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ORIGINAL PAPER

Synthesis and characterization of soluble polyamides from bis-[(4′-aminobenzyl)-4-benzamide] ether and various diacids

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Abstract New aromatic diamine containing preformed amide, ether, and methylene; bis-[(4′-aminobenzyl)-4-benzamide] ether (BABE), was synthesized and characterized by FT-IR, NMR, and mass spectrometry. Aromatic–aliphatic polyamides were prepared from BABE with aliphatic/aromatic diacids via Yamazaki's polymerization. The polyamides were characterized by FT-IR, ^{1}H NMR, inherent viscosity [η_{inh}], solubility tests, differential scanning calorimetry [DSC], thermogravimetric analysis [TGA], and X-ray diffraction [XRD]. Polyamides had inherent viscosities 0.35–0.84 dL/g, soluble in aprotic polar solvents like *N*-methyl-2-pyrrolidone, *N*, *N*-dimethyl acetamide and dimethyl sulphoxide containing LiCl due to an amorphous to partially crystalline morphology; as XRD patterns indicated. DSC analysis of polyamides showed glass transition temperatures 166–268 °C. Polyamides showed high thermal stability as they did not degrade below 300 °C, had 10% weight loss temperature higher than 375 °C, and the char yields at 900 °C were 22–55%; indicating potential applications as engineering materials.

Keywords Soluble aromatic-aliphatic polyamides · Bis-[(4′-aminobenzyl)-4-benzamide] ether · Direct phosphorylative polycondensation · Aryl-ether and methylene linkages · Thermal properties

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Introduction

Rigid-rod aromatic polyamides have constantly attracted wider interest because of their unique mechanical, thermal, and morphological properties [1–6]. However, processing of these materials is generally difficult due to their limited solubility and infusibility. Some of these problems are caused by strong inter-molecular hydrogen bonds and donor–acceptor interactions [7] of the amide functions, which cause chain stiffness. To develop processable high-performance materials, modifications that increase the solubility while maintaining the thermal stability are of particular interest. Thermally stable, soluble, and photoactive aromatic polyesters, poly(ester-imide)s, poly(amide-ester-amide)s, and polyamides with improved processability were reported. Some of the successful approaches to increase solubility and processability of polymers are the introduction of bulky pendant groups [8–15], flexible alkyl side chains [16, 17], unsymmetric [18–20], heterocyclic rings [21–23], and cardo groups [24–27]. Another approach which increased the solubility of rigid-rod polyamides is incorporation of the flexible or bridging functional groups [28–31] in the polymer backbone.

In laboratory, aromatic polyamides are usually synthesized from aromatic dicarboxylic acid chloride and aromatic diamines by the low temperature solution polycondensation method [32]. To overcome the difficulties in handling moisture sensitive acid chloride, high molecular weight polyamides were synthesized by Higashi–Yamazaki's phosphorylation method by direct polycondensation of an aromatic dicarboxylic acid in presence of di- or tri-phenyl phosphite [33–38].

We reported the synthesis of polyamides [39] containing flexible ether and methylene linkages in the polymer backbone by the low temperature solution polycondensation method. In continuation to earlier studies now we report a series of eight novel aromatic-aliphatic polyamides bearing ether, preformed amide and methylene linkages from aromatic diamine, bis-[(4′-aminobenzyl)-4-benzamide] ether (BABE) with different aliphatic/aromatic diacids via Yamazaki's direct phosphorylative polymerization. The polyamides were characterized by FT-IR spectroscopy, inherent viscosity, solubility, thermal analysis, and XRD studies to correlate the structure property relationship.

Experimental

Materials

N-Methyl-2-pyrrolidone (NMP) and *N*, *N*-dimethyl acetamide (DMAc) were purified by distillation under reduced pressure over calcium hydride and stored over molecular sieves 4 Å. Pyridine was refluxed over potassium hydroxide pellets under nitrogen, distilled and stored over 4 Å molecular sieves. Adipic acid, azelic acid, sebasic acid, isophthalic acid, terephthalic acid, and 4,4′oxy-bis(benzoic acid) were used after purification by crystallization. Bis (4-carboxyphenyl) dimethyl silane and 1,1-bis [4″-(4′-carboxy methylene phenoxy phenyl) cyclohexane were prepared by reported procedure. LiCl was dried under vacuum at 150 °C for 6 h prior to use. Triphenyl phosphite (Merck) was used as received.

Measurements

All melting points were determined on a Mel-Temp melting point apparatus and are uncorrected. The transmission IR spectra of polymers were recorded as a KBr pellet on a Perkin-Elmer 883 IR spectrophotometer. ^{1}H-NMR spectra and ^{13}C-NMR spectra were recorded on a Bruker NMR spectrophotometer (400 MHz) in DMSO-d_6. Mass spectra were recorded on IIMS 30 double beam mass spectrometer. Inherent viscosity measurements were made with a 0.5% (w/v) polyamide solution in DMAc containing 4% LiCl at 30 ± 0.1 °C using suspended level Ubbelohde viscometer. The solubility of polymers was determined at 3 wt% concentration in various solvents at room temperature or on warming if needed. Differential scanning calorimetry (DSC) analysis was performed on Q10 TA Instrument at a heating rate of 20 °C/min and thermogravimetric analysis (TGA) was performed on TGA Q5000 at a heating rate of 10 °C/min under a flow of nitrogen gas. Wide angle X-ray diffraction (WAXD) measurements were made in the powder form of polyamides on Jeol JDX-8030 X-ray diffractometer with nickel filtered CuKα ($\lambda = 1.5418$ Å).

Synthesis of monomer

Bis-[(4′-aminobenzyl)-4-benzamide] ether (BABE) was synthesized in 93% yields by the procedure reported by us earlier [39]. M. P. 208–210 °C, FT-IR (KBr, Fig. 1a): 3400 and 3387 (–NH_2), 3241 (–NH), 1651 (amide-I, carbonyl stretching) and 1604 cm^{-1} (amide-II, NH deformation). ^{1}H-NMR (DMSO-d_6): in ppm, 10.02 (s, –NH), 7.55–7.6 (d), 6.96–7.0 (d), 6.84–6.94 (d), 6.49–6.53 (d), 4.94 (s, broad, –NH_2), and 3.37 (s, CH_2). ^{13}C-NMR (DMSO-d_6): in ppm, 170.17 (carbonyl C), 152.77, 147.42, 135.13, 123.31 (tertiary Cs), 129.76, 121.09, 118.94, 114.27 (CH) and 42.92 (CH_2); MS: m/z 466 (M^+).

Synthesis of polyamides

Synthesis of polyamides was performed by Yamazaki's phosphorylation method.

Synthesis of polyamide (YPA-1) from BABE and adipic acid

In a 100 mL three neck round bottom flask equipped with reflux condenser, magnetic stirrer, calcium chloride guard tube, and nitrogen gas inlet were placed 0.466 g (1 mmol) BABE, 0.146 g (1 mmol) adipic acid, 0.200 g lithium chloride [8 wt% based on solvent *N*-methyl-2-pyrrolidone (NMP) and pyridine mixture] and 0.744 g (0.63 mL, 2.4 mmol) triphenyl phosphite (TPP), 0.5 mL pyridine, and 2 mL NMP. The mixture was stirred well and temperature was slowly raised to

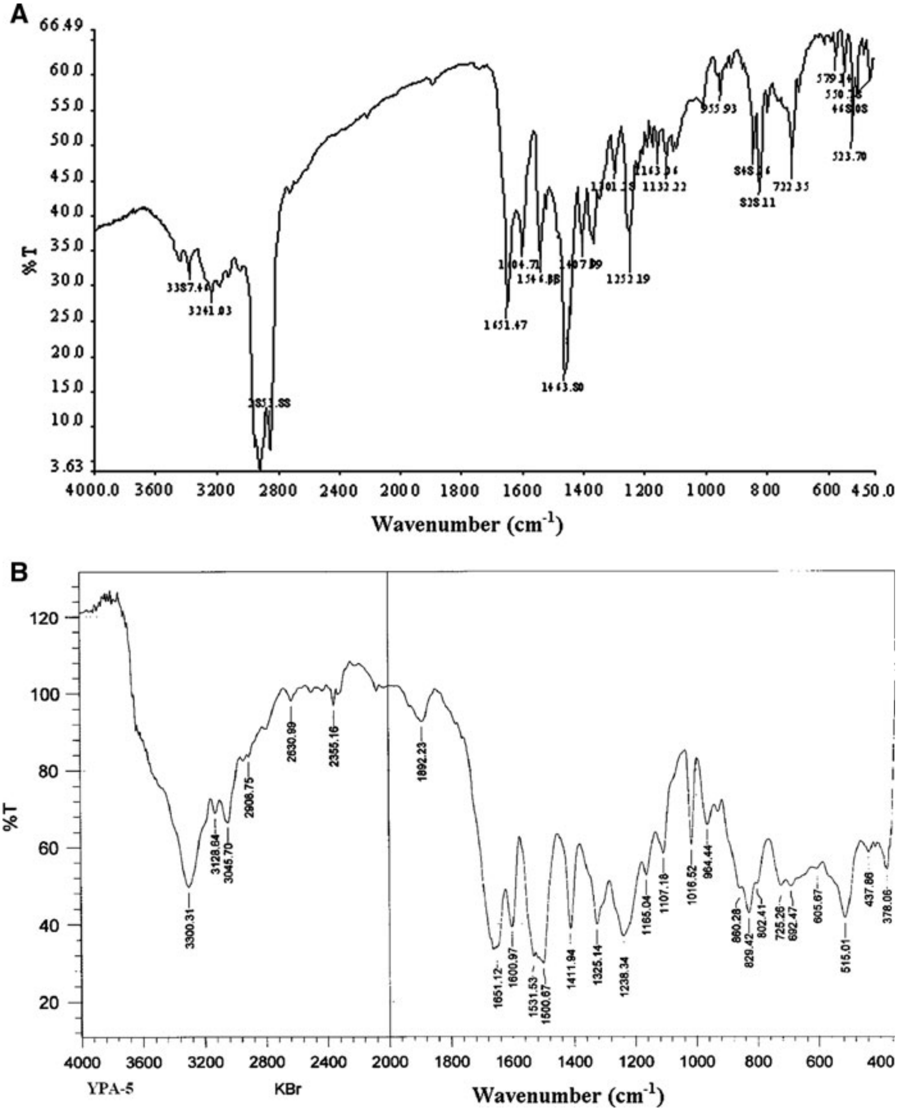


Fig. 1 **a** FT-IR spectrum of BABE. **b** FT-IR spectrum of polyamide YPA-5

100 °C over a period of 30 min and the mixture maintained at 100 °C for 3 h. After cooling the viscous solution was poured into rapidly stirred 200 mL methanol. The precipitated polymer (YPA-1) was filtered, washed with methanol and dried under vacuum at 100 °C for 8 h. The yield was 98% and the inherent viscosity of polymer was 0.51 dL/g.

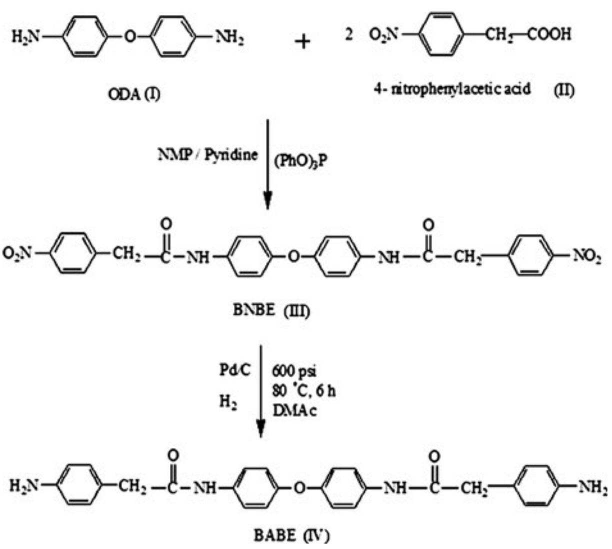
Similarly other polyamides, YPA-2 to YPA-8, were synthesized with different aliphatic/aromatic diacids.

Results and discussion

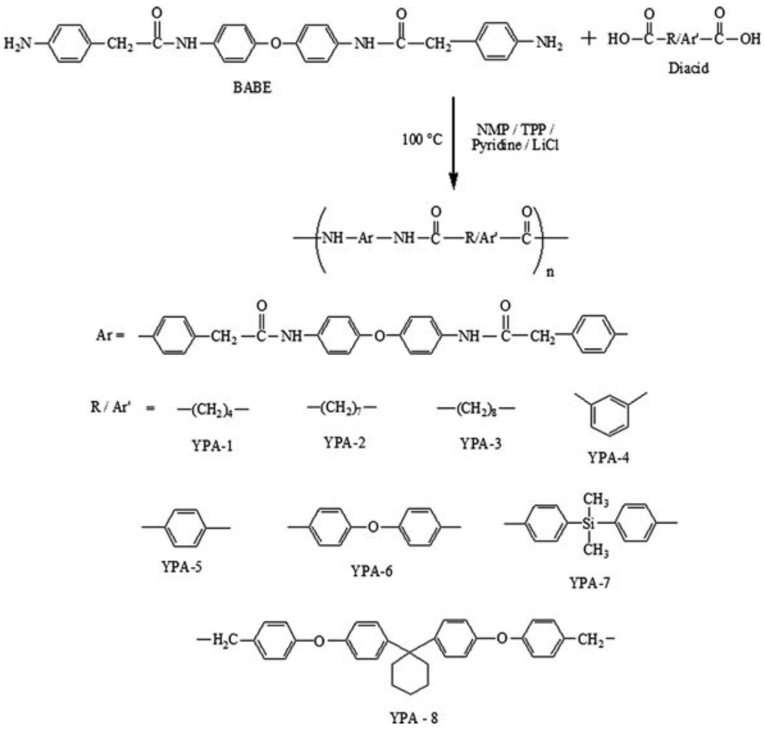
New aromatic diamine, bis-[(4′-aminobenzyl)-4-benzamide] ether (BABE) containing preformed amide, ether linkage and methylene group was synthesized (Scheme 1) and characterized by FT-IR, NMR (^{1}H and ^{13}C), and mass spectrometry [39].

High molecular weight aromatic–aliphatic polyamides bearing ether, preformed amide and methylene linkages were prepared by polymerization of BABE with different aliphatic/aromatic diacids containing additional ether/silicon/cardo cyclohexylidene and methylene units, using Yamazaki's phosphorylation method (Scheme 2). Triphenyl phosphite was used as the condensing agent, wherein the mixture of *N*-methyl-2-pyrrolidone (NMP) and pyridine (4:1 by volume) containing 8 wt% anhydrous lithium chloride was employed as solvent. The polymerization was carried out at 100 °C for 3 h under nitrogen. The polymerization proceeded smoothly giving viscous solutions, which were precipitated by pouring the viscous solution in methanol.

The results of synthesis of polyamides are presented in Table 1. All the polymers were obtained in almost quantitative yields (97–99%). The inherent viscosities were in the range 0.35–0.84 dL/g in (DMAc + 4% LiCl) indicating moderate to high molecular weight buildup. The formation of the polyamides was confirmed by FT-IR spectra. The FT-IR spectrum of a representative polyamide, YPA-5 (Fig. 1b) exhibited characteristic absorption bands at 1,651 and 1,531 cm^{-1} due to amide-I (C=O stretching) and amide-II (N–H deformation), respectively. This clearly proves that polymerization was successful, as amide-II absorption in BABE appeared at 1,604 cm^{-1}. The absorption band at 3,300 cm^{-1} was of –NH. The absorption bands



Scheme 1 Synthesis of bis-[(4′-aminobenzyl)-4-benzamide] ether (BABE)



Scheme 2 Synthesis of polyamides from BABE and various diacids

Table 1 Yield, inherent viscosity of polyamides from BABE and different diacid

Serial No.	Polymer code	Diacid	Yield (%)	Inherent viscosity[a] η_{inh} (dL/g)
1.	YPA-1	Adipic acid	99	0.51
2.	YPA-2	Azelic acid	97	0.80
3.	YPA-3	Sebasic acid	98	0.84
4.	YPA-4	Isophthalic acid	98	0.61
5.	YPA-5	Terephthalic acid	98	0.80
6.	YPA-6	4,4′-Oxy-bis(benzoic acid)	99	0.44
7.	YPA-7	Bis(4-carboxyphenyl) dimethyl silane	98	0.52
8.	YPA-8	1,1-Bis [4″-(4′-carboxy methylene phenoxy phenyl) cyclohexane	98	0.35

Polymerization was carried out with 1 mmol each of BABE and diacid

BABE bis-[(4′-aminobenzyl)-4-benzamide] ether

[a] Measured with a 0.5% (w/v) polymer solution in (DMAc + 4% LiCl) at 30 ± 0.1 °C

at 3128, 3045 (aromatic C–H stretching) and at 2,908 cm^{-1} (aliphatic C–H stretching) were also seen. The disappearance of absorption bands at 3,400 and 3,387 cm^{-1} due to primary amine group confirmed the polyamide formation.

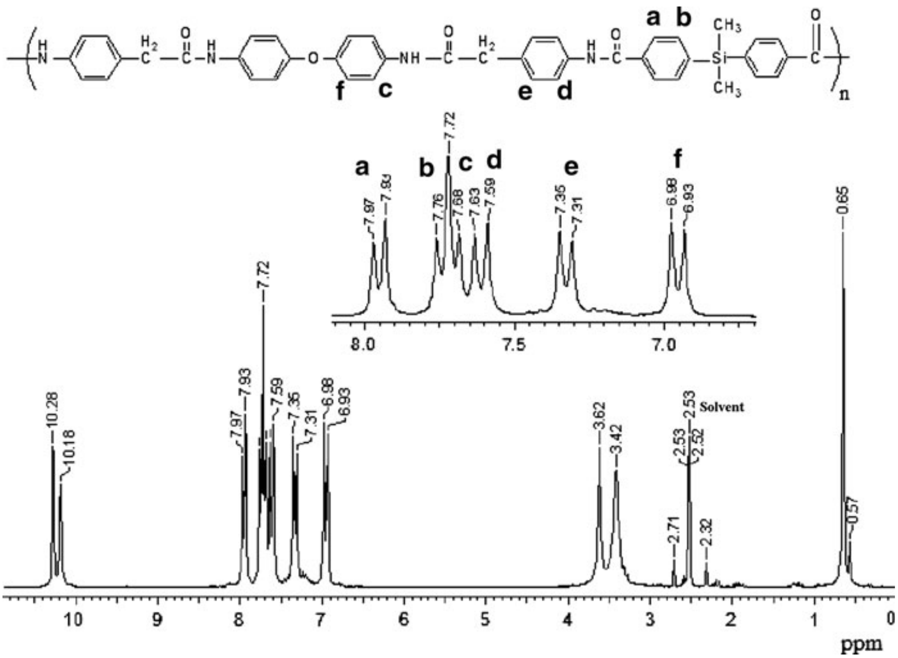


Fig. 2 ^{1}H NMR spectrum of YPA-7

Further polyamide structures were characterized by NMR technique. As a representative, ^{1}H-NMR spectrum of the polyamide YPA-7 is presented in Fig. 2 and it shows NMR signals (singlets) at 10.28, 10.18 ppm due to amide (CO–NH) proton of Ar–NH–CO–Ar and Ar–NH–CO–CH_2 structures of the polyamide. NMR peaks, as clear doublets, at 7.97–7.93, 7.76–7.72, 7.72–7.68, 7.63–7.59, 7.35–7.31 and 6.98–6.93 were assigned to "a-f type" aromatic protons. Singlets at 3.42 and 0.65 were assigned to (CH_2) and (CH_3, attached to Si) functionality. Thus, ^{1}H-NMR spectrum of YPA-7 is consistent with its structure.

The solubility behavior of the polyamides in various solvents is tabulated in Table 2. All the polyamides, except YPA-5 and YPA-6, showed the solubility in aprotic solvent such as NMP, DMAc, and DMSO. Polyamides synthesized from aliphatic dicarboxylic acids showed better solubility whereas polyamides YPA-5 and YPA-6, based on terephthalic acid and 4,4′-oxy-bis(benzoic acid), respectively, showed limited solubility probably due to 1,4-catenation.

Polymers were insoluble in common organic solvents like $CHCl_3$, DCM, THF, and acetone, etc. However, polyamides YPA-8; based on bulky cyclohexylidene cardo unit containing diacid was soluble in pyridine and *m*-cresol, and YPA-7 containing silicon, dissolved in hot pyridine and hot *m*-cresol due to the loose chain packing of these polymer chains which probably allowed penetration of the solvent to solubilize the polymer.

The thermal properties of the polymers were evaluated by differential scanning calorimetry and thermogravimetry. Table 3 incorporates the thermal data such as initial decomposition temperature (T_i), 10% weight loss temperature (T_{10}),

Table 2 Solubility of polyamides

Solvent ↓	Polymer →							
	YPA-1	YPA-2	YPA-3	YPA-4	YPA-5	YPA-6	YPA-7	YPA-8
DMAc	++	++	++	++	+−	+−	++	++
NMP	+	++	++	++	+−	+−	++	++
DMSO	++	++	++	++	+−	−	++	++
DMF	+−	+−	+−	+−	+−	−	++	++
Pyridine	S	S	S	S	S	S	+	++
m-Cresol	+−	+−	+−	S	+−	+−	+	++
$CHCl_3$	−	−	−	−	−	−	−	−
DCM	−	−	−	−	−	−	−	−
THF	−	−	−	−	−	−	−	−
Acetone	−	−	−	−	−	−	−	−
H_2SO_4	++	++	++	++	++	++	++	++

(++): Soluble at room temperature, (+): soluble on heating, S: swelling, (+−): partially soluble, (−): insoluble

Table 3 Thermal properties of polyamides

Polymer code	T_i (°C)[a]	T_{10} (°C)[b]	T_{max} (°C)[c]	Residual wt. (%) at 900 °C	T_g (°C)[d]
YPA-1	318	365	546	23	192
YPA-2	315	373	532	29	168
YPA-3	300	370	442	37	166
YPA-4	330	401	564	26	268
YPA-5	335	398	560	25	–
YPA-6	319	433	498	55	–
YPA-7	321	402	447	54	236
YPA-8	320	396	552	36	206

Thermogravimetric analyses were conducted at a heating rate of 10 °C/min under nitrogen

[a] Temperature at which weight loss initiated

[b] Temperature at which 10% weight loss was observed

[c] Temperature at which maximum rate of weight loss was observed; determined from DTG curve

[d] Determined by DSC measured at a heating rate 20 °C/min

temperature of maximum rate of decomposition (T_{max}) and residual weight (char yield) at 900 °C.

The glass transition temperature (T_g), determined by DSC (Fig. 3), of the polyamides were in the range 166–268 °C. It was observed that T_g values of polyamides based on aliphatic dicarboxylic acids; YPA-1 to YPA-3, were lower than those based on aromatic dicarboxylic acids viz YPA-4 and YPA-7. Further T_g decreased among aliphatic diacid based polyamides YPA-1 to YPA-3 (192, 168, 166 °C) as the number of $–CH_2–$ groups increased. This is obvious as there was increased flexibility as $–CH_2–$ chain length increased from adipic, azelic, and

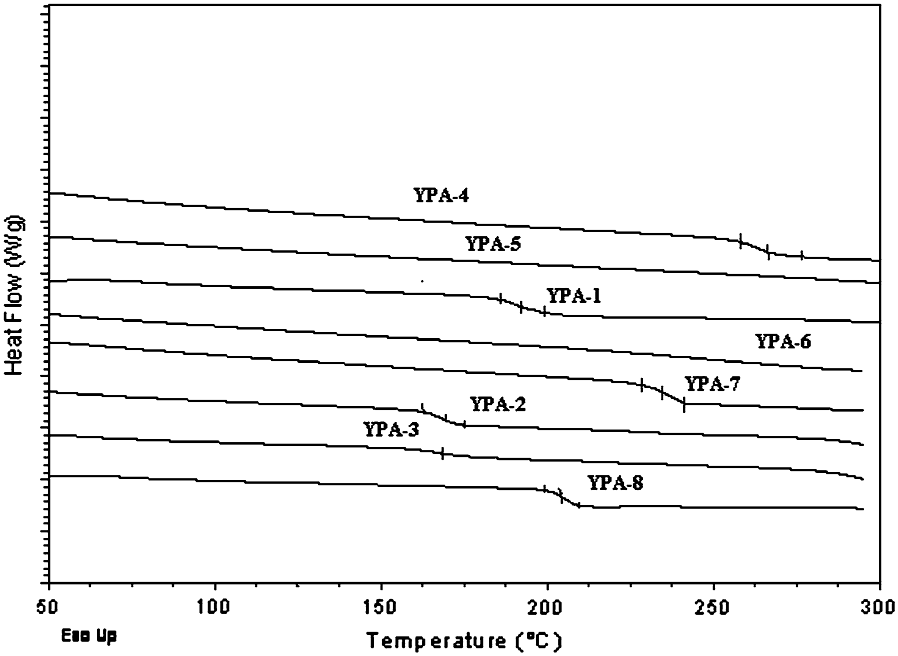


Fig. 3 DSC curves of YPA-1 to YPA-8

sebasic acids. There is drastic lowering in T_g (192–168 °C) for YPA-1 and YPA-2 because –CH_2– length increased from $(CH_2)_4$ to $(CH_2)_7$. But there is not much lowering of T_g (168–166 °C) for YPA-3 compared to YPA-2 since $(CH_2)_7/(CH_2)_8$ length is nearly same. YPA-8 also showed lowered T_g (206 °C) due to the presence of cardo cyclohexylidene, ether, and methylene flexible structures.

In TG analyses, all the polyamides showed a similar pattern of decomposition, with no weight loss below 300 °C (Fig. 4). Initial decomposition temperature (T_i) and 10% weight loss temperature (T_{10}) were in the range 300–335 °C and 365–433 °C, respectively, indicating the high thermal stability; for the structure units of these polyamides contained amide groups capable of hydrogen bonds and phenyl rings, besides the molecules structure was symmetric. The char yields of the polyamides at 900 °C were in the range 23–55% and T_{max} between 442 and 564 °C.

The wide angle X-ray diffractograms of polyamides (Fig. 5) showed that there was only one broad peak for all the polyamides, except YPA-5 and YPA-6, confirming amorphous morphology. Polyamides YPA-5 and YPA-6 exhibited partly crystalline nature which is reflected in limited solubility.

Conclusions

A novel aromatic diamine bearing ether, preformed amide, and methylene linkages bis-[(4′-aminobenzyl)-4-benzamide]ether (BABE) was synthesized and number of

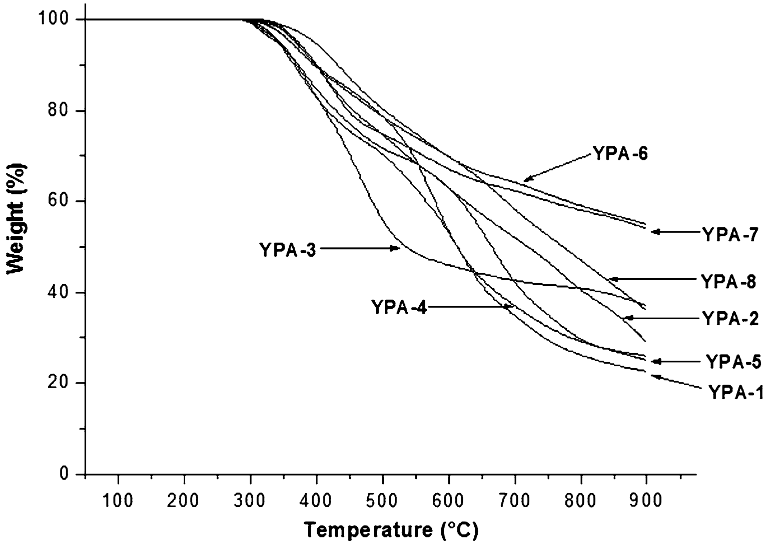


Fig. 4 TG curves of YPA-1 to YPA-8

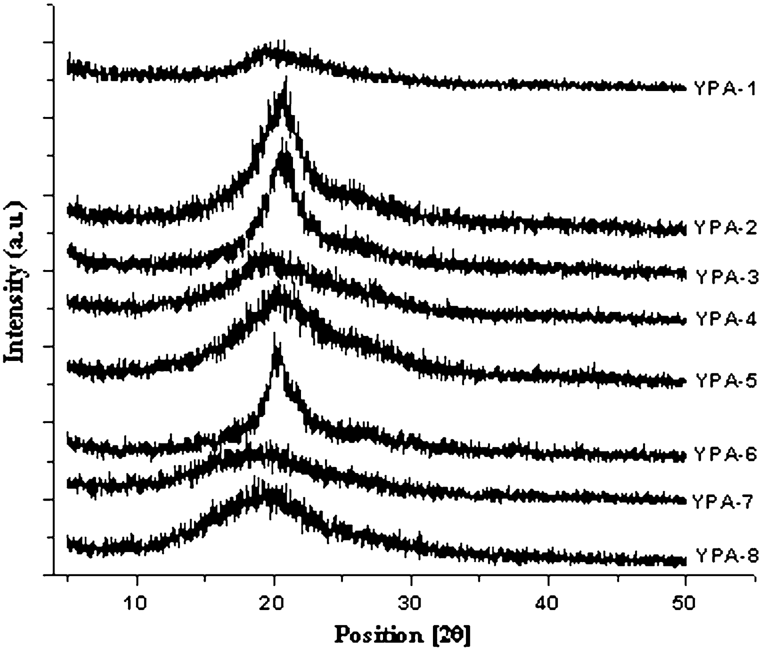


Fig. 5 XRD curves of YPA-1 to YPA-8

aromatic–aliphatic polyamides were prepared by polymerization of with different aliphatic/aromatic diacids containing additional ether/silicon/cardo cyclohexylidene and methylene units, using Yamazaki's phosphorylation method. The polymers had inherent viscosities in the range 0.35–0.84 dL/g. The polyamides were readily soluble in common aprotic polar solvents containing LiCl. The polyamides derived from diacids having bulky pendant group and cardo group showed increased solubility. Polyamides exhibited glass transition temperature in the range 166–298 °C whereas thermogravimetric analysis data indicated that all the polymers were stable up to 300 °C in nitrogen atmosphere.

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