

Synthesis and Characterization of New Aromatic Poly(amide-imide)s Derived from Bis(3-trimellitimidophenyl) Phenyl Phosphine Oxide and Various Aromatic Diamines

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New aromatic poly(amide-imide)s with high inherent viscosities were prepared by direct polycondensation reaction of diimide-diacid (**I**) and aromatic diamines using triphenyl phosphite in N-methyl-2-pyrrolidone (NMP)/pyridine solution containing dissolved CaCl_2 . The bis(3-trimellitimidophenyl) phenyl phosphine oxide (**I**) was readily obtained by the condensation reaction of bis(3-aminophenyl) phenyl phosphine oxide (BAPPO) with trimellitic anhydride. The resulting poly(amide-imide)s showed high thermostability. Their decomposition temperatures at 10% weight loss in nitrogen atmosphere were above 532 °C and the anaerobic char yield at 800 °C ranged from 56% to 74%. Almost all the poly(amide-imide)s showed high glass transition temperature above 233 °C by differential scanning calorimetry (DSC) measurements. These polymers were readily soluble in various organic solvents and by their casting into transparent, tough and flexible films can be easily achieved.

Keywords: Aromatic poly(amide-imide)s, Polycondensation, Characterization, Thermostability

INTRODUCTION

Aromatic polyimides are well accepted as high-performance polymer materials for their excellent mechanical strength and high thermal stability and balanced mechanical and electric properties [1-5]. Polyimides are mainly used in the aerospace and electronics industries in the form of film and moldings, but high melting points and insolubility in organic solvents limited their applications [6-9]. The initial method of overcoming the intractability of the polyimides utilized the poly(amic acid) stage, which could be fabricated into certain end-use forms by virtue of their good solubility in aprotic solvents [10]. Subsequent cyclodehydration of the poly(amic acid) resulted in the polyimide form of the end-use article.

While this process has been employed successfully for thin films, the problems associated with the necessary removal of volatiles make this technique unsatisfactory for other polyimides products such as thick laminated composites and compression molded materials. To overcome these problems, some copolymers poly(amide-imide)s (PAIs) have been introduced. The presence of amide groups in these copolymers can improve their solubilities and, in contrast to poly(amic acid)s solutions, their solutions are quite stable. Furthermore, poly(amide-imide)s are lighter in color than the corresponding polyimides. This class of polymers with bulky and kinked units in their backbones revealed a good compromise between thermostability and processability. Although poly(amide-imide)s are known as soluble, processable and high-temperature polymers, their glass transition temperatures (T_g) were somewhat lower than the corresponding polyimides. Aromatic PAIs can be prepared using imide-containing

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aromatic dicarboxylic acid and various aromatic diamines [11-16].

Phosphorus containing polymers are generally flame retardant. Other interesting features of phosphorus polymers include good adhesion to substrates, metal ion binding characteristics and increased polarity [17-19]. A wide variety of phosphorus-containing polymers have been synthesized in academic and industrial laboratories, but very few have been commercialized mostly due to the problems of cost and stability [17]. Phosphine, phosphinite, phosphonite, phosphate, phosphinate, phosphonate, phosphate and phosphine oxide are the general types of phosphorus moieties that have been incorporated into polymeric backbones. Among these, the phosphine oxide group possesses the advantage of having the hydrolytically stable P-C bond (compared to P-O-C) and the oxidatively stable P=O bond (compared to phosphine). Polymers containing phosphine oxide moieties typically display good flame resistance, high thermal oxidative stability, enhanced solubility, and improved miscibility and adhesion.

The introduction of triphenyl phosphine oxide unit into aromatic polymer backbone has been proven to improve the solubility in organic solvents while retaining high T_g [20]. Atomic oxygen (AO) resistant polymer technology development has recently focused on placing phenyl phosphine oxide groups into the backbone of aromatic polymers [21-25]. The polymers typically contain 3-7% phosphorus and exhibit high T_g s and high young's moduli. Studies have shown that polymers containing chemically bound phenyl phosphine oxide (PPO) units [21-25] as well as phosphazene groups [26,27] have significantly increased AO and oxygen plasma resistance. Various polymers containing a triphenyl phosphine oxide such as polyamide [28,29], polyimide [30], polyether [31], polycarbonates [32], polyaspartimides [20] and epoxies [33] have also been described in the literature.

In 1974, Yamazaki and co-workers [34,35] reported a procedure for the synthesis of aromatic polyamides, which involved the complexation of a dicarboxylic acid with triphenyl or diphenyl phosphite and pyridine in a solvent consisting of NMP containing LiCl. In our earlier studies [11], a number of poly(amide-imide)s were successfully synthesized from the direct polycondensation of imide rings containing diacids and aromatic diamines by using the

phosphorylation reaction. The present article describes a series of aromatic poly(amide-imide)s with good thermal stabilities and high T_g s, which were synthesized from bis(3-trimellitimidophenyl) phenyl phosphine oxide and various aromatic diamines by using triphenyl phosphite and pyridine as condensing agents. The obtained poly(amide-imide)s contain alternating (amide-amide)-(imide-imide) links in the macromolecule backbone. The solubilities and physical and thermal properties of the polymers are reported in this article.

EXPERIMENTAL

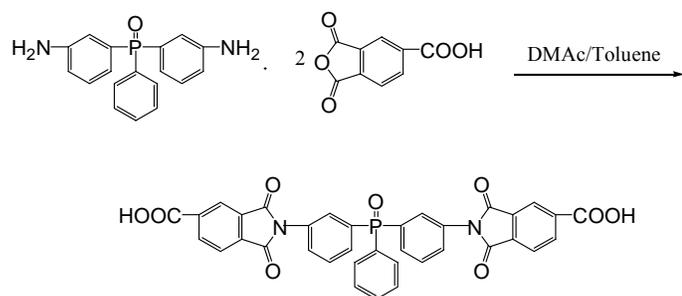
Materials

Chemicals were purchased from Merck and Fluka chemical companies. Trimellitic anhydride (with > 98% purity) and triphenyl phosphite (TPP) (with > 97% purity) were used as received. Reagent-grade calcium chloride was dried under vacuum at 180 °C before use. N-methyl-2-pyrrolidone (NMP), N,N-dimethylacetamide (DMAc), N,N-dimethylformamide (DMF) and dimethyl sulfoxide were purified by distillation under reduced pressure over calcium hydride and stored over molecular sieves 4°A. 4-Phenyl-2,6-bis(4-aminophenyl) pyridine was prepared by the condensation of *p*-nitroacetophenone, benzaldehyde and ammonium acetate, followed by the reduction of the intermediate dinitro compound [8]. Bis(3-aminophenyl)phenyl phosphine oxide (BAPPO) was prepared by nitration of triphenylphosphine oxide in the presence of H₂SO₄ and HNO₃ and subsequent reduction with 10% Pd-C and hydrazine hydrate [20].

Apparatus

¹H NMR and ¹³C NMR spectra were recorded on a Bruker Avance DPX-250 250 MHz spectrometer in DMSO-*d*₆ at 25 °C at 250.13 and 62.89 MHz for ¹H and ¹³C spectra, respectively, using TMS as an internal standard. FT-IR spectra were recorded on a Shimadzu FTIR-8300 spectrometer. The resolution of the spectrometer was 4 cm⁻¹. The IR spectra of solids were obtained using KBr pellets. Melting points were measured by a Buchi 535 melting point apparatus at a heating rate of 1 °C min⁻¹. Inherent viscosities were measured by a standard procedure using a Cannon Fensk routine viscometer in DMF at a concentration of 0.5 g dl⁻¹ at 30 °C. Thermo

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Diimide-diacid I

Scheme 1

gravimetric analysis (TGA) data for polymers were obtained using a Mettler TA4000 system under nitrogen atmosphere (valve $T/t_{1/2}$) at a rate of $10\text{ }^{\circ}\text{C min}^{-1}$ with the range of FS in 10 mg. Elemental analysis were performed with a Perkin-Elmer 2400 CHN analyzer. Ultraviolet λ_{max} values were obtained in DMSO (99.99%) at a concentration of 0.1 mg ml^{-1} at $25\text{ }^{\circ}\text{C}$ using a Shimadzu Multispec-1501 spectrometer.

Synthesis of Diimide-diacid (I)

Bis(3-aminophenyl) phenyl phosphine oxide (BAPPO) (6.16 g, 20 mmol) was dissolved in 15 ml of dried DMAc in a 50 ml flask. After BAPPO was dissolved completely, trimellitic anhydride (8.07 g, 42 mmol) was added to the resulting solution. The mixture was stirred at room temperature for 1h. Toluene (10 ml) was then added, and the mixture was heated at reflux for about 3 h until about 0.35 ml of water was distilled off azeotropically using Dean-Stark trap. After cooling, methanol (200 ml) was added and the precipitated product was isolated by filtration, purified by recrystallization from DMF-methanol solution, and dried in vacuum, to give diimide-diacid (I) (12.86 g 98% yield) as yellow solid $\text{mp} > 300\text{ }^{\circ}\text{C}$ (see Scheme 1). Diimide-diacid (I) showed the IR absorption bands (KBr) at 2420-3500 (acid-OH), 1780 (imide, symmetric C=O stretching), 1730 (acid C=O stretching and asymmetric imide C=O stretching). $^1\text{H NMR}$ (DMSO- d_6 , δ , ppm) bands at: 8.40, 8.37, 8.25, 8.05, 8.01, 7.93, 7.80, 7.78. $^{13}\text{C NMR}$ (DMSO- d_6 , δ , ppm): 166.38, 166.14, 162.63, 137.00, 135.80, 135.11, 134.00, 132.84, 132.60, 132.36, 132.31, 131.29, 130.44, 130.29, 124.12, 123.76 (see Fig. 1). Elemental analysis: calculated for $\text{C}_{36}\text{H}_{21}\text{N}_2\text{O}_9\text{P}$: C, 65.85; H, 3.22; N, 4.26; found: C, 65.80; H,

3.25; N, 4.20.

Synthesis of Model Compound (II)

In a flask equipped with a reflux condenser, a mixture consisting of NMP (4.0 ml) and calcium chloride (0.35 g) was prepared. Then diimide-diacid (I) (1 mmol, 0.656 g), aniline (2.2 mmol, 0.205 g), pyridine (1.2 ml) and TPP (0.8 ml) were added to the flask and the resulting mixture was refluxed for 3 h. After cooling, the reaction mixture was poured into methanol (500 ml) with constant stirring and the resulting precipitates filtered off and dried in vacuum to afford compound II, 0.797 g 99% yield, as a yellow solid to show UV λ_{max} , 292 nm, in DMSO and $\text{mp} > 300\text{ }^{\circ}\text{C}$. Model compound (II) showed IR (KBr) absorption bands at 1778 and 1722 (symmetric and asymmetric C=O stretching vibration), 1371 (C-N stretching vibration) and 1105 and 732 cm^{-1} (imide ring deformation). The absorption bands of amide groups appeared at 3388 (N-H stretch) and 1680 cm^{-1} (C=O stretch). $^1\text{H NMR}$ (DMSO- d_6 , δ , ppm): 10.52, 8.37, 8.19, 8.10, 7.94, 7.74, 7.33. Elemental analysis; calculated for $\text{C}_{48}\text{H}_{31}\text{N}_4\text{O}_7\text{P}$: C, 71.46; H, 3.87; N, 6.94; found: C, 70.80; H, 3.97; N, 6.70.

Synthesis of Poly(amide-imide)s, a Typical Procedure

A mixture of diimide-diacid (I) (1 mmol, 0.656 g), diamine (d) (1 mmol, 0.108 g), calcium chloride (1 g), triphenyl phosphate (2.6 mmol), pyridine (2 ml) and NMP (5.0 ml) was heated with stirring at $105 \pm 5\text{ }^{\circ}\text{C}$ for 3 h under nitrogen. The viscosity of the reaction mixture was increased after 1 h and an additional NMP (5.0 ml) was added to the reaction mixture. After cooling, the reaction mixture was poured into a large amount of methanol (500 ml) with constant stirring to isolate the desired polymer (PAId). The precipitated polymer was separated by filtration and washed with methanol (35 ml) and hot water (100 ml) and dried at $120\text{ }^{\circ}\text{C}$ under vacuum for 24 h. All poly(amide-imide)s were synthesized by a similar procedure. The yields were quantitative. Inherent viscosities, λ_{max} values, the results of elemental analyses and the yield of the obtained polymers are summarized in Tables 1 and 2.

RESULTS AND DISCUSSION

Monomer Synthesis

As shown in Scheme 1, the monomer was obtained by the

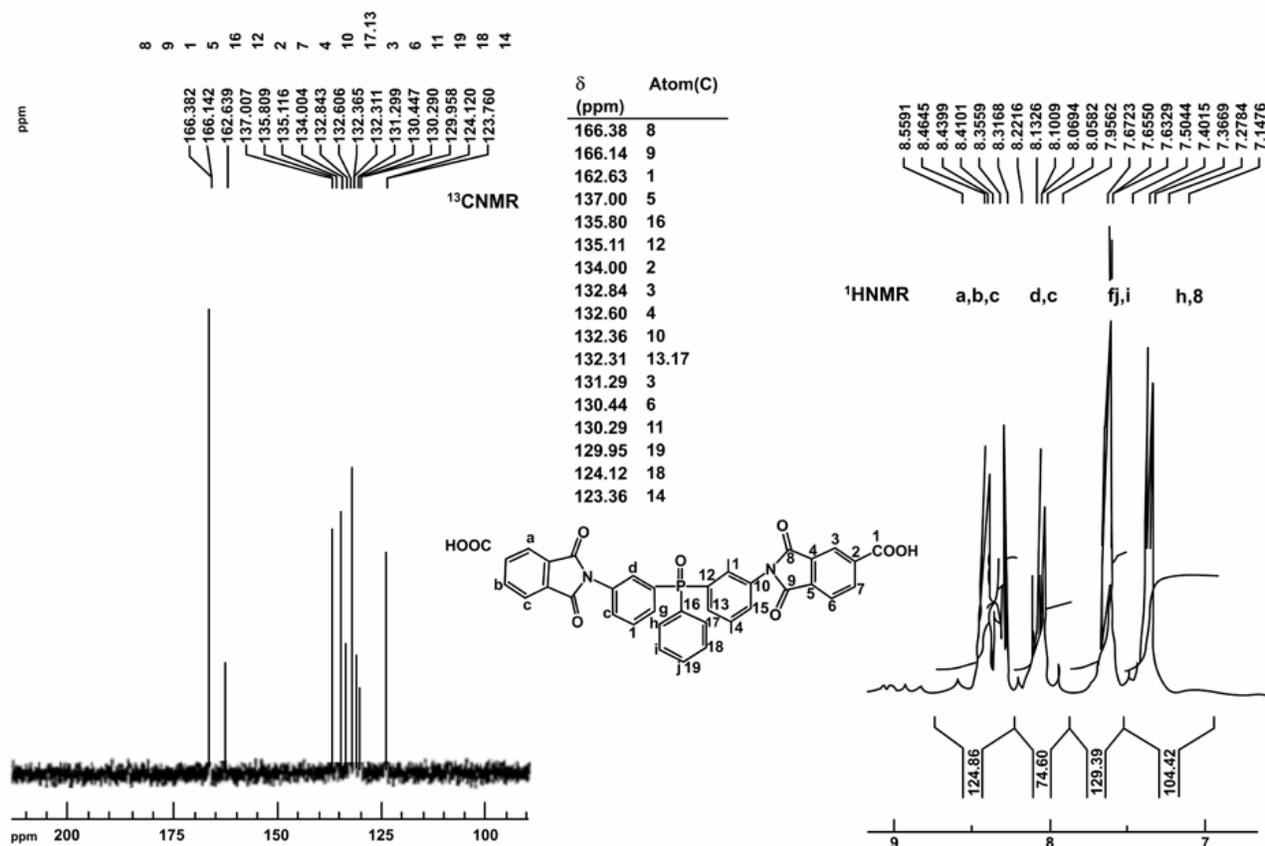


Fig. 1. NMR spectra of diimide-diacid (I).

condensation of diimide-diacid (I) with two molar equivalent of trimellitic anhydride in refluxing DMAc. The condensation reaction between the amines and anhydride groups, and the subsequent cyclodehydration reaction, were carried out in heterogeneous solutions. The structure of the monomer was confirmed by means of FT-IR and NMR spectroscopy and elemental analysis. FT-IR spectra of diimide-diacid (I) are presented in Fig. 2. The spectra showed absorption bands at 2420-3500 (acid-OH), 1780 (imide, symmetric C=O stretching), 1730 (acid C=O stretching and asymmetric imide C=O stretching) and 1380 cm^{-1} (imide ring vibration, axial), confirming the presence of imide ring and carboxylic acid group in the structure [11]. The formulation of diacid-diimide (I) is also confirmed by its NMR spectrum (Fig. 1). In the ^1H NMR spectrum, the protons $\text{H}_{a,b,c}$ adjacent to the carboxylic group or imide ring (ortho-oriented protons) and $\text{H}_{e,d}$ adjacent

to imide ring resonate at the downfield region, due to the inductive effect of the carboxyl group. While, the resonance of the imide ring, the $\text{H}_{h,g}$ adjacent to phosphin oxide group, are shifted to the upfield region, due to the inductive effect of the phosphin oxide group. The $\text{H}_{f,i}$ appeared at higher field with respect to the other protons available in the molecule. The protons of carboxylic acid groups did not show up in the ^1H NMR spectrum. This is probably due to the fast exchange of the protons with a trace amount of moisture associated with the solvent.

The ^{13}C NMR spectrum of diimide-diacid (I) shows 17 signals, including 3 signals of carbon atoms associated with carboxylic acid and imide ($\text{C}_{1,8,9}$) and 14 signals of carbon atoms present in the aromatic rings ($\text{C}_{2-7, 10-19}$). The expected signals for aromatic carbons are 15, while the actual NMR measurement showed 14 signals, which is due to overlap of

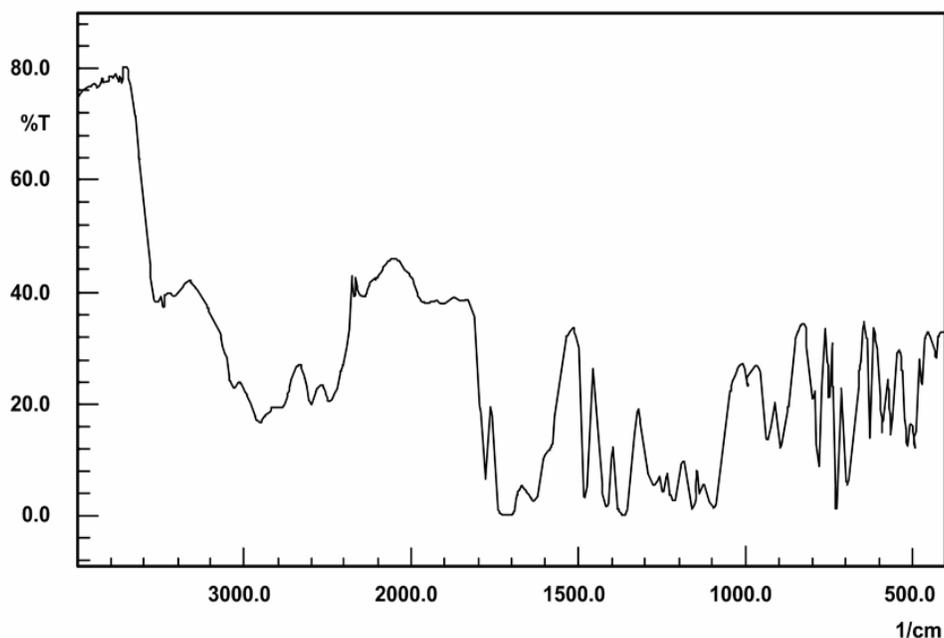
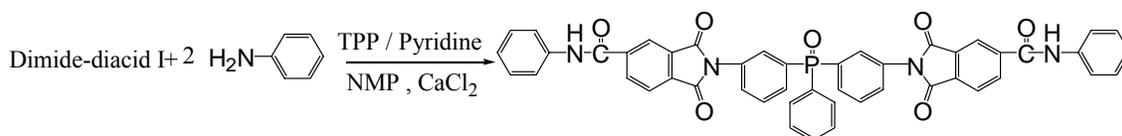


Fig. 2. IR spectrum of diimide-diacid I.



Scheme 2

two carbons bands (Fig. 1). The carbons C_1 , C_8 and C_9 of carboxy group and imide carbonyl groups appear at 166.38-162.63 ppm, and different shifts of C_8 and C_9 were caused by the asymmetric structure of termellitic anhydride. These spectra are in agreement with the predicted structures of the monomer.

The Model Compound (II)

As shown in Scheme 2, model compound (II) was synthesized from monomer I and aniline. The FT-IR spectrum of the model compound (II) is shown in Fig. 3. The spectrum exhibited characteristic absorption bands for the imide ring at 1778 and 1722 (asymmetric and symmetric C=O stretching vibration), 1371 (C-N stretching vibration) and 1105 and 732 cm^{-1} (imide ring deformation). The absorption of amide groups appeared at 3388 (N-H stretch) and 1664 cm^{-1} (C=O stretch) (Fig. 3). The ^1H NMR spectrum of the model

compound (II) shows the amide groups protons as the most downfield signal at 10.52 ppm, and the aromatic protons at 7.30-8.37 ppm. The λ_{max} of the model compound (II) was also determined, for comparison, which appeared at a λ_{max} of 292 nm.

Synthesis of New Polymers

New poly(amid-imide)s were prepared from diimide-diacid (I) and various aromatic diamines by a phosphorylation reaction with TPP, as a promoter, in NMP in the presence of pyridine and calcium chloride. Structure and codes of the synthesized polymers are shown in (Scheme 3). All polymerization proceeded homogeneously throughout the reactions. In some cases, additional amounts of NMP (2-6 ml) were added gradually to the reaction mixture in order to control the stirring. In general, the molecular weight of the

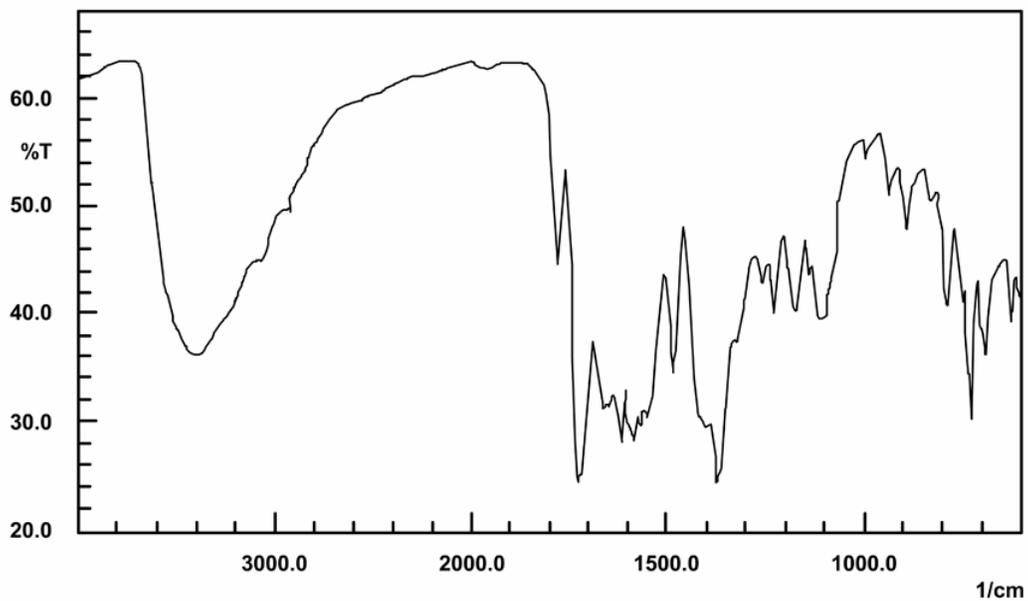
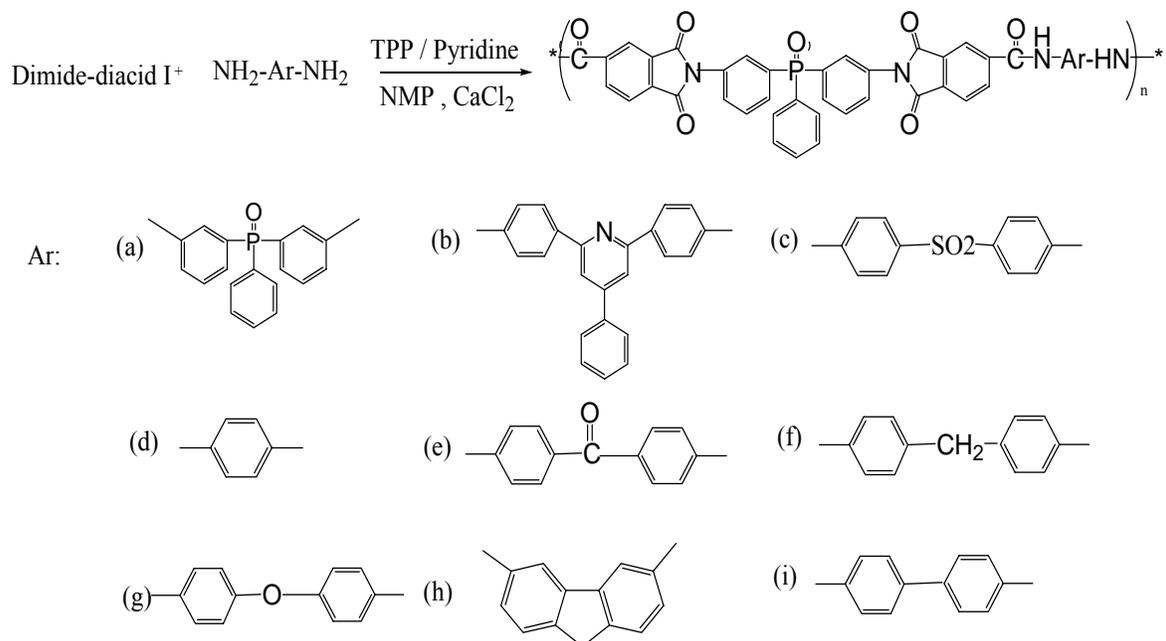


Fig. 3. IR spectra of model compound(II).



Scheme 3

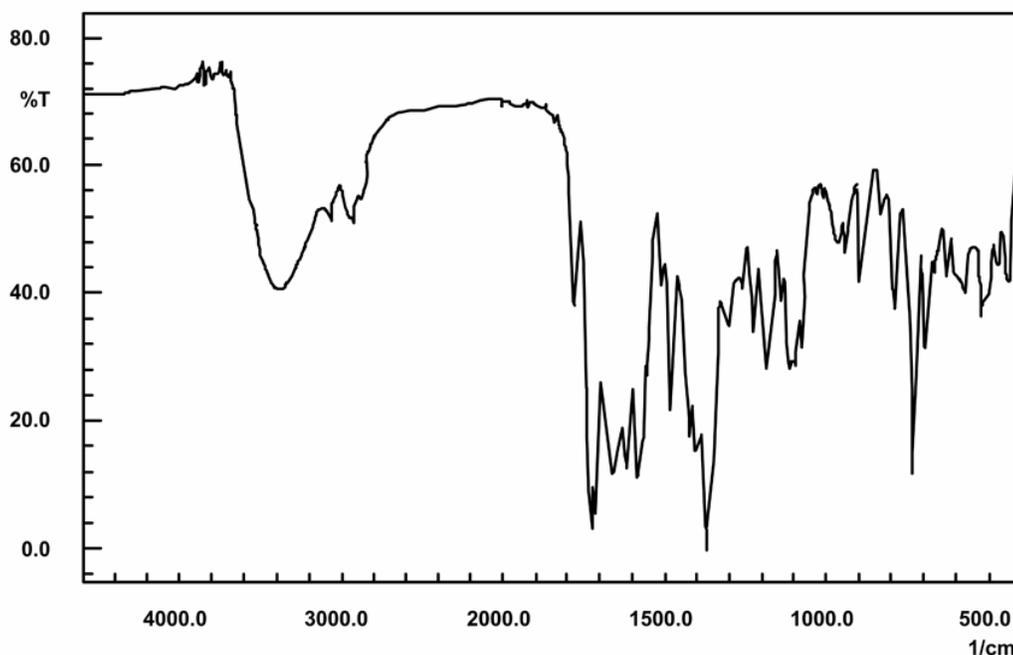

Fig. 4. IR spectra of PAIa.

Table 1. Synthesis and Some Physical Properties of Poly(amide-imide)s

Polymer	η_{inh}^a	$\lambda_{max} (nm)^b$	Yield (%)
PAIa	1.26	-	97
PAIb	1.30	300	99
PAIc	1.13	292	96
PAId	1.39	300	93
PAIe	1.18	292	95
PAIf	1.27	300	94
PAIg	1.32	292	95
PAIh	1.21	292	97
PAIi	1.34	300	94

^a Measured in DMF at a concentration of 0.5 g dl⁻¹ at 30 °C. ^b Obtained in DMSO at a concentration of 0.1 mg ml⁻¹.

resulted polymer was highly dependent on the monomer concentration during the initial period of the reaction and the amount of inorganic chlorides. High molecular weight polymers were obtained by the use of a higher initial reactant concentration and the addition of a proper amount of supplemental NMP into the viscous reaction media before the

formation of the swollen gel. In this study, no attempt was made to maximize molecular weight. Viscosities of the polymerization mixtures were normally very high, and the nature of the precipitated polymers implied high molecular weight products. As shown in Table 1, these polymers were obtained as yellowish materials in quantitative yields with inherent viscosities of up to 1.39 dl g⁻¹. The composition and structure of the resulting poly(amide-imide)s were characterized by their IR and NMR spectra and elemental analysis. A typical IR spectrum is shown in Fig. 4. The FT-IR spectrum of polymer PAIa exhibited characteristic absorption bands for the imide ring at 1778, 1734 (asymmetrical and symmetrical C=O stretching vibrations), 1367 (C-N stretching vibration), 1186, and 732 cm⁻¹ (imide ring deformations). The absorptions of amide groups appeared at 3377 (N-H stretch) and 1660 cm⁻¹ (C=O stretch). The inherent viscosities (η_{inh}) and ultraviolet maximum wavelengths (λ_{max}) for the polymers are given in Table 1. The results of the elemental analysis of all the poly(amide-imide)s are listed in Table 2.

In all cases, the carbon values found were lower than the calculated ones, most possibly due to the hygroscopic nature of the amide groups of the polymers. The water uptakes

Table 2. Elemental Analysis of Poly(Amide-Imide)s

Polymer	Moisture uptake ^a	Formula Mw		Elemental analysis ^b (%)		
				C	H	N
PAIa	4.65	(C ₅₄ H ₃₄ N ₄ O ₈ P ₂) _n	Calcd	69.82	3.68	6.03
				Found	66.11	3.87
PAIb	4.12	(C ₅₉ H ₃₆ N ₅ O ₇ P) _n	Calcd	73.97	3.78	7.31
				Found	71.08	3.95
PAIc	4.08	(C ₄₈ H ₂₉ N ₄ O ₉ PS) _n	Calcd	66.30	3.36	6.44
				Found	63.60	3.52
PAId	4.36	(C ₄₂ H ₂₅ N ₄ O ₇ P) _n	Calcd	69.23	3.45	7.68
				Found	66.30	3.61
PAIe	4.18	(C ₄₉ H ₂₉ N ₄ O ₈ P) _n	Calcd	70.67	3.51	6.72
				Found	67.85	3.65
PAIf	3.94	(C ₄₉ H ₃₁ N ₄ O ₇ P) _n	Calcd	71.87	3.81	6.84
				Found	69.13	3.95
PAIg	3.52	(C ₄₈ H ₂₉ N ₄ O ₈ P) _n	Calcd	70.24	3.56	6.82
				Found	67.82	3.68
PAIh	3.39	(C ₄₉ H ₂₉ N ₄ O ₇ P) _n	Calcd	72.05	3.57	6.85
				Found	69.67	3.70
PAIi	3.12	(C ₄₈ H ₂₉ N ₄ O ₇ P) _n	Calcd	71.64	3.63	6.96
				Found	69.45	3.73

^a Moisture uptake (%) = (W-W₀)/W₀ × 100%; W = weight of polymer sample after standing at room temperature, and W₀ = weight of polymer sample after drying in vacuum at 100 °C for 10 h. ^b For C and N: corrected value = found value × (100% + moisture uptake%). For H: corrected value = found value × (100% - moisture uptake (%)).

Table 3. Solubility of the Poly(amide-imide)s^a

No.	Solvent ^b						
	Conc. H ₂ SO ₄	NMP	DMAc	DMF	DMSO	<i>m</i> -Cresol	Pyridine
PAIa	++	++	++	++	++	+	+
PAIb	++	++	++	++	++	+	+
PAIc	++	++	++	++	++	+	+
PAId	++	+	+	+	+	-	-
PAIe	++	++	++	++	++	+	+
PAIf	++	++	++	++	++	+	+
PAIg	++	+	+	+	+	-	-
PAIh	++	++	++	++	++	+	+
PAIi	++	+	+	+	+	-	-

^a Concentration, 5 mg ml⁻¹; ++, soluble at room temperature; +, soluble after 60 °C heating; -, insoluble.

^b NMP, N-methyl-2-pyrrolidone; DMAc, dimethylacetamide; DMF, dimethylformamide; DMSO, dimethylsulfoxide.

Table 4. Thermal Behavior of Poly(amide-imide)s

Polymer	DSC		TGA
	T _g ^a (°C)	T ₁₀ ^b	Char yield ^c (%)
PAIa	262	670	62
PAIb	258	600	63
PAIc	241	590	74
PAId	263	650	66
PAIe	238	580	65
PAIf	233	532	62
PAIg	235	590	56
PAIh	248	547	57
PAIi	253	640	67

^a From the second heating traces of DSC measurements conducted with a heating rate of 10 °C min⁻¹.

^b Temperature at which 10% weight loss was recorded by TG at heating rate of 10 °C min⁻¹ under nitrogen atmosphere. ^c Char yield in TGA at 800 °C under nitrogen atmosphere.

ranged from 3.12-4.65%; these values were calculated from weight change of the vacuum-dried polymer samples after they were exposed at air at room temperature (ca. 27 °C) for 1 h.

Solubility

The solubilities of poly(amide-imide)s were tested qualitatively in various solvents. The results are shown in Table 3. All polymers showed excellent solubilities in aprotic polar solvents such as NMP, DMAc, DMF, and DMSO. In additions to highly polar solvents, they were also soluble or showed swelling in less polar solvents like *m*-cresol and pyridine. The high solubility of these poly(amide-imide)s was apparently due to the introduction of *m*-phenylene structure and phosphine oxide linkages, which resulted in increasing free volume between the polymer chains and decreasing intermolecular interactions.

Thermal Properties

The thermal properties of all the poly(amide-imide)s were evaluated by thermogravimetry (TG) and differential scanning calorimetry (DSC). The thermal behavior data of all polymers are listed in Table 4. DSC measurements were conducted with a heating rate of 10 °C min⁻¹ in nitrogen. The T_g values of the

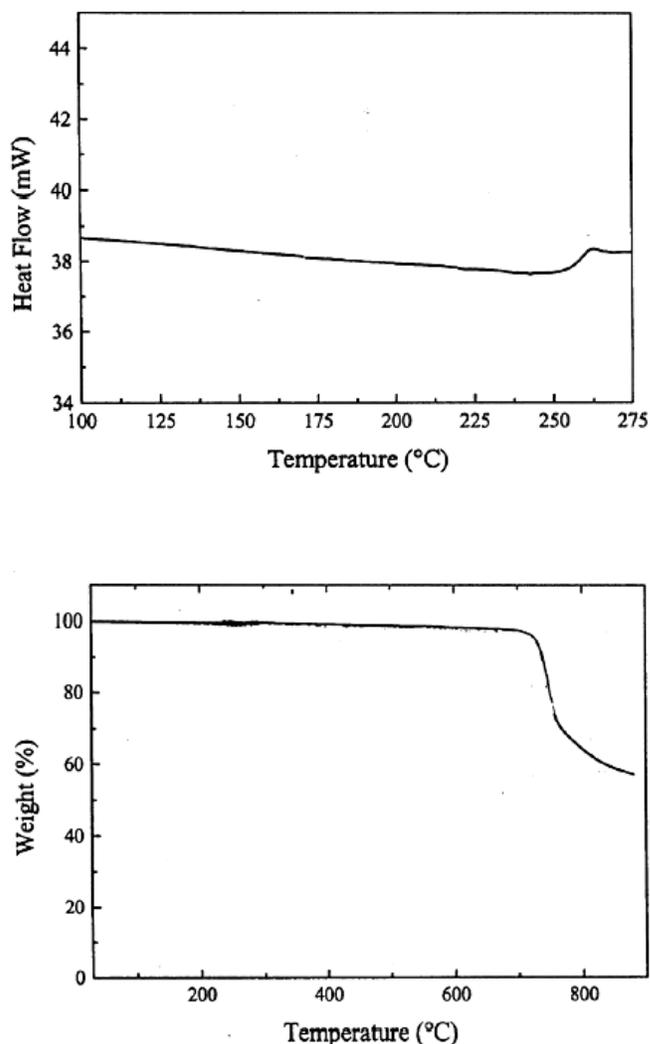


Fig. 5. DSC, TGA thermogram of PAIa at heating rate of 10 °C min⁻¹.

poly(amide-imide)s were in the range of 233-263 °C, depending on the structure of diamine component. Polymers containing the mono-phenyl group in diamine, which had higher rigidity, exhibited higher T_g values. Polymer having the polar sulfone groups in its backbone also showed high T_g values. The thermal stability of the poly(amide-imide)s was characterized by TG analysis conducted at a heating rate of 10 °C min⁻¹. The temperature of 10% weight loss (T₁₀) in nitrogen was determined from original thermograms. The T₁₀ values of these poly(amide-imide)s remained in the range of 532-670 °C. They left more than 56% char yield at 800 °C

(Fig. 5).

Finally, phosphine oxide-containing polymers exhibited high T_g , good thermal stability and good other mechanical properties in comparison to other polymers such as the polymer containing sulfone moieties in its main chain [36,37]. In addition, these PAIs show better processing characteristics than polyamide and polyimides with analogous structures [38,39].

CONCLUSIONS

A series of wholly aromatic PAIs containing phosphine oxide linkage were synthesized by direct polycondensation of diacid-diimide (**I**) with various aromatic diamines using TPP and pyridine as condensing agents. These polymers showed excellent solubilities and were characterized by excellent thermal stability as well as high glass transition temperatures. The yellow polymers had η_{inh} and λ_{max} values of 1.13-1.39 dl/g and 292-300 nm, respectively. Finally, these PAIs exhibit better processing characteristics than polyamides and polyimides with analogous structures.

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