CO₃, 10 mL of NMP, and 25 mL of toluene.



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Anal. cal. For $C_{41.6}H_{25.8}O_{2.6}F_6N_{0.6}$ (689.65)_n g/mol: C, 72.45; H, 3.77; O, 6.03; F, 16.53; N, 1.21. Found: C, 72.21, H, 3.61, N, 1.18. FTIR (KBr) (cm⁻¹): 3040 (aromatic C—H stretching), 2970 (aliphatic C—H stretching), 1712 (C=O symmetric stretching), 1611 (C=C ring stretching band), 1415 (C—F stretching), 1171, 1052 (C—O—C symmetric stretching), 818 (C—N bending). Poly(arylene ether) d

The reaction mixture composition of poly(arylene ether) "d" is 0.5 g (1.24 mmol) of BTF, 0.20 g (0.497 mmol) of PA, 0.17 g (0.746 mmol) of BPA, 0.41 g (2.98 mmol) of K₂CO₃, 10 mL of NMP, and 25 mL of toluene.



Anal. cal. For $C_{39.4}H_{25.2}O_{2.4}F_6N_{0.4}$ (656.59)_n g/mol: C, 72.07; H, 3.87; O, 5.84; F, 17.36; N, 0.85. Found: C, 71.87, H, 3.72, N, 0.82. FTIR (KBr) (cm⁻¹): 3040 (aromatic C—H stretching), 2970, 2935 (aliphatic C—H stretching), 1705 (C=O symmetric stretching), 1606 (C=C ring stretching band), 1482, 1415 (C—F stretching), 1123, 1052 (C—O—C symmetric stretching), 882, 822 (C—N bending). Poly(arylene ether) e

The reaction mixture composition of poly(arylene ether) "e" is 0.5 g (1.24 mmol) of BTF, 0.098 g (0.249 mmol) of PA, 0.23 g (0.994 mmol) of BPA, 0.41 g (2.98 mmol) of K₂CO₃, 10 mL of NMP, and 25 mL of toluene.



Anal. cal. For $C_{37,2}H_{24,6}O_{2.2}F_6N_{0.2}$ (623.59)_n g/mol: C, 71.65; H, 3.97; O, 5.64; F, 18.28; N, 0.45. Found: C, 71.24, H, 3.88, N, 0.42. FTIR (KBr) (cm⁻¹): 3039 (aromatic C—H stretching), 2970, 2935, 2874 (aliphatic C—H stretching), 1708 (C=O symmetric stretching), 1610 (C=C ring stretching band), 1480, 1418 (C—F stretching), 1121, 1052 (C—O—C symmetric stretching), 883, 822 (C—N bending).

RESULTS AND DISCUSSION

Synthesis of poly(arylene ether)s

Polymerizations of the BTF with stoichiometric amounts of different bisphenols (PA and BPA) were carried out in the presence of excess of K_2CO_3 in

NMP as solvent according to the reported protocol.^{11,12} Toluene was used for azeotropic removal of water. The detailed structures of the polymers prepared are shown in Scheme 1. During the initial stage of the polymerization, the reaction temperature was maintained at 140-150°C, and the water generated was effectively removed through a Dean-Stark trap. Upon completion of bisphenoxide formation, the reaction temperature was raised to 180°C for effective nucleophilic displacement reaction. High molar mass polymers were obtained within 6 h as judged by the dramatic increase of the viscosity of the reaction medium in all the cases. Although a viscosity versus time plot is not provided, we found that there is an increase in viscosity as the polymerization time is raised to 6 h and after that no

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Scheme 1 Synthesis of the poly(arylene ether).

appreciable change in viscosity was observed. In fact, we continued few of the reactions up to 8 h but did not find any deterioration in the viscosity values.¹² The resulting polymers were precipitated in a large excess of methanol containing a few mL of HCl for better removal of the excess base present in the system. Fibrous solids were isolated. These products were washed several times with hot water for the removal of inorganic impurities present in the system, and dried in a vacuum at 120°C for overnight.

About 800 mg of each of the dry polymers were dissolved in 10 mL of dichloromethane and poured in a flat bottom Petri dishes. The solvent was evaporated at the control rate at room temperature for 24 h. The films were kept overnight at 100°C, under vacuum to ensure the complete removal of any residual solvent in the film. The transparent films were obtained in all cases.

Polymer solubility and molecular weight

The solubility of poly(arylene ether)s were investigated in various organic solvents and reported in Table I. The polymer "**a**" and "**b**" containing high molar ratios of PA showed good solubility in polar aprotic solvents like NMP, DMF, and in THF, but were insoluble in nonpolar solvent like toluene at room temperature. This may be due to the presence of polar PA moiety in higher molar ratios in these polymers.

The inherent viscosity of the synthesized poly(arylene ether)s were evaluated in DMF having concentration 0.5% (w/v) ranging from 0.51-0.94 dL/g. These values along with GPC results of the polymers shown in Table II. GPC results indicates the formation of high molar masses.

Polymer characterization

Structures of the synthesized poly(arylene ether)s were confirmed from FTIR and NMR spectroscopy. These characterization methods as well as elemental analysis did not indicate a degradation of the trifluoromethyl groups.¹² FTIR spectra of the polymers showed similar pattern absorption band at 1776–1765 cm⁻¹ (C=O asymmetric stretching), 1714–1698 cm⁻¹ (C=O symmetric stretching), 1171–1052 cm⁻¹ (C=O symmetric stretching) showed the formation of ether bonds.¹² Stretching band at 2970–2935 cm⁻¹ corresponding to aliphatic (C–H stretching) was absent in polymer "**a**" but was observed in copolymers (**b–e**) with increase in BPA content in the respective copolymers. The proton attached to

 TABLE I

 Solubility of Semi Fluorinated Poly(arylene ether)s

Polymer	NMP	DMF	DMSO	THF	CHCl ₃	Acetone	Toluene
а	+	+	_	+	+	_	_
b	+	+	—	+	+	-	-
с	+	+	—	+	+	-	+
d	+	+	—	+	+	-	+
e	+	+	_	+	+	_	+

+, soluble at room temperature; -, insoluble at room temperature.

Properties of Poly(arylene ether)s					
Polymer	η_{inh}	M _n	M_w	PDI	Film quality
а	0.86	65,000	134,000	2.1	Clear, flexible
b	0.94	56,000	135,000	2.4	Clear, flexible
с	0.51	39,000	86,000	2.2	Clear, flexible
d	0.70	53,000	112,000	2.1	Clear, flexible
e	0.71	54,000	114,000	2.1	Clear, flexible

TABLE II

 η_{inh} , inherent viscosity in DMF at 28°C; M_n and M_w are the number average and weight average molecular weight, respectively; PDI, poly dispersity index.

the carbon marked 19 appeared at 8.03 ppm, which is the most deshielded one. This is due to the presence of α -carbonyl group. The intensity of the corresponding peak decreases by lowering the composition of PA monomer in polymer "a"-"e." The proton attached to carbon 31 appeared at 1.66 ppm indicate --CH₃ protons of BPA, which was not found in the homopolymer. As the BPA content increases in polymers "b''-"e," the intensity of the -CH₃ protons increases accordingly. The integrated peak area under these peaks exactly matches with the molar ratio of BPA in the respective polymers. Most down field shift of carbonyl carbon peak in carbon NMR spectra at 168.92 ppm corresponds to carbon marked 21. Representative ¹H NMR spectra of the polymer "a" is shown in Figure 1. The exact analytical data for all these polymers are presented in the experimental section.

Thermal properties

TGA analysis

The synthesized poly(arylene ether)s showed very good thermal stability in nitrogen environment. The TGA thermograms of these polymers are shown in Figure 2. Depending on the monomer composition in copolymer, these polymers showed 5% weight loss in the temperature range of 516–536°C and 10% weight loss in between 535 and 556°C under nitrogen as given in the Table III. These poly(arylene ether)s have comparable thermal stability with the similar polymer structures reported earlier.¹² Polymer "a" has highest thermal stability when compared with the other copolymers because it does not have any aliphatic -CH3 group in the polymer chain.¹² The poly(arylene ether)s bearing phthalimidine cardo groups were more thermally stable in comparison with the similar structure of poly(arylene ether)s without cardo moiety. This difference is probably due to the structural differences as cyclic amide group has better thermal resistance than the normal aliphatic group. The amount of carbonized residue of poly(arylene ether)s in nitrogen was about 70 wt % at 700°C, which indicates that the polymers are highly thermally stable.

DSC measurements

None of the synthesized polymers exhibited crystallization or melt transition in DSC. The polymers



Figure 1 ¹H NMR (400 MHz, CDCl₃) of polymer "a".



Figure 2 TGA thermogram of the poly(arylene ether)s.

showed glass transition temperature characteristic of amorphous or glassy nature. The glass transition temperatures of the polymers are summarized in Table III. DSC plots of the polymers are shown in Figure 3. Glass transition temperature of the polymers depend on several factors like rigidity of polymer backbone, polymer symmetry, nature of side groups, and intermolecular forces.^{7,11,12} Glass transition temperatures of the poly(arylene ether)s also depend significantly on the bisphenols from which they are synthesized and on the amount of molar ratio of the particular monomer used. The polymer containing high percentage of PA showed highest T_{g} value than the other polymers. This can be explained on the basis of the rigid cardo structure of PA. The homopolymer showed the highest glass transition temperature than the similar cardo polymers reported previously.¹² This can be attributed to the more polar nature of the cardo group arising from the imide moiety in the PA increasing the cohesive energy density of the polymer and as a result higher T_g is observed. The T_g of the copolymers decreases as the BPA content increases. This gives a fair scope to manipulate the T_g of the homopolymer for a suitable application.

TABLE III Thermal Properties of Poly(arylene ether)s

		T_d		
Polymer	DSC $T_g/^\circ C$	5 % wt loss	10 % wt loss	Char yield/700°C
а	258	536	556	70
b	253	536	556	73
с	236	516	535	68
d	227	520	535	69
e	213	525	539	68



Figure 3 DSC curves of the poly(arylene ether)s.

Mechanical properties and rheology

The polymer films showed good mechanical properties, the results are summarized in Table IV. Polymers containing high BPA content (**d** and **e**) exhibited ductile mechanical behavior, with an elongation at break up to 45%. In contrast, the analogous poly(arylene ether)s (**a**, **b**, and **c**) containing higher amount of the PA exhibited relatively high tensile strength but somewhat brittle mechanical behavior with an elongation at break below 15%. The stress and strain curve for polymer "**e**" is shown in Figure 4. Melt viscosity of the synthesized polymers showed non-Newtonian (pseudoplastic) behavior, and the viscosity decreases with increase in the applied shear rate.

Water absorption and dielectric behavior

Water absorption is a great concern when the polymers are used in electronic devices as dielectric insulating material. The absorbed water in the polymer structure affects their dielectric performance. Hence, the water absorption data is an essential parameter

TABLE IV					
Mechanical and Dielectric Properties					
of the Polv(arvlene ether)s					

				Water	
	Tensile		Young's	absorption	Dielectic
	Strength I	Elongatior	n modulus	rate/%	constant/1
Polym	ner (MPa) a	nt break/%	6 (GPa)	(after 48 h)	MHz
a	65 (±4)	11 (±2)	1.67 (±0.02)	0.23	2.65
b	65 (±4)	15 (±3)	1.64 (±0.025)	0.34	2.59
с	63 (±3.5)	10 (±2)	1.56 (±0.022)	0.29	2.53
d	56 (±5)	29 (±4)	1.45 (±0.025)	0.28	2.55
e	59 (±6.5)	45 (±5)	1.38 (±0.024)	0.30	2.49



Figure 4 Stress and strain curve for polymer "e".

for the materials designed for such type of application. The water absorption value for these polymers lies between 0.23 and 0.34% after 48 h. These values are comparable with semifluorinated poly(arylene ether)s. The exact vales for different poly(arylene ether)s are given in the Table IV.

The dielectric constant of these polymer films was measured by using a 1 MHz capacitance meter at 30°C, RH 45%. The capacitance values of polymer thin films were evaluated by keeping the film in between two copper electrodes. The capacitance values were finally converted to the dielectric constant values using the equation given in the experimental section. The dielectric constant values of the polymer films are presented in Table IV. In general, these polymers exhibited low dielectric constant. The dielectric constant values of the synthesized polymers are comparable with analogous semifluorinated poly (arylene ether)s reported earlier.¹²

CONCLUSIONS

Five new trifluoromethyl-activated novel poly(arylene ether) have been successfully synthesized. The synthesized polymers are soluble in various organic solvents. The resulting homopolymer showed glass transition temperature as high as 258°C. These polymers exhibited high thermal stability ranging from 516 to 536°C for 5% weight loss. All synthesized poly (arylene ether)s showed the decrease in viscosity with increase in applied shear rate indicating non-Newtonian (pseudoplastic) behavior of the polymer melts. The polymer showed water absorption as low as 0.23% and dielectric constant of 2.49 at 1 MHz.

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