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ORIGINAL PAPER

Synthesis and characterization of new poly(ether–ester–imide)s as a generation of soluble and thermally stable polymers

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Abstract A new-type of dicarboxylic acid **6** was synthesized from the reaction of 1,4-bis[4-aminophenoxy]butane **4** with trimellitic anhydride **5** in a solution of glacial acetic acid/pyridine (Py) at refluxing temperature. 1,4-Bis[4-nitrophenoxy]butane **3** was prepared by reaction of 4-nitrophenol **1** with 1,4-dibromo butane **2** in *N*,*N*-dimethylformamide (DMF) solution. Then dinitro **3** was reduced to 1,4-bis[4-aminophenoxy]butane **4** by using 10% Pd–C, ethanol, and hydrazine monohydrate. So six new thermally stable and organosoluble poly(ether–ester–imide)s (PEEIs) **8a–f** with good inherent viscosities were synthesized by the direct polycondensation reaction of new 1,4-bis[4-(trimellitimido)phenoxy]butane **6** with several aromatic diols **7a–f** through direct polycondensation using tosyl chloride (TsCl)/pyridine (Py)/DMF system as condensing agent. The resulted polymers were fully characterized by means of FTIR and ^{1}H NMR spectroscopy, elemental analyses, inherent viscosity, solubility tests, differential scanning calorimeter (DSC), thermogravimetric analysis (TGA), and derivative of thermogravimetric (DTG).

Keywords Poly(ether–ester–imide)s · Thermal properties · Glass transition · Direct polycondensation

Introduction

High-performance polymeric materials are currently receiving considerable attention for their potential applications in advanced technologies demands. Aromatic polyimides are well known high-performance polymers that show excellent thermal,

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mechanical, and electrical properties [1, 2]. However, applications may be rather limited due to their high softening or melting temperatures and their insoluble nature in most organic solvents [3].

Modification of high-performance materials by increasing the solubility and lowering the transition temperatures while maintaining thermal stability are of particular interest. Copolycondensation is one of the possible ways for medication of polymer properties. Thus, for the processing of polyimides many copolyimides, such as poly(amide–imide)s, poly(ester–imide)s, and other copolymers have been prepared [4–9]. Aromatic polymers that contain aryl ether linkages generally have lower glass transition temperatures, greater chain flexibility, and tractability in compare to their corresponding polymers these groups in the chain [10–12]. The lower glass transition temperatures and also improved solubility are attributed to the flexible linkages that provide a polymer chain with a lower energy of internal rotation [13].

We consider here the preparation and basic characterization of thermally stable poly(ether–ester–imide)s (PEEIs) **8a–f** from the direct polycondensation reaction of 1,4-bis[4-(trimellitimido)phenoxy]butane **6** as a new diacid monomer with six aromatic diols such as 4,4′-sulfonyldiphenol **7a**, bisphenol A **7b**, 2,7-dihydroxynaphthalene **7c**, 1,4-dihydroxyanthraquinone **7d**, hydroquinone **7e**, and resorcinol **7f** by direct polycondensation in a tosyl chloride (TsCl)/pyridine (Py)/*N*,*N*-dimethylformamide (DMF) system.

Experimental

Materials

All chemicals were purchased from Fluka Chemical Co. (Switzerland), Aldrich Chemical Co. (Milwaukee), Merck Chemical Co. (Germany), and Acros Organics N.V./S.A. (Belgium).

Instruments

^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker 300 MHz instrument (Germany). Fourier transform infrared (FTIR) spectra were recorded on Galaxy series FTIR 5000 spectrophotometer (England). Spectra of solid were performed by using KBr pellets. Vibration transition frequencies were reported in wave number (cm^{-1}). Band intensities were assigned as weak (w), medium (m), shoulder (sh), strong (s), and broad (br). Inherent viscosities were measured by a standard procedure by using a Technico® Merk Viscometer. Thermal Gravimetric Analysis (TGA) data for polymers were taken on a Mettler TA4000 System under N_2 atmosphere at rate of 10 °C/min. Differential scanning calorimeter (DSC) was conducted with a DSC Metller 110 (Switzerland) at a heating rate of 10 °C/min in a nitrogen atmosphere. Elemental analyses were performed by Vario EL equipment.

Monomer synthesis

1,4-Bis[4-nitrophenoxy]butane **3**

4-Nitrophenol **1** (6.00 g, 43.11 mmol) and dry K_2CO_3 (2.97 g, 21.55 mmol) in 30 mL dimethyl formamide (DMF) were added in a 100 mL round-bottomed flask. Then a solution of 1,4-dibromo butane **2** (4.46 g, 20.67 mmol) in 5 mL dry dimethyl formamide was added drop wisely to reaction mixture. The reaction mixture was heated for 6 h at 120 °C, then was cooled and poured onto crushed ice. The precipitated white product was collected by filtration, dissolved in CH_2Cl_2 and washed successively with NaOH (2 M), HCl (1 M), and water. Then, CH_2Cl_2 solution was dried over Na_2SO_4 and concentrated in vacuum and product was recrystallized from ethanol, affording 4.59 g (65.4%) of white solid **3**, m.p.: 140–142 °C, FTIR (KBr, cm^{-1}): 3058 (w), 1609 (s), 1534 (s), 1411 (m), 1348 (s), 1181 (s), 1111 (s), 987 (s), 852 (s), 756 (m), 702 (s), 543 (m). ^1H NMR (300 MHz, DMSO-d_6, TMS): δ; 8.17–8.20 (t, 4H), 7.12–7.15 (t, 4H), 4.19 (s, 4H), 1.92 (s, 4H) ppm. ^{13}C NMR (300 MHz, DMSO-d_6): δ; 164.37, 142.78, 126.31, 115.41, 68.10, 26.17 ppm. Elemental analysis: calculated for $C_{16}H_{16}N_2O_6$: C, 57.83; H, 4.85; N, 8.43; found: C, 57.68; H, 4.81; N, 8.33.

1,4-Bis[4-aminophenoxy]butane **4**

1,4-Bis[4-aminophenoxy]butane **4** (1.00 g, 3.67 mmol) and 0.1 g of 10% Pd–C, and 20 mL of ethanol were introduced into a 100-mL round-bottomed flask to which 7 mL of hydrazine monohydrate was added drop wisely over a period of 1 h at 85 °C. After the complete addition, the reaction was continued at reflex temperature for another 5 h. Then, the mixture was filtered to remove the Pd–C and the filtrate was poured into water and dried to afford 0.71 g (87%), m.p.: 152–154 °C. FTIR (KBr, cm^{-1}): 3322 (m), 3214 (m), 3032 (w), 2906 (w), 1604 (m), 1591 (s), 1504 (s), 1438 (m), 1307 (m), 1282 (m), 1172 (s), 1107 (m), 985 (w), 829 (m), 511 (m). ^1H NMR (300 MHz, DMSO-d_6, TMS): δ; 6.62–6.65 (d, 4H), 6.47.650 (d, 4H), 4.58 (s, 4H), 3.85 (s, 4H), 1.76 (s, 4H) ppm. ^{13}C NMR (300 MHz, DMSO-d_6): δ; 150.40, 142.72, 126.33, 115.42–115.81, 68.11, 26.14 ppm. Elemental analysis: calculated for $C_{16}H_{20}N_2O_2$: C, 70.56; H, 7.40; N, 10.29; found: C, 70.28; H, 7.36; N, 10.21.

1,4-Bis[4-(trimellitimido)phenoxy]butane **6**

Into a 100-mL, round-bottom flask, 0.204 g (0.75 mmol) of 1,4-bis[4-aminophenoxy]butane **4**, 0.29 g (1.5 mmol) of trimellitic anhydride **5**, 20 mL of a mixture of acetic acid and pyridine (3:2), and a stirring bar were placed. The mixture was stirred at room temperature overnight and then refluxed for 4 h. After that time the solvent was removed under reduced pressure, and the residue was dissolved in 100 mL of cold water; then, 5 mL of concentrated HCl was added. The solution was stirred until a brown precipitate formed, and then the precipitate was filtered off and dried in vacuum to give 0.414 g (89%) of dicarboxylic acid **6**, m.p.: >320 °C, FTIR (KBr, cm^{-1}): 2500–3500 (m, br), 1782 (w), 1720 (s, br), 1610 (w), 1512 (m), 1388 (s, br), 1292 (w), 1248 (m), 1176 (w), 1099 (w), 1055 (w), 976 (w), 823 (m), 721 (s), 524 (m). ^{1}H NMR (300 MHz, DMSO-d_6, TMS): δ; 12.79 (s, br, 2H), 8.39–8.42 (t, 2H), 8.29–8.39 (t, 2H), 8.04–8.07 (t, 2H), 7.34–7.37 (q, 4H), 7.06–7.11 (q, 4H), 4.12 (s, 4H), 1.88–1.93 (s, 4H) ppm. Elemental analysis: calculated for $C_{34}H_{24}N_2O_{10}$: C, 65.81; H, 3.90; N, 4.51; found: C, 65.71; H, 3.88; N, 4.50.

Polymer synthesis

The PEEIs were prepared by the following procedure. In order to synthesize polymer **8e**, a solution of pyridine (0.15 mL, 1.9 mmol) with TsCl (0.09 g, 0.460 mmol) after 30 min stirring at room temperature, was treated with DMF (0.15 mL, 1.86 mmol) for 30 min and the mixture was added drop wisely to a solution of diacid **6** (0.115 g, 0.186 mmol) in pyridine (0.15 mL). The mixture was maintained at room temperature for 20 min and then a solution of hydroquinone **7e** (0.02 g, 0.186 mmol) in pyridine (0.15 mL) was added drop wisely at room temperature then the whole solution was stirred at 120 °C for 2 h. As the reaction proceeded, the solution became viscous. Then the viscous liquid was precipitated in 50 mL of methanol to yield 0.12 g (94%) of the polymer **8e**.

Result and discussion

Monomer synthesis

1,4-Bis[4-aminophenoxy]butane **4** was synthesized by using a two-step reaction. At first 1,4-bis[4-nitrophenoxy]butane **3** was prepared by the reaction of two equmolars 4-nitrophenol **1** and one equimolar 1,4-dibromo butane **2**. Then dinitro compound **3** was reduced by using 10% Pd–C, ethanol, and hydrazine monohydrate (Scheme 1).

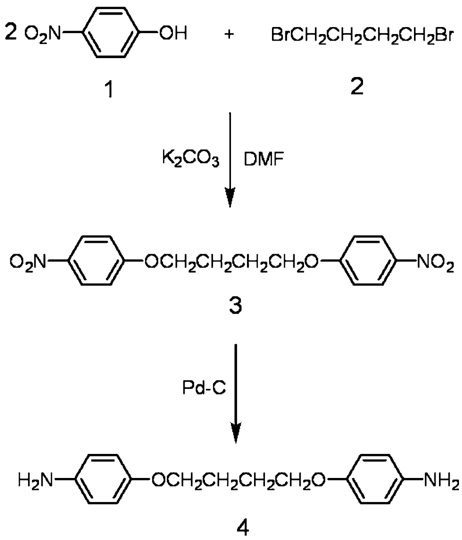
The chemical structure and purity of dinitro compound **3** were proved by elemental analysis, ^{1}H NMR, and FTIR spectroscopy and diamine compound **4** were proved with elemental analysis, FTIR, ^{1}H NMR, and ^{13}C NMR spectroscopy.

In elemental analyses, the measured results of these compounds closely corresponded to the calculated ones, demonstrating that the expected compounds obtained. The FTIR spectrum of diamine **4** showed two peaks at 3321 and 3212 cm^{-1}, which were assigned to the NH_2 groups. Also, the absorptions found at 1062–1296 cm^{-1} were assigned to the vibration of the C–O band.

^{1}H NMR spectrum of diamine **4** showed peaks as doublet of doublet at 6.62–6.65 and 6.47–6.50 were assigned to the H(a) and H(b) related to aromatic protons. Also, a broad singlet peak at 3.85 ppm which was assigned to the H(d) protons of the NH_2 groups in the compound. The peak at 4.58 ppm which was assigned to H(c) protons of methylene groups and the peak at 1.76 ppm which was assigned to H(e) protons of two similar methylene groups (CH_2–O) (Fig. 1).

As shown in Scheme 2, the dicarboxylic acid **6** was obtained by the condensation of the appropriate diamine **4** with two mole equivalents of trimellitic anhydride **5** in refluxing mixture of glacial acetic acid and pyridine. The condensation reaction

Scheme 1 Synthesis of diamine **4**



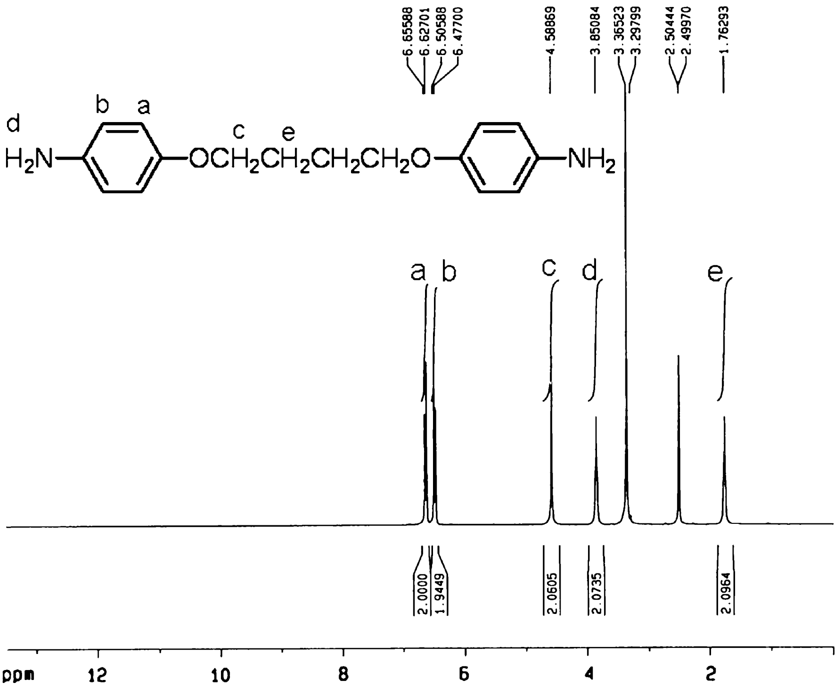
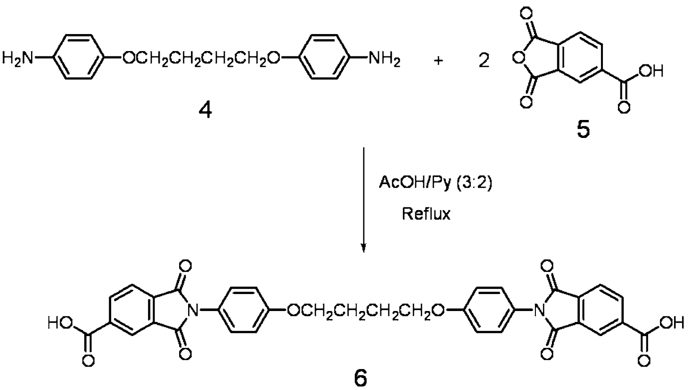


Fig. 1 ^1H NMR spectrum of diamine **4**

between the amines and anhydride groups, as well as the subsequent cyclodehydration reaction was carried out in the heterogeneous solution. The chemical structure and purity of dicarboxylic acid **6** were proved with elemental analysis,



Scheme 2 Synthesis of diacid **6**

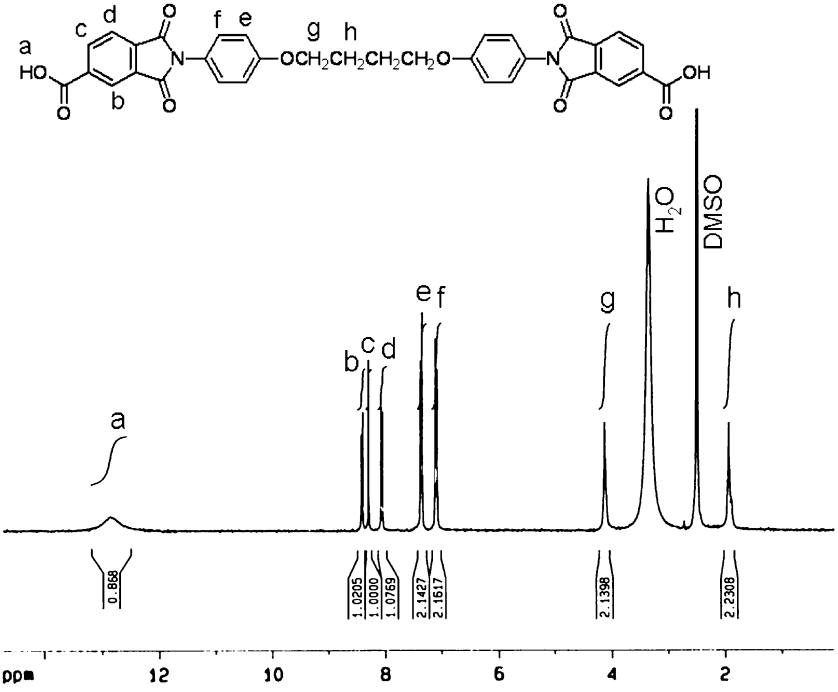


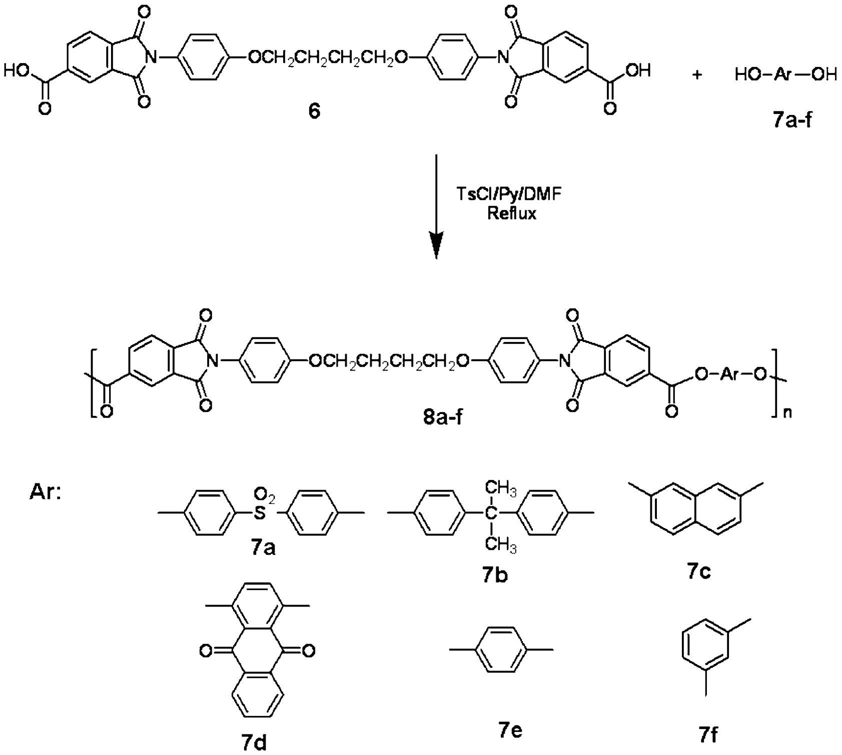
Fig. 2 ^{1}H NMR spectrum of diacid **6**

FTIR, ^{1}H NMR spectroscopy. The FTIR spectrum of the dicarboxylic acid **6** showed absorption bands around 2500–3500 cm^{-1} (acidic H's), 1782 cm^{-1} (asymmetric imide C=O stretching), 1720 (symmetric imide C=O stretching and acid C=O stretching), and 1388 cm^{-1} (imide ring vibration) confirming the presence of imide ring and carboxylic acid groups in the structure.

The ^1H NMR spectroscopic data of the dicarboxylic acid **6** are in the range of 7.04–8.42 ppm, which protons of the trimellitoyl groups create resonance signals around downfield regions (above 8 ppm). Peaks related to diamine moiety in the center of this compound were appeared in 7.06–7.37 ppm regions (below 8 ppm). Also a broad singlet peak at 12.79 ppm which was assigned to the H(a) proton of the acidic groups in this compound (Fig. 2).

Polymer synthesis

In this work, the Vilsmeier adduct was used for the polycondensation of aromatic diacid and aromatic diols in the following way (Scheme 3): TsCl was dissolved in Py to yield sulfonium salt and after a certain period of time the solution was treated with DMF for 30 min. The reaction mixture was added to a solution of diacid in Py to form activated acid. After 30 min a solution of diol in Py was added and the whole solution was maintained at room temperature and then at an elevated temperature for a period of time. For direct polycondensation of monomer **6** the reported optimized conditions [14] were selected and polymerization was carried out under these conditions (Scheme 3).



Scheme 3 Synthesis rout of PEEIs **8a–f**

All the polycondensations proceeded readily in a homogeneous solution. Tough and stringy precipitates formed when the viscous polymer solutions were trickled into the stirring methanol.

The syntheses and some physical properties of these new PEEIs **8a–f** are given in Table 1. All the polymers were obtained in high yields (83–94%), and the inherent viscosities were 0.49–0.72 dL/g which were measured in DMF solutions. Also the resulting polymers showed a range of color between red and brown.

Polymer characterization

The structure of polymers was confirmed as PEEIs by means of FTIR spectroscopy and elemental analyses while the representative FTIR spectrum of PEEI **8f** was shown in Fig. 3. The polymer showed the C=O asymmetric stretching of imide at

Table 1 Synthesis and some physical properties of PEEIs **8a–f**

Diol	Polymer	Yield (%)	η_{inh} (dL/g)[a]	Color
7a	**8a**	94	0.69	White
7b	**8b**	92	0.72	White
7c	**8c**	83	0.66	Gray
7d	**8d**	91	0.51	Yellow
7e	**8e**	94	0.49	Gray
7f	**8f**	88	0.67	White

[a] Measured at a concentration of 0.5 g/dL in DMF at 25 °C

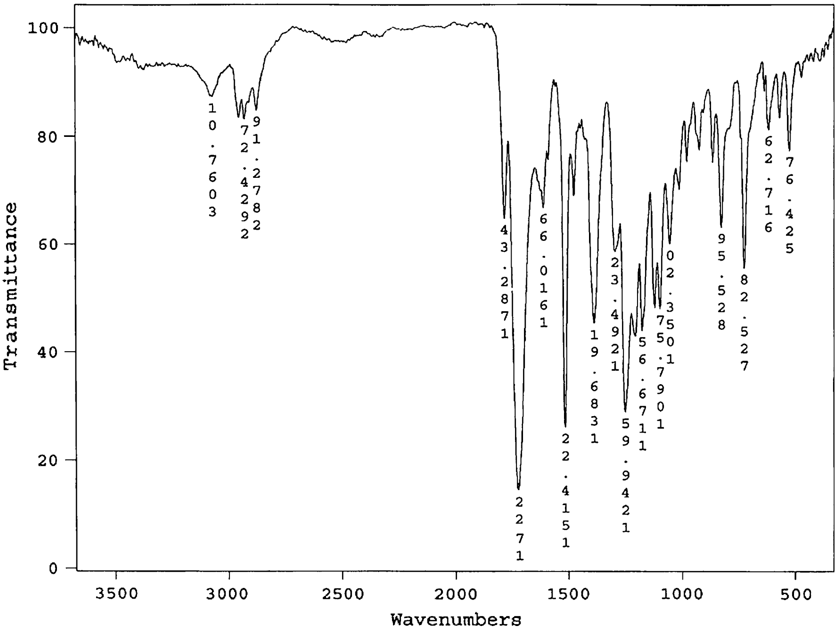


Fig. 3 FTIR spectrum of PEEI **8f**

1782 cm^{-1}, the C=O symmetric stretching of imide and ester groups at 1722 cm^{-1}, C–N stretching at 1388 cm^{-1}, C–O–C stretching at 1055–1248 cm^{-1}. All of the polymers exhibited strong absorptions around 1388 and 723 cm^{-1}, which show the presence of the heterocyclic imide groups. FTIR spectroscopy data for all of PEEIs **8a–f** are listed in Table 2.

The ^{1}H NMR spectrum of PEEI **8a** showed peaks that confirmed its chemical structure (Fig. 4). The aromatic protons related to polymer backbone appeared in

Table 2 FTIR characterization of PEEIs **8a–f**

Polymer	Spectra data
8a	FTIR peaks (cm^{-1}): 3070 (w), 2914 (w), 1780 (w), 1722 (s), 1610 (m), 1514 (s), 1388 (s), 1292 (m), 1248 (m), 1174 (w), 1094 (w), 1055 (w), 823 (w), 725 (s), 524 (m)
8b	FTIR peaks (cm^{-1}): 3072 (w), 2916 (w), 1780 (w), 1722 (s), 1610 (w), 1512 (s), 1388 (m), 1248 (m), 1122 (w), 1055 (w), 825 (m), 725 (s), 526 (w)
8c	FTIR peaks (cm^{-1}): 3067 (w), 2924 (w), 1782 (m), 1722 (s, br), 1610 (m), 1514 (s), 1386 (s), 1249 (s), 1176 (w), 1097 (w), 1053 (w), 825 (w), 725 (s), 617 (w), 524 (w)
8d	FTIR peaks (cm^{-1}): 3064 (w), 2968 (m), 1776 (w), 1716 (s), 1678 (m), 1639 (w), 1591 (m), 1464 (m), 1383 (s), 1321 (w), 1201 (m), 1113 (w), 723 (w)
8e	FTIR peaks (cm^{-1}): 3072 (w), 2904 (w), 1782 (w), 1720 (s, br), 1610 (m), 1512 (s), 1388 (s), 1292 (m), 1248 (m), 1176 (w), 1099 (w), 1055 (w), 823 (w), 721 (s), 524 (m)
8f	FTIR peaks (cm^{-1}): 3072 (w), 2926 (m), 1782 (m), 1722 (s), 1610 (w), 1514 (s), 1388 (s), 1248 (m), 1122 (w), 1055 (w), 825 (m), 723 (s), 526 (w)

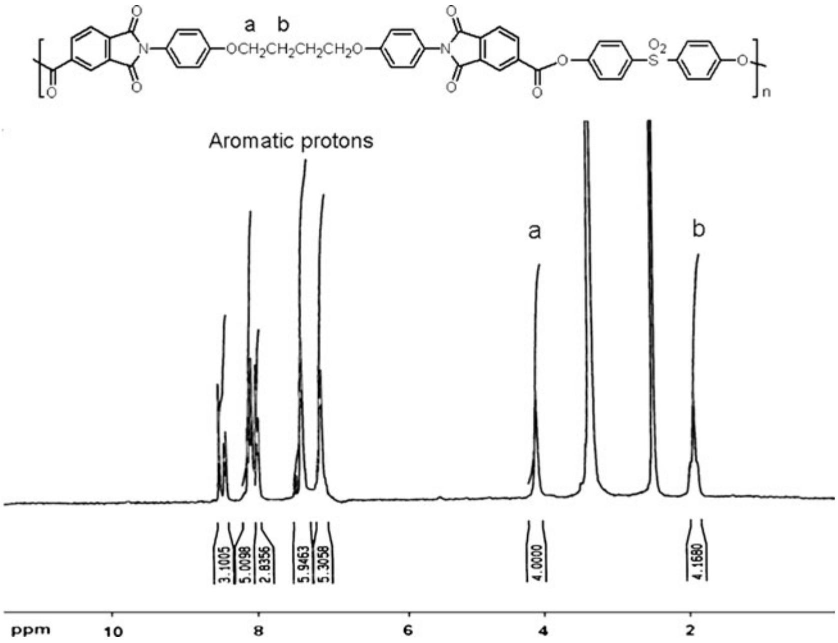


Fig. 4 ^{1}H NMR spectrum of PEEI **8a**

Table 3 Elemental analysis of PEEIs **8a–f**

Polymer	Formula		C%	H%	N%
8a	$C_{46}H_{30}N_2O_{12}S$	Calcd	66.18	3.62	3.36
	$(834.8)_n$	Found	66.06	3.63	3.26
8b	$C_{49}H_{36}N_2O_{10}$	Calcd	72.41	4.46	3.45
	$(812.82)_n$	Found	72.01	4.37	3.34
8c	$C_{44}H_{28}N_2O_{10}$	Calcd	70.96	3.79	3.76
	$(744.7)_n$	Found	70.18	3.71	4.70
8d	$C_{48}H_{28}N_2O_{12}$	Calcd	69.90	3.42	3.40
	$(824.74)_n$	Found	68.84	3.36	3.33
8e	$C_{40}H_{26}N_2O_{10}$	Calcd	69.16	3.77	4.03
	$(694.64)_n$	Found	68.52	3.70	4.01
8f	$C_{40}H_{26}N_2O_{10}$	Calcd	69.16	3.77	4.03
	$(694.64)_n$	Found	68.88	3.75	3.98

the region of 7.08–8.51 ppm. The peaks in the region of 4.12 and 1.93 ppm are assigned for methylene groups in the polymer chain. Also the elemental analyses of the resulting PEEIs **8a–f** were in good agreement with the calculated values for the proposed structure (Table 3).

Solubility of the PEEIs

The solubility of PEEIs **8a–f** was investigated as 0.01 g of polymeric sample in 2 mL of solvent. These PEEIs have good solubility in aprotic organic solvents. Remarkably, all of these PEEIs were easily soluble at room temperature in aprotic polar solvents such as *N*-methyl-2-pyrrolidone (NMP), *N*,*N*-dimethylacetamide (DMAc), *N*,*N*-dimethylformamide (DMF), and insoluble in solvents such as chloroform, ethanol, and methanol (Table 4).

Table 4 Solubility of PEEIs **8a–f**

Solvent	**8a**	**8b**	**8c**	**8d**	**8e**	**8f**
DMAc	+	+	+	+	+	+
DMSO	+	+	+	+	+	+
DMF	+	+	+	+	+	+
NMP	+	+	+	+	+	+
MeOH	–	–	–	–	–	–
EtOH	–	–	–	–	–	–
$CHCl_3$	–	–	–	–	–	–
CH_2Cl_2	–	–	–	–	–	–
H_2O	–	–	–	–	–	–

+ Soluble at room temperature, – insoluble at room temperature

Thermal properties

TGA and derivative of thermogravimetric (DTG) analysis at a rate of 10 °C/min in a nitrogen atmosphere were utilized to examine the thermal properties of these PEEIs, and the obtained results are summarized in Table 5. Figure 5 shows TGA curve of PEEIs **8a–f**.

The thermal stability of the polymers was studied on the basis of 5 and 10% weight losses (T5 and T10, respectively) of the polymers and the residue at 800 °C (char yield). The results revealed that the PEEIs were thermally stable up to 380 °C. TGA data showed that the resulting polymers were thermally stable. Thermal stability of polymer **8a** was higher than the other polymers, because this polymer has a polar sulfonyl group in the main chain. The presence of the *para*-phenylene groups in the polymer **8e** led to the better thermal stability in comparison with

Table 5 Thermal behavior of PEEIs **8a–f**

Polymer	T_g^a	T_5 (°C)[b]	T_{10} (°C)[b]	Char yield[c]	LOI[d]
8a	194	390	410	45.98	36
8b	182	380	390	38.61	33
8c	–[e]	370	410	42.96	35
8d	216	390	400	42.35	35
8e	–	380	390	43.23	35
8f	–	350	400	39.65	33

[a] Glass transition temperature was recorded at a heating rate of 10 °C/min in a nitrogen atmosphere

[b] Temperature at which 5 or 10% weight loss was recorded by TGA at a heating rate of 10 °C/min under N_2

[c] Weight percentage of material left after TGA analysis at a maximum temperature of 800 °C under N_2

[d] Limiting Oxygen Index

[e] Not determined

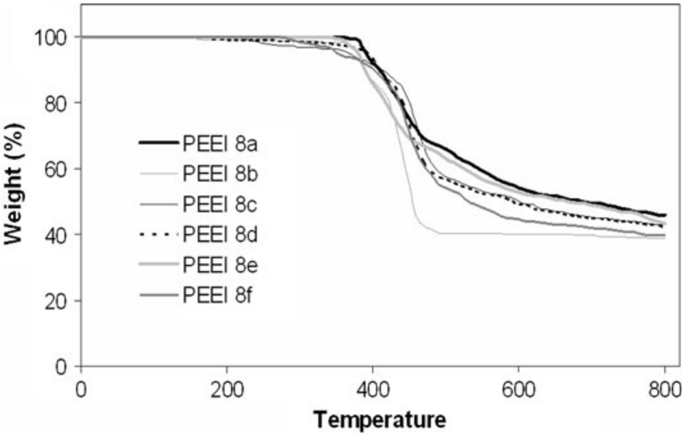


Fig. 5 TGA curves of PEEIs **8a–f**

polymer **8f** which contain *meta*-phenylene. As shown in Fig. 5 the thermally properties of polymer **8b** containing aliphatic group is less than the other polymers. Also the DSC analyses for PEEIs showed T_g around 182–216 °C (Table 5).

The char yield can be a decisive factor to estimate the limited oxygen index (LOI) of polymers according to the Van Krevelen–Hoftyzer's equation [15]:

$$\mathrm{LOI} = 17.5 + 0.4\mathrm{CR}$$

where CR is the char yield.

PEEIs **8a–f** had LOI values around 35, which were calculated from their char yield. On the basis of the LOI values, such macromolecules can be classified as self-extinguishing polymers.

Conclusion

In this article, we have successfully synthesized new dicarboxylic acid **6** containing ether and methylene groups. A series of new thermally stable PEEIs **8a–f** were prepared from dicarboxylic acid **6** with various aromatic dioles **7a–f** by direct polycondensation reaction in a system of tosyl chloride (TsCl), pyridine (Py), and *N*,*N*-dimethylformamide (DMF). The presented results also clearly demonstrate that incorporating the imide group into the polymer main chain as well as combination of the wholly aromatic backbone and several functional groups remarkably enhanced the thermal stability of the new polymers. These polymers are expected to have higher solubility due to the presence of ether and alkyl groups in the polymer chain. These properties could make these PEEIs attractive for practical applications such as processable high-performance engineering plastics.

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