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ORIGINAL PAPER

Polyimides based on furanic diamines and aromatic dianhydrides: synthesis, characterization and properties

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Abstract Novel polyimides containing furan moieties were prepared from the resulting furanic diamine monomers with various aromatic dianhydrides including 1,2,4,5-benzene-tetracarboxylic dianhydride, 3,3′,4,4′-biphenyltetracarboxylic dianhydride, 4,4′-oxydiphthalic anhydride, and hexafluoroisopropylidene 2,2-bis(phthalic anhydride), via a two-step process. The resulting polyimides were characterized by solubility tests, viscosity measurements, FTIR, ^{1}H NMR spectroscopy, differential scanning calorimetric (DSC), and thermogravimetric analysis (TGA) analysis. The polyimides with inherent viscosities in the range of 0.048–0.095 L/g showed excellent solubility in aprotic amide and organic solvents, such as *N*,*N*-dimethylacetamide, *N*-methyl-2-pyrrolidinone, dimethylformamide and acetone, chloroform, etc. DSC showed glass transition temperatures (T_g) in the range of 116–143 °C. These polymers showed excellent thermal stability up to 390 °C.

Keywords Furan polymers · Polyimides · Diamine monomers · Solubility

Introduction

Polyimides, as aromatic polymers, are well-known and one of the most important classes of high-performance polymers because of outstanding characters, e.g. excellent thermostability, mechanical and insulation electrical properties and good chemical resistance [1–4]. These materials are widely used in many applications such as aerospace engineering, microelectronics, coatings, membranes, etc. [5–7].

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However, despite the excellent combined properties, one major drawback of these polymers is that they are typically insoluble in common organic solvents and do not melt before decomposition which limits their possible practical applications. Strong interaction between polyimide chains and their rigid structure are the main reasons for these behaviours. In order to overcome these processing problems, different methods have been introduced based on the modification of polymer structure. Attempts in this area include (i) introducing flexible segments into the polymer chain such as (–NHCO–, –O–, $–SO_2–\cdots$) [8–10], (ii) substituting symmetrical aromatic rings with unsymmetrical ones which led to a reduction in crystallinity [11] and (iii) incorporating bulky pendant groups to minimize crystallization [12, 13].

As a part of our research interest in the development of materials based on monomers from renewable resources [14], we have showed that the substitution of aromatic groups by furanic units containing two furanic heterocycles linked by an aliphatic bridge in the final macromolecules of some polymers such as polyamides [15], polyoxadiazoles [16], polyureas [17] or PET [18, 19] did not affect their thermal and mechanical properties and their chemical resistance.

Our recent investigation in the field of soluble and thermally stable materials resulted in the development of some poly(amide-imide)s based on difuranic dihydrazides and aromatic dianhydrides which exhibited good solubility in organic solvents due probably to the presence of both amide and furanic moieties [20].

The present study is devoted to the synthesis of some non-amide-containing furanic polyimides by solution polycondensation of difuranic diamines and aromatic dianhydrides in order to evaluate the effect of furanic moieties on the final properties. The absence of amide functions could reduce interactions between chains and then improve their solubility and their melt processing properties. To the best of our knowledge, polyimides-bearing furanic heterocycle appeared in very few reports [21–23].

Experimental

Materials

All chemicals were purchased from Aldrich Chemical Company. *N*,*N*-dimethylacetamide (DMA), *N*-methyl pyrrolidinone (NMP), dimethyl sulfoxide (DMSO), *N*,*N*-dimethylformamide (DMF), pyridine (Py) were treated with potassium hydroxide pellets for 24 h and distilled under reduced pressure over phosphorus pentoxide.

The aromatic dianhydrides, 1,2,4,5-benzene-tetracarboxylic dianhydride (**PMDA**), 3,3′,4,4′-Biphenyltetracarboxylic dianhydride (**BPDA**), 4,4′-oxydiphthalic anhydride (**ODPA**) and hexafluoroisopropylidene 2,2-bis(phthalic anhydride) (**6FDA**) were purified by refluxing for 2 h in acetic anhydride and subsequently crystallising them from the same medium. Furfurylamine (**FA**) or phthalic anhydride (**PA**), high purity products, were employed as received.

Instruments

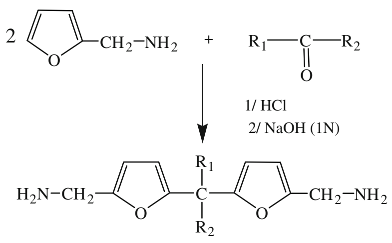
IR spectra were recorded on a Perkin-Elmer Infrared Spectrophotometer (FTIR 1650). The FTIR samples were prepared by casting films of the materials on KBr plates. ^{1}H NMR spectra were recorded on Bruker Avance 300 MHz spectrometers in $CDCl_3$ solutions (ref. d($CHCl_3$) 7.26 ppm). The inherent viscosity, η_{inh}, was measured at 25 ± 0.1 °C using Ubbelohde capillary viscometer. The concentrations of polymer solution in DMSO were 1.5 g/L. Differential scanning calorimetry (DSC) analyses were performed on a Perkin-Elmer differential scanning calorimeter DSC 7 at a heating rate of 10 °C/min under flowing nitrogen. T_g was taken as the midpoint of the inflection observed on the curve of heat capacity versus temperature. Melting points were measured by DSC method. Thermogravimetric analysis (TGA) was conducted with a TA Instruments, and experiments were carried out on approximately 10 mg of samples under controlled flux of nitrogen at 50 mL/min at a heating rate of 10 °C/min.

Synthesis of diamines

Difuranic diamines (**1a**–**6a**) were synthesized as already reported [17]. This synthesis involves the coupling reaction of **FA** with ketones or aldehydes in an acidic medium (Scheme 1). These diamines were purified by vacuum distillation and characterized by FTIR, ^{1}H NMR and ^{13}C NMR spectroscopy.

Synthesis of model compounds

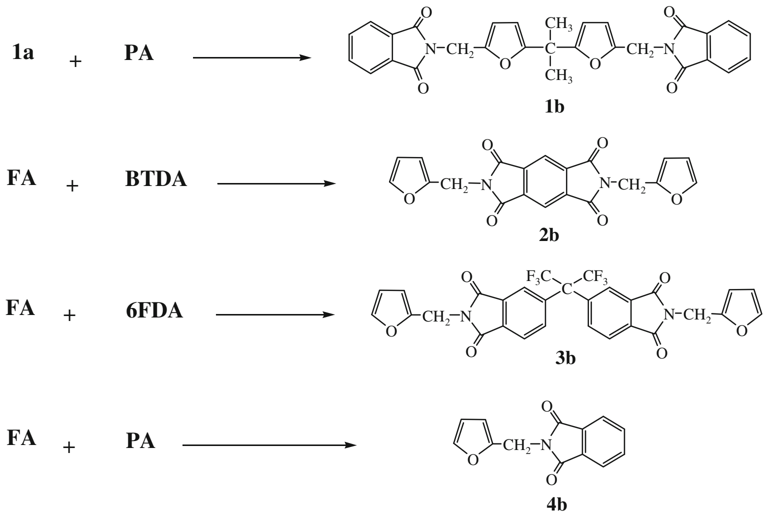
The dimeric or trimeric imidic model compounds **1b**–**4b** (Scheme 2) were prepared as follows. A solution of 10 mmol of amine or diamine and the appropriate stoichiometric corresponding anhydride or dianhydride in 10 mL of DMA was introduced in a 100-mL, two-necked, round-bottomed flask fitted with a gas inlet and outlet,



1a - 6a

Diamine	**1a**	**2a**	**3a**	**4a**	**5a**	**6a**
R_1 / R_2	CH_3 / CH_3	CH_3 / C_2H_5	CH_3 / C_3H_7	C_2H_5 / C_2H_5	CH_3 / C_5H_{11}	H / CH_3
Yield (%)	90	88	82	80	68	78
b.p. (°C) /0.07Torr	140	120	160	150	160	150

Scheme 1 Synthesis of difuranic diamines



Scheme 2 Synthesis of model compounds

condenser and a magnetic stirrer. This solution was stirred under nitrogen at 0 °C for 6 h and then 3 mL of acetic anhydride/pyridine (Ac_2O/Py) (6:4, v/v), was added. The resulting mixture was stirred at room temperature for 1 h and then refluxed at 100 °C for 4 h. Model compounds were isolated by precipitation in water and filtration.

Synthesis of polyimides

The synthesis of polyimide typically was carried out as follows. A 100-mL, two-necked, round-bottomed flask equipped with a magnetic stirrer, condenser and nitrogen inlet and outlet, was charged with 2.5 mmol of the dianhydride and 5 mL of dry DMA. The mixture was stirred at 25 °C until completely dissolved and 2.5 mmol of diamine in 5 mL of DMA was then added in one portion. The resulting mixture was stirred at 0 °C for 24 h. Imidization reaction was performed by the addition of 3 mL of a mixture of Ac_2O/Py (6:4, v/v). The solution was stirred for 1 h at room temperature then heated under nitrogen atmosphere in an oil bath at 100 °C for 4 h. After cooling, the viscous reaction mixture was poured into 200 mL of water, with constant stirring to produce a precipitate that was washed with methanol and hot water, collected on a filter, and dried at 100 °C under vacuum for 12 h.

Results and discussion

Model compounds

In order to gain a better insight into the interest of the two known synthetic procedures [20, 24] of polyimides, a model compound study was undertaken. Four

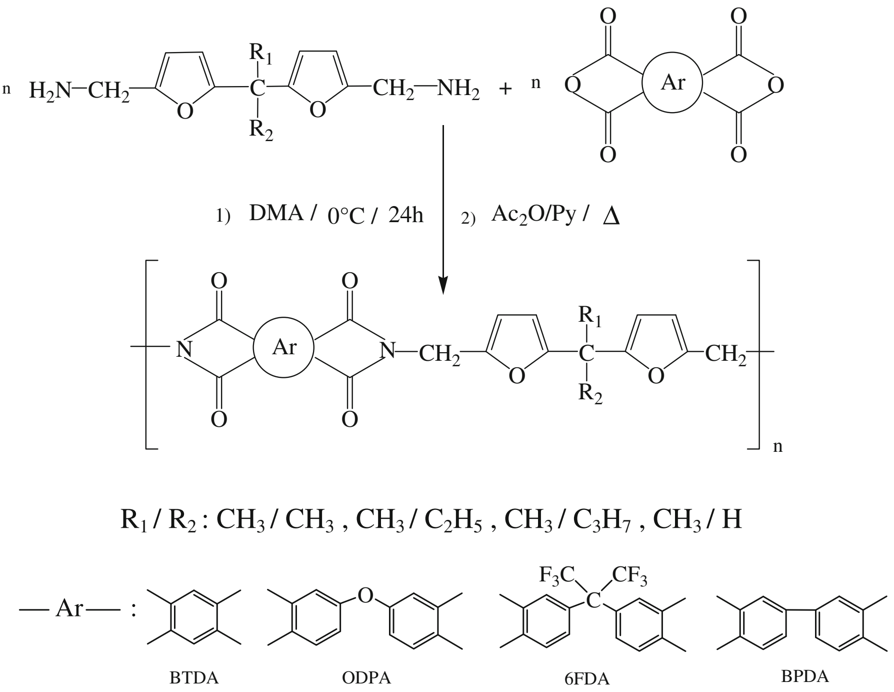
Table 1 FTIR and NMR data related to model compounds

Compound	Yield (%)	m.p. (°C)	FTIR (KBr) cm^{-1}	^1H NMR ($CDCl_3$) ppm
1b	80	115	1772, 1716, 1388, 1085, 710, 3100, 960, 800, 762	4.80/CH_2; 1.55/CH_3; (6.16, 5.85)/H-Fu; (7.71, 7.83)/Ar
2b	91	205	1769, 1705, 1392, 1111, 716, 3118, 941, 802, 753	4.91/CH_2; (6.32, 6.40, 7.34)/H-Fu; 8.29/Ar
3b	88	77	1779, 1723, 1386, 1109, 716, 3124, 944, 849, 747	4.87/CH_2; (6.31, 6.38, 7.34)/H-Fu; (7.77, 7.80, 7.93)/Ar
4b	70	100	1770, 1706, 1392, 1109, 713, 3120, 943, 801, 734	4.85/CH_2; (6.29, 6.36, 7.32)/H-Fu (7.71, 7.85)/Ar

imidic model derivatives **1b–4b** were prepared by stoichiometric addition reaction of **1a** with **PA** and **FA** with **BTDA**, **6FDA** or **PA** followed by cyclodehydration of the ensuing amic-acid intermediates as shown in Scheme 2. Our attempt to prepare these compounds according to the classical procedure previously used in the synthesis of furanic amide-imide compounds [20] failed. Efforts were made to improve this situation by varying reaction parameters and precipitation solvents but the results remained unsatisfactory in term of amic-acid yield isolated after the first step. This problem induced us to examine the possibility of cyclodehydration of amic-acid intermediates in situ as already reported [25]. It was found that this technique provided more satisfactory performance, and the best results were obtained when addition reaction was conducted in DMA at room temperature during 6 h followed by imidization at 100 °C in the presence of Ac_2O/Py as catalyst during 12 h. Table 1 shows that these optimized conditions led to model compounds in good yields and high selectivity. The FTIR spectra are entirely consistent with the expected structures of these furanic imides (cm^{-1}, KBr): 1770–1780 (symmetrical C=O stretching vibration), 1705–1720 (asymmetrical C=O stretching vibration), 1380–1390 (symmetrical C–N stretching vibration), 1110 and 715 (imide ring deformation), 1580 (C=C Fu), 1020 (furan ring breathing). The ^1H NMR spectra also agree with the structures proposed. In particular, no signals related to amide functions were observed thus indicating the complete cyclodehydration of amic-acid precursors.

Polyimides synthesis

The synthesis of polyimides was carried out using a two-step technique involving first poly(amic-acide)s formation by ring opening poly-addition and then their conversion in situ by cyclodehydration to give the corresponding polyimides. Scheme 3 illustrates the general reaction pathway for these syntheses which led to the corresponding polyimides $\mathbf{P_{1–8}}$. A brief study conducted on a model system namely (**1a** + **PA**) showed that the use of DMA as solvent and (Py + Ac_2O) as cyclodehydration agent gave the best results. It was noted also that the ratio Ac_2O/Py and the amount of optimized mixture introduced in the medium after the ring



Scheme 3 Synthesis of polyimides $\mathbf{P_{1-8}}$

opening poly-addition phase played an important role for successful cyclodehydration as emphasised previously [24, 26]. These optimized conditions led, for the various combinations used here, to the corresponding polyimides in quantitative yields with inherent viscosities ranging from 0.048 to 0.095 L/g. These two basic sets related to the optimized syntheses are given in Table 2.

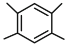
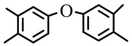
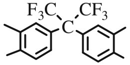
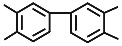
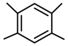
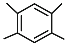
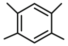
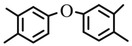
Polyimides characterization

The FTIR spectra of all the polyimides reported in Table 2 were consonant with the corresponding expected structures. The absence of acid and amide absorptions in these spectra confirmed the complete imidization of poly(amic-acid)s intermediates. This result was confirmed also by the ^{1}H NMR spectra which showed, in particular, the absence of resonance related to the NH amide functions and the presence of peak at 4.6–4.8 ppm corresponding to the methylenes linked to both imidic and furanic heterocycles. The ^{1}H NMR chemical shifts and assignments are summarized in Table 3, and Fig. 1 shows a typical example of such a spectrum.

Polyimides properties

Furanic polyimides $\mathbf{P_{1-8}}$ solubilities were studied qualitatively in various solvents at room temperature and upon heating and then compared with those of the

Table 2 Yields and inherent viscosities related to polyimides $\mathbf{P_{1–8}}$

Polyimide	$\mathbf{P_1}$	$\mathbf{P_2}$	$\mathbf{P_3}$	$\mathbf{P_4}$	$\mathbf{P_5}$	$\mathbf{P_6}$	$\mathbf{P_7}$	$\mathbf{P_8}$
R_1/R_2	CH_3/CH_3	CH_3/CH_3	CH_3/CH_3	CH_3/CH_3	CH_3/C_2H_5	CH_3/C_3H_7	H/CH_3	H/CH_3
Ar								
η_{inh}^{a} (L g^{-1})	0.074	0.058	0.068	0.048	0.062	0.095	0.060	0.050
Yield (%)	95	88	91	91	93	98	96	90

[a] Inherent viscosity measured at a concentration of 1.5 g/L in DMSO at 25 °C

Table 3 FTIR and NMR data related to polyimides $\mathbf{P_{1–8}}$

Polymer	IR (KBr) (cm^{-1})	^{1}H NMR (CDCl$_3$) (ppm)
$\mathbf{P_1}$	1775; 1721; 1387; 1112; 730 (imide functions)	4.82/CH$_2$; (6.19, 5.87)/H-Fu
	3107; 1020; 963; 793; 760 (furan-related peaks)	1.51/(R$_1$, R$_2$); 8.20/Ar
$\mathbf{P_2}$	1776; 1718; 1384; 1091; 717 (imide functions)	4.77/CH$_2$; (6.17, 5.92)/H-Fu
	3098; 1019; 960; 794; 767 (furan-related peaks)	1.53/(R$_1$, R$_2$); (7.95, 7.43, 7.34)/Ar
$\mathbf{P_3}$	1780; 1713; 1386; 1105; 717 (imide functions)	4.79/CH$_2$; (6.19, 5.87)/H-Fu
	3068; 1019; 950; 794; 767 (furan-related peaks)	1.52/(R$_1$, R$_2$); (7.98, 7.89, 7.74)/Ar
$\mathbf{P_4}$	1772; 1712; 1385; 1108; 713 (imide functions)	4.61/CH$_2$; (6.22, 5.95)/H-Fu
	3067; 1019; 969; 795; 767 (furan-related peaks)	1.49/(R$_1$, R$_2$); (7.98, 8.17, 8.19)/Ar
$\mathbf{P_5}$	1774; 1720; 1387; 1106; 715 (imide functions)	4.82/CH$_2$;(6.19, 5.89)/H-Fu
	3108; 1020; 962; 793; 763 (furan-related peaks)	(0.66, 1.46, 1.93)/(R$_1$, R$_2$); (8.26)/Ar
$\mathbf{P_6}$	1775; 1721; 1387; 1102; 712 (imide functions)	4.70/CH$_2$; (6.19, 5.89)/H-Fu
	3035; 1021; 950; 795; 757 (furan-related peaks)	(0.61, 0.95, 1.40, 2.09)/(R$_1$, R$_2$); (8.10)/Ar
$\mathbf{P_7}$	1774; 1713; 1343; 1104; 727 (imide functions)	4.72/CH$_2$; (6.26, 5.99)/H-Fu
	3102; 1017; 965; 795; 762 (furan-related peaks)	(1.40, 4.11)/(R$_1$, R$_2$); (8.57)/Ar
$\mathbf{P_8}$	1774; 1716; 1387, 1097, 723 (imide functions)	4.64/CH$_2$; (6.19, 5.95)/H-Fu
	3101; 1017; 968; 796; 767 (furan-related peaks)	(1.37, 4.09)/(R$_1$, R$_2$); (8.17, 7.45)/Ar

corresponding aromatic polyimides or furanic poly(amide-imide)s reported previously [20, 27]. It can be seen as shown in Table 4 that furanic polyimides were easily dissolved both in polar solvents such as DMA, DMF, NMP, DMSO and m-cresol and in less polar solvents such as $CHCl_3$, CH_2Cl_2 and THF. This good solubility was not influenced by the nature of the aromatic units or the structure of aliphatic bridge in furanic units. These polymers exhibited better solubility than homologous polyimides containing linear stiff sequences of 1,4 aromatic units which can be attributed to the presence of flexible furanic units into the polymer backbones [6, 27]. For comparison, the solubility behaviour of some furanic poly(amide-imide)s $\mathbf{PAI_1}$, $\mathbf{PAI_5}$ and $\mathbf{PAI_7}$ reported previously is also presented in Table 4. These polymers exhibited comparable solubility in strong polar solvents but were less soluble in low polar solvents ($CHCl_3$, CH_2Cl_2 and THF) than the corresponding polyimides $\mathbf{P_1}$, $\mathbf{P_5}$ and $\mathbf{P_7}$. This indicates that elimination of amide functions in polymer backbone decreased the interaction between polymer chains to enhance solubility as anticipated in the introduction.

Thermal behaviour

Differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) were used to evaluate the thermal properties of the polyimides. DSC and TGA curves of the polyimides are shown in Figs. 2 and 3, respectively, and thermal analysis data from the TGA and DSC curves of the polyimides are summarized in Table 5. The glass transition temperatures of the polyimides ranged from 116 to 143 °C which was depended on the chemical structure of the two series of monomers. The polyimide $\mathbf{P_5}$ derived from 2a-BTDA exhibits the highest T_g

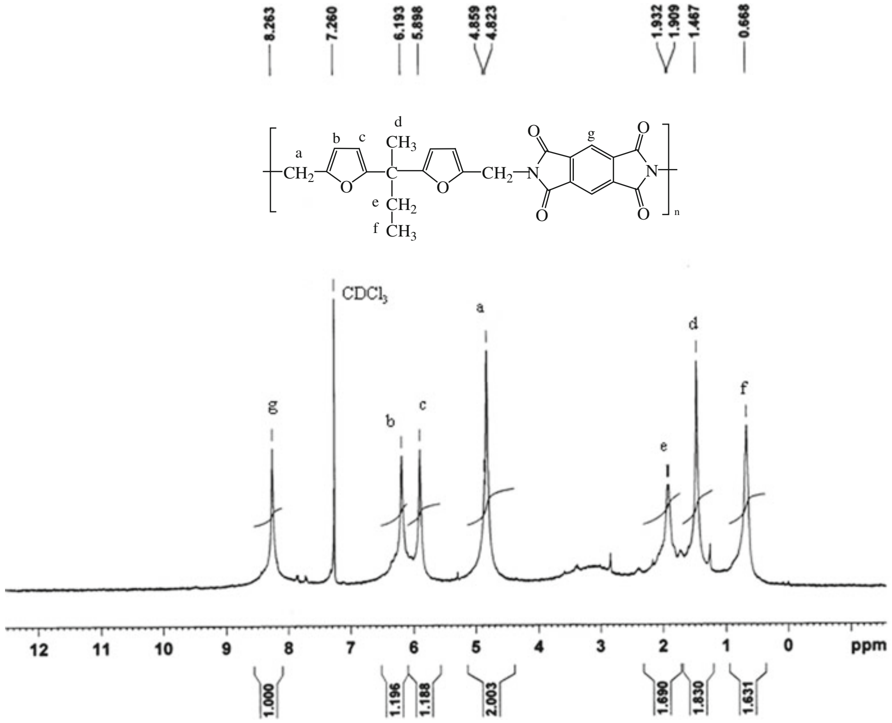


Fig. 1 ^{1}H NMR spectrum [300 MHz, $CDCl_3$, reference: $\delta(CHCl_3)$ 7.26 ppm] of $\mathbf{P_5}$

Table 4 Solubility of the various polyimides

Polyimide	Organic solvents[a]							
	DMAc	DMF	NMP	DMSO	m-cresol	THF	$CHCl_3$	CH_2Cl_2
$\mathbf{P_1}$	++	++	++	++	+	++	++	++
$\mathbf{PAI_1^b}$	++	++	+	+	+−	−	−	−
$\mathbf{P_2}$	++	+	++	++	++	++	++	++
$\mathbf{P_3}$	++	++	++	++	++	++	++	++
$\mathbf{P_4}$	++	++	+	+	+	+−	++	+
$\mathbf{P_5}$	++	++	++	++	+	++	++	+−
$\mathbf{PAI_5^b}$	++	++	++	++	+	−	−	−
$\mathbf{P_6}$	++	++	++	++	+	++	+−	+−
$\mathbf{PAI_6^b}$	++	++	++	++	+	−	−	−
$\mathbf{P_7}$	++	+	++	++	++	+−	+−	+−
$\mathbf{P_8}$	++	++	++	+	+	++	+−	+−

[a] Qualitative solubility was determined by dissolving 100 mg of polyimide in 900 mg of solvent at room temperature or upon heating

[b] Results reported in ref. [20]. ++ soluble at room temperature, the solid polymer was completely dissolved in the solvent. + soluble upon heating, +− partially soluble, −− insoluble

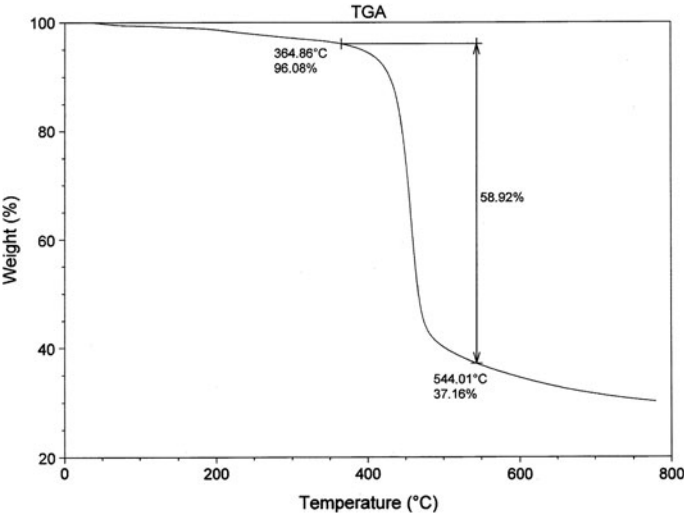


Fig. 2 TGA tracing of polyimide $\mathbf{P_2}$

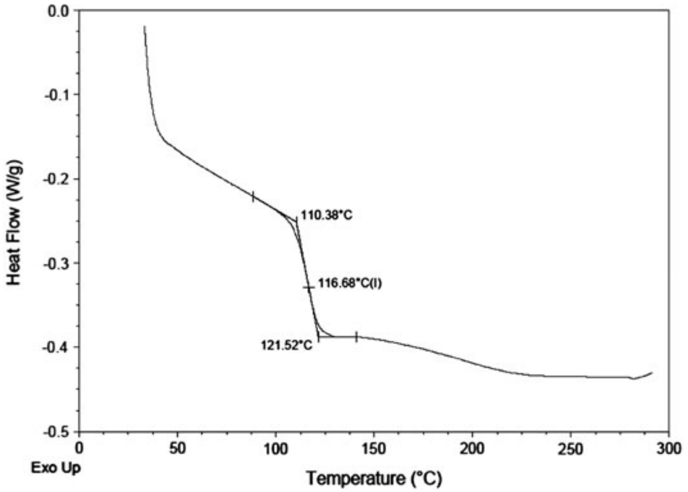


Fig. 3 DSC thermogram of polyimide $\mathbf{P_7}$

Table 5 Thermal behaviour of polyimides $\mathbf{P_1}$–$\mathbf{P_8}$

Polyimide	$\mathbf{P_1}$	$\mathbf{P_2}$	$\mathbf{P_3}$	$\mathbf{P_4}$	$\mathbf{P_5}$	$\mathbf{P_6}$	$\mathbf{P_7}$	$\mathbf{P_8}$
T_g (°C)	138	116	134	129	143	134	116	129
T_d (°C)	347	364	402	384	336	273	299	357

because of the rigid backbone, and **P_2** derived from 2a-ODPA exhibits a lower T_g because of the presence of a flexible ether linkage between the phthalimide units.

Conclusion

A two-step synthetic method to produce soluble polyimides with high-molecular weights from corresponding bifuranic diamines and aromatic dianhydrides was developed. The successful polymerization was proved by both FTIR and ^{1}H NMR spectra. Experimental results indicated that the resulting polyimides that hold inherent viscosities of 0.048–0.095 L/g could easily be dissolved in many aprotic amide and organic solvents and showed excellent thermal stability up to 390 °C. Work is in progress to assess the properties and possible applications of these materials.

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