Synthesis and Properties of Novel Flame-Retardant Poly(amide-imide)s Containing Phosphine Oxide Moieties in Main Chain by Microwave Irradiation

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ABSTRACT: Eight new flame-retardant poly(amide-imide)s with high inherent viscosities containing phosphine oxide moieties in main chain were synthesized from the polycondensation reaction of *N,N′*-(3,3′-diphenylphenylphosphine oxide) bistrimellitimide diacid chloride **7**, with eight ;aromatic diamine **8a–h** by two different methods such as solution and microwave-assisted polycondensation. Results showed that the microwave-assisted polycondensation by using a domestic microwave oven proceeded rapidly, compared with solution polycondensation and were completed within about 10–12 min. The resulting poly(amide-imide)s **9a–h** showed high thermal stability and flame-retardant properties. All of the obtained polymers were fully characterized by means of elemental analysis, viscosity measurements, solubility test, and FTIR spectroscopy. Thermal properties of the PAIs **9a–h** were investigated by using thermal gravimetric analysis (TGA), derivative thermogravimetric analysis (DTG), and differential scanning calorimetry (DSC). Char yield measurements at 600°C demonstrated that incorporating phosphine oxide moieties in polymer backbone markedly improves their flame retardancy. All of the earlier polymers were soluble at room temperature in various organic solvents such as NMP, DMF, DMSO, DMAc, and concentrated sulfuric acid. © 2006 Wiley Periodicals, Inc. J Appl Polym Sci 101: 4263–4269, 2006

Key words: flame-retardant polymers; phosphine oxide moieties; *N,N′*-(3,3′-diphenylphenylphosphine oxide) bistrimellitimide diacid chloride

INTRODUCTION

During the earlier decades, conventional materials such as metals, wood, glass, and ceramic have been increasingly replaced by synthetic polymers, due to their versatility, low density, good mechanical and physical properties, and easy molding process.[1,2] However, these advantages of polymeric materials are shadowed by easily free catching and low stability in high temperatures in comparison with metals. Since in the last few years, considerable attention has been paid for preparing flame retardant polymers.[3,4] Most of the flame retardant polymers were developed and among these, phosphorus containing polymers are the most widely used.[5–9] The general types of phosphorus moieties have been incorporated into different polymeric backbones such as epoxy resin, poly(amic-acid), polycarbonate, poly(vinyl chloride), polyester, polyimide, and poly(methyl methacrylate).[10–16] Among the polymers with phosphorus moieties, the polymers with phosphine oxide moieties have the major advantages, such as good flame retardant properties, high thermal oxidative stability, enhanced solubility in organic solvents, improved miscibility, and good adhesion to other compounds.[17–22]

In this research, we prepared a new series of flame retardant poly(amide-imide)s **9a–h** containing phosphine oxide moieties in main chain. We tested microwave-assisted polycondensation along with a solution polycondensation reaction between bis(3-aminophenyl) phenyl phosphine oxide diacid chloride **7** as a monomer with phosphine oxide moiety with eight aromatic diamine **8a–h**.

EXPERIMENTAL

Materials

All chemicals were purchased from Fluka Chemical (Switzerland), Aldrich Chemical (Milwaukee, WI), and Merck Chemical (Germany).

Techniques

^{13}C NMR spectra (500 MHz) was recorded on a bruker advanced 500 instruments (Germany). Fourier transform infrared (FTIR) spectra were recorded on Galaxy

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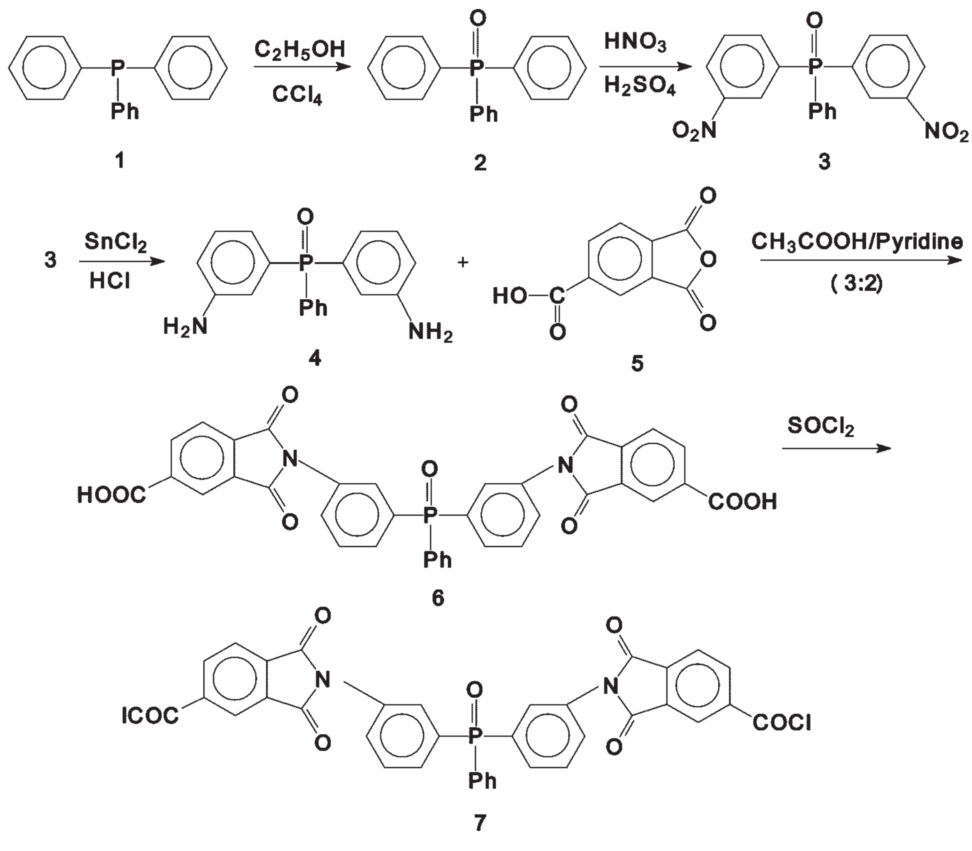


Scheme 1

series FTIR 5000 spectrophotometer (England). Spectra of solids were performed by using KBr pellets. Vibrational 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 Regd Trad Merk Viscometer. Thermal Gravimetric Analysis (TGA) and derivative thermogravimetric analysis (DTG) data for polymers were taken on a Mettler TA4000 System under N_2 atmosphere at a rate of 10°C/min. Differential Scanning Calorimetry (DSC) were recorded on a Stanton Redcraft STA-780 with heating rate 10°C/min in air. Elemental analyses were performed by the Arak Petrochemical Company, Arak, Iran. A Samsung domestic microwave oven (2450 MHz, 900 W) was used for carrying out polycondensation reactions.

Monomer synthesis

N,N′-(3,3′-diphenylphenylphosphine oxide) bistrimellitimide diacid chloride 7

This compound was prepared according to a typical procedure that was shown in Scheme 1.

Triphenyl phosphine oxide 2

Into a 100-mL round-bottomed flask (4.94 g, 20 mmol) of triphenyl phosphine **1**, 12 mL of absolute ethanol, 20 mL of tetrachloromethane, and a stirring bar were placed. The mixture was refluxed for 4 h. Then the solvent was removed under reduced pressure and the residue was added in 100 mL of cold water. White crystalline was formed, then the precipitate filtered off and dried, to give 4.24 g (81%) of compound **2**: mp 154–156°C, FTIR (KBr): 3074 (m), 3038 (m), 1674 (m), 1589 (m), 1483 (m), 1437 (s), 1180 (s), 1118 (s, br), 997 (w), 854 (w), 721 (s, br), 696 (s) cm^{-1}.

Bis(3-nitrophenyl) pheny phosphine oxide 3

Into a 250-mL round-bottomed flask equipped with a stirrer, triphenyl phosphine oxide **2** (27.8 g, 100 mmol) and a mixture of 200 mL of 96% sulfuric acid were added. The reactant was dissolved and the reaction mixture was cooled to −5°C with ice/salt bath. Then a solution of 14.5 g (22 mmol) of fuming nitric acid in 100 mL of sulfuric was added in a dropwise manner over 2 h, and the reaction system was maintained at room temperature for further 8 h. The reaction solution was then hydrolyzed over 2 L of ice. After the ice was melted, the mixture was extracted with chloroform and washed with aqueous sodium bicarbonate solution until the neutral pH and the solvent were removed. Finally, the solid residue was recrystalized with absolute ethanol and then 25.70 g (70%) of yellow crystals of bis(3-nitrophenyl) phenyl phosphine oxide **3** was obtained. mp 245–248°C, FTIR (KBr): 3082 (m), 2924 (w), 2882 (w), 1674 (w), 1608 (w), 1523 (s), 1467 (w), 1346 (s), 1278 (m), 1197 (s), 1140 (m), 1095 (m), 881 (m), 731 (m), 675 (s) cm^{-1}.

Bis(3-aminophenyl) pheny phosphine oxide 4

Bis(3-nitrophenyl) phenyl phosphine oxide **3** (15.4 g, 50 mmol) into a 500-mL round-bottomed flask with 144 g of powder tin(II) chloride was dihydrated to which a solution of 160 mL fuming hydrochloric acid in 320 mL ethanol was added. The reaction mixture was stirred at room temperature for 5 h, after which the solution was concentrated and then neutralized by a 25% NaOH aqueous solution. The obtained solution was then extracted with chloroform and the organic layer was collected and concentrated under reduced pressure. Finally, the obtained solid was dried under reduced pressure and then 9.41 g (73%) of yellow crystals of bis(3-aminophenyl) phenyl phosphine oxide **4** was obtained. mp 115–117°C, FTIR (KBr): 3325 (m), 3200 (m), 3070 (w), 1630 (m), 1593 (s), 1483 (m), 1437 (s), 1313 (m), 1276 (w), 1165 (s), 830 (w), 729 (m), 705 (s), 503 (m) cm^{-1}. Analysis: Calculated for $C_{18}H_{17}N_2OP$: C, 70.12; H, 5.56; N, 9.09; found: C, 69.71; H, 5.64; N, 8.91.

N,N′-(3,3′-diphenylphenylphosphine oxide) bistrimellitimide 6

Into a 250-mL round-bottomed flask (5.0 g, 16 mmol) of bis(3-aminophenyl) pheny phosphine oxide **4**, (7.3 g, 32 mmol) trimellitic anhydride **5**, 80 mL mixture of acetic acid and pyridine (3:2), and a stirrer bar were placed. The reaction mixture was stirred at room temperature for overnight and was refluxed for 4 h. 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 white yellow precipitate was formed, then the precipitate was filtered off and purified by recrystallization from DMF-H_2O solution and dried in vacuum to give 9.45 g (90%) of compound (6): mp 310–314°C, FTIR (KBr): 2700–3200 (m, br), 1780 (sh), 1724 (s, br), 1485 (m), 1425 (m), 1373 (s), 1219 (m), 750 (w), 727 (m) cm^{-1}. ^{13}C NMR (DMSO-d_6): δ; 166.84, 166.56, 162.24, 135.45, 134.03, 133.21, 133.14, 133.05, 132.93, 132.58, 132.36, 131.85, 130.74, 130.54, 129.91, 129.81, 128.06, 124.68, 124.31 ppm. Analysis: Calculated for $C_{36}H_{21}N_2O_9P$: C, 65.86; H, 3.22; N, 4.26; found: C, 65.70; 3.30; N, 4.00.

N,N′-(3,3′-diphenylphenylphosphine oxide) bistrimellitimide diacid chloride 7

Into a 100-mL round-bottom flask were placed *N,N′*-(3,3′-diphenylphenylphosphine oxide) bistrimellitimide **6** (6.56 g, 10 mmol) and 40 mL of thionyl chloride. The mixture was heated on an oil bath up to 60°C, until the suspension mixture was converted to a clear solution. Then, the solution was stirred for overnight at room temperature. Unreacted thionyl chloride was removed under reduced pressure, and the residue was washed with dry *n*-hexane two times. Then the precipitate was filtered off and purified by recrystallization from Tetrahydrofuran (THF) and dried in vacuum to give 6.6 g (90%) of yellow crystals of compound **7**. mp 213–215°C, FTIR (KBr): 1795 (sh), 1742 (s, br), 1510 (m), 1431 (m), 1375 (s), 1221 (m), 753 (w), 729 (m) cm^{-1}. Analysis: Calculated for $C_{36}H_{19}N_2O_7PCl_2$: C, 62.34; H, 2.76; N, 4.03; P, 4.47; found: C, 61.90; 2.90; N, 3.90; P, 3.80.

Polymer synthesis

Poly(amide-imide) **9a** was prepared from the reaction of *N,N′*-(3,3′-diphenylphenyl phosphine oxide) bistrimellitimide diacid chloride **7** with 1,4-phenylene diamine **8a** by two different methods:

Method A: solution polycondensation with pyridine as a base Into a 25-mL round-bottomed flask fitted with a stirring bar were placed 1,4-phenylene diamine **8a** (0.108 g, 1.0 mmol) and 9 mL of DMAc. The mixture was cooled in an ice-water bath, and pyridine (0.24 g, 3.2 mmol) was added to this solution. Then *N,N′*-(3,3′-diphenylphenylphosphine oxide) bistrimellitimide diacid chloride **7** (0.693 g, 1.0 mmol) was added all at once. The polymerization proceeded as the acid chloride was dissolved. The reaction mixture was stirred in an ice-water bath for 1 h, the cooling bath was removed, the stirring continued at room temperature for overnight, and then heated at 80°C for 12 h. The reaction mixture was poured into 50 mL of methanol. The precipitated polymer was collected by filtration, washed thoroughly with methanol, and dried at 80°C for 12 h under vacuum to leave 0.60 g (83%) of polymer **9a**.

Method B: polymerization by microwave irradiation in porcelain dish An equimolar mixture of *N,N′*-(3,3′-diphenylphenylphosphine oxide) bistrimellitimide diacid chloride **7** (0.693 g, 1.0 mmol) and 1,4-phenylene diamine **8a** (0.108 g, 1.0 mmol) was placed in a porcelain dish, and the mixture was grounded until fine powder was formed. Then 1 mL of *o*-cresol was added to the mixture and mixed up until homogeneous solution was formed. Then the reaction mixture was irradiated in microwave oven at full power for 10 min. The reaction mixture was poured into 50 mL of methanol, and the resulting polymer was filtered off and

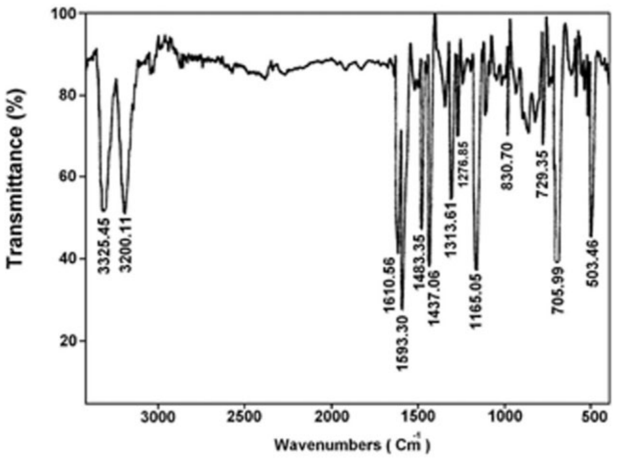


Figure 1 FTIR spectrum of diamine 4.

dried at 80°C for 12 h under vacuum to leave 0.70 g (96%) of solid polymer **9a**.

RESULTS AND DISCUSSION

Monomer synthesis

N,N′-(3,3′-diphenylphenylphosphine oxide) bistrimellitimide diacid chloride **7** was prepared in five-steps from simple organic compounds such as triphenyl phosphine **1**, as shown in Scheme 1. At first, a diamine compound **4** with phosphine oxide moiety was obtained, then the diimide-acid **6** was synthesized by condensation reaction of two equimolar amounts of trimellitic anhydride **5** with one equimolar amount of diamine compound **4** in a mixture of acetic acid–pyridine (3:2). After evaporation of solvent and dissolving the residue in cold water, a gummy-like solid was obtained that breaks up in a concentrate HCl and gives a white yellow solid when recrystallized with DMF-H_2O. Then the resulting diimide-acid **6** was converted to its diacid chloride derivative **7** by reaction with excess thionyl chloride. The diimide-acid chloride **7** was purified by washing with *n*-Hexan and recrystallizion with extra-pure tetrahydrofuran.

The chemical structures and purities of phosphorus containing diamine **4**, diimide-acid **6**, and diimide acid chloride **7** were also analyzed by elemental analysis, ^{13}C NMR, and FTIR spectroscopy. The measured results in elemental analyses closely corresponded to the calculated ones, demonstrating that the expected compounds were obtained. Figure 1 displays the FTIR spectra of bis(3-aminophenyl) phenyl phosphine oxide **4**. Peaks appearing at 3325 and 3200 cm^{-1} indicated the existence of amine groups (N—H stretching vibration). Other peaks at 1165 (P═O) and 1437 cm^{-1} (P—Ph) confirmed the presence of phosphine oxide moiety in this compound. FTIR spectra of diimide-acid **6** showed absorption bands at 2700–3200 (acid O—H stretching), 1780 (imide, asymmetric C═O stretching), 1724 (acid C═O and symmetric imide stretching), 1380, and 750 cm^{-1} (imide characteristic ring vibration) confirmed the presence of imide ring and carboxylic groups in this compound.

The ^{13}C NMR spectra of diimide-acid **6** showed 19 signals, including 3 signals of carbon atoms associated with carbonyl groups (C_1) in carboxylic acid and imide rings (C_8, C_9) and 15 signals of carbon atoms present in aromatic rings ($C_{2–7}$, $C_{10–19}$)(Fig. 2). These peaks in ^{13}C NMR spectra along with elemental analyses data confirmed the proposal structure of compound **6**.

In the FTIR spectra of compound **7** disappearing a broad peak at 2700–3200 cm^{-1} confirmed a complete conversion of diimide-acid **6** to diimide-acid chloride **7**. On the other hand, because of the electron withdrawing character of the Cl groups, two carbonyl peaks of the diimide-acid chloride **7** in comparison with starting diimide-acid **7** were shifted to a higher frequency (1810 and 1760 cm^{-1} in comparison with 1760 and 1720 cm^{-1}).

Polymer synthesis

PAIs **9a–h** were synthesized by microwave-assisted polycondensation as well as solution polycondensation reactions of an equimolar mixture of monomer **7** with eight different derivatives of aromatic diamine **8a–h**, as shown in Scheme 2. The microwave-assisted polycondensation reactions were carried out in a porcelain dish in the presence of a small amount of *o*-cresol. *o*-Cresol was used as a polar organic medium that acts as a primary microwave absorber and as a solvent for both of the starting monomers and the resulting polymers. This allowed the reaction mixture to be uniformly heated and thereby subsequent polycondensation reaction occurred, leading to the high

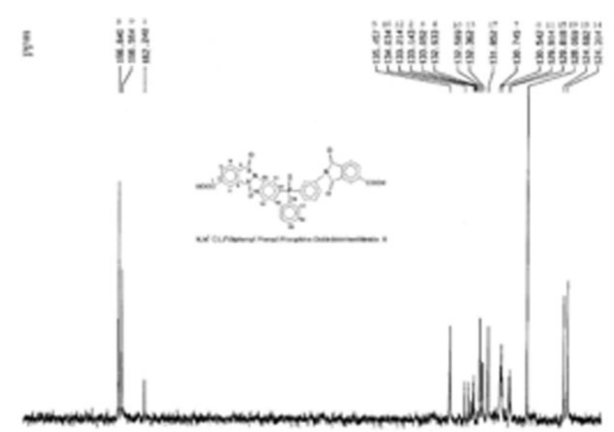
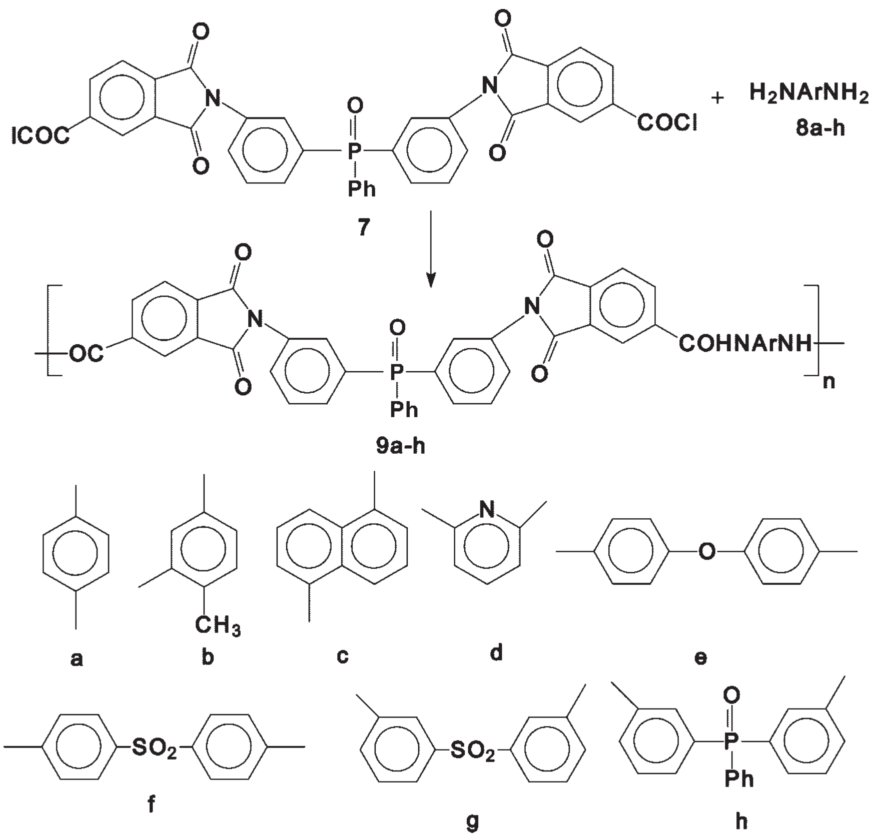


Figure 2 ^{13}C NMR spectrum of imide acid 6.



Scheme 2

yield polymer formation. The optimum reaction times (irradiation time) were found between 10 and 12 min. Less than this time, the polymers obtained will have lower inherent viscosities and more than this time, the materials will be degraded. The reaction yields and some physical data of polymers that were obtaind by this method were listed in Table I. Furthermore, we compared the microwave-assisted polycondensation method with conventional solution polcondensation method, and the polymerization reaction of diacid chloride **7** with eight different derivatives of aromatic diamine **8a–h** was performed in DMAc solution in the presence of pyridine as a base. Although we have obtained high yield of PAIs **9a–h**, lower inherent viscosities were resulted. Therefore, microwave-assisted polycondensation proceeded rapidly, and the products had higher inherent viscosities in a shorter period of time in comparison with solution polycondensation. The reaction yields and some physical data of the solution polycondensations were listed in Table II. PAIs **9a–h** derived from monomer **7** showed different colors from yellow to brown. As shown in Table I, these polymers were obtained in quantitative yields with inherent viscosities between 1.20 and 1.60 dL/g.

TABLE I
Synthesis and Some Physical Properties of PAIs (9a–h) by Microwave Irradiation

Aromatic diamine	Polymer	Yield (%)	Time (min)[a]	η_{inh} (dL/g)[b]
8a	**9a**	95	10	1.40
8b	**9b**	90	10	1.35
8c	**9c**	93	11	1.20
8d	**9d**	90	10	1.45
8e	**9e**	92	12	1.60
8f	**9f**	89	11	1.50
8g	**9g**	96	12	1.30
8h	**9h**	90	11	1.35

[a] Time of irradiation by microwave oven.
[b] Measured at a concentration of 0.5 g/dL in DMF at 25°C.

TABLE II
Synthesis and Some Physical Properties of PAIs (9a–h) by Solution Polycondensation

Aromatic diamine	Polymer	Yield (%)	η_{inh} (dL/g)[a]
8a	**9a**	81	1.10
8b	**9b**	80	0.75
8c	**9c**	82	0.80
8d	**9d**	80	0.69
8e	**9e**	85	0.60
8f	**9f**	79	0.75
8g	**9g**	83	1.30
8h	**9h**	86	1.35

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

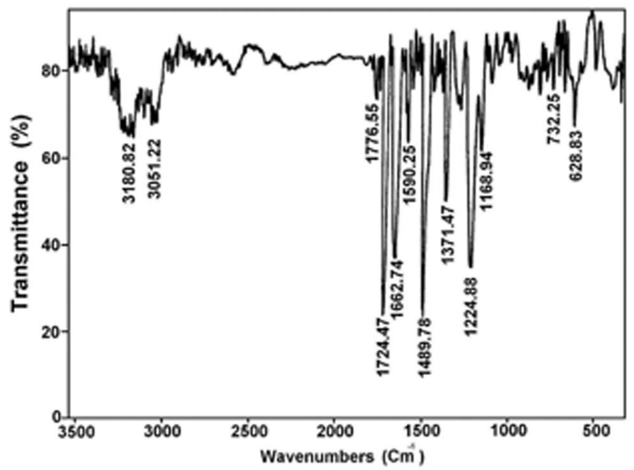


Figure 3 FTIR spectrum of PAI 9a.

The structures of these polymers were confirmed as PAIs by means of FTIR spectroscopy and elemental analyses. The representative FTIR spectrum of PAI **9a** was shown in Figure 3. The polymer exhibited characteristic absorption bands at 1776, 1724 cm^{-1} for the imide ring (asymmetric and symmetric C═O stretching vibrations), 13,717 cm^{-1} (C—N stretching vibration), 1168, and 732 cm^{-1}(imide ring deformations). The absorption bands of amide groups appeared at 3180 (N—H stretching) and 1662 cm^{-1}(C═O stretching).

The elemental analyses of the resulting PAIs **9a–h** were in good agreement with the calculated values for the proposed structures (Table III).

The solubility of PAIs **9a–h** was investigated as 0.01 g of polymeric sample in 2 mL of solvent. All of the polymers are soluble in organic solvents such as DMF, DMAc, DMSO, NMP, and acetone at room temperature, and are insoluble in solvents such as chloroform, methylene chloride, methanol, ethanol, and water. The high solubility of obtained PAIs 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 (Table IV).

TABLE III
Elemental Analysis of Poly(amide-imide)s 9a–h

Polymer	Formula		C%	H%	N%
9a	$C_{42}H_{25}N_4O_7P$	Calcd	69.23	3.45	7.68
	$(728.65)_n$	Found	67.80	3.00	6.60
9b	$C_{43}H_{27}N_4O_7P$	Calcd	69.55	3.63	7.54
	$(742.43)_n$	Found	68.10	2.95	6.7
9c	$C_{46}H_{27}N_4O_7P$	Calcd	70.96	3.46	7.19
	$(778.46)_n$	Found	68.90	2.90	6.10
9d	$C_{41}H_{24}N_5O_7P$	Calcd	67.50	3.29	9.59
	$(729.41)_n$	Found	66.00	3.70	8.30
9e	$C_{48}H_{29}N_4O_8P$	Calcd	70.26	3.53	6.82
	$(820.48)_n$	Found	68.90	2.90	6.00
9f	$C_{48}H_{29}N_9O_7SP$	Calcd	66.30	3.36	6.44
	$(868.81)_n$	Found	65.70	3.00	6.00
9g	$C_{48}H_{29}N_9O_7SP$	Calcd	66.30	3.36	6.44
	$(868.81)_n$	Found	65.20	2.95	5.50
9h	$C_{54}H_{34}N_4O_8P_2$	Calcd	69.82	3.68	6.03
	$(868.81)_n$	Found	67.90	3.10	5.40

TABLE IV
Solubility of PAIs (9a–h)

Solvent	9a	9b	9c	9d	9e	9f	9g	9h
H_2SO_4	+	+	+	+	+	+	+	+
DMAc	+	+	+	+	+	+	+	+
DMSO	+	+	+	+	+	+	+	+
DMF	+	+	+	+	+	+	+	+
NMP	+	+	+	+	+	+	+	+
THF	−	−	−	−	−	−	−	−
$CHCl_3$	−	−	−	−	−	−	−	−
Acetone	−	−	−	−	−	−	−	−
EtOH	−	−	−	−	−	−	−	−
MeOH	−	−	−	−	−	−	−	−

+, Soluble at room temperature.
−, Insoluble at room temperature.

Thermal properties

The thermal properties of three PAIs **9a**, **9c**, and **9h** were investigated by means of DSC as well as TGA and DTG in a nitrogen atmosphere at a heating rate of 10°C/min (Fig. 4).

All of these polymers showed similar decomposition behavior. Initial decomposition temperature, 5 and 10% weight loss temperature (T_5, T_{10}), and char yields at 600°C for three PAIs **9a**, **9c**, and **9h** were summarized in Table V. These polymers exhibited good resistance to thermal decomposition up to 290–330°C in nitrogen and began to decompose gradually above that temperature. The temperatures of 5% weight loss for all the polymers ranged from 295 to 335°C, and the residual weight at 600°C ranged from 18.0 to 38.0% in nitrogen.

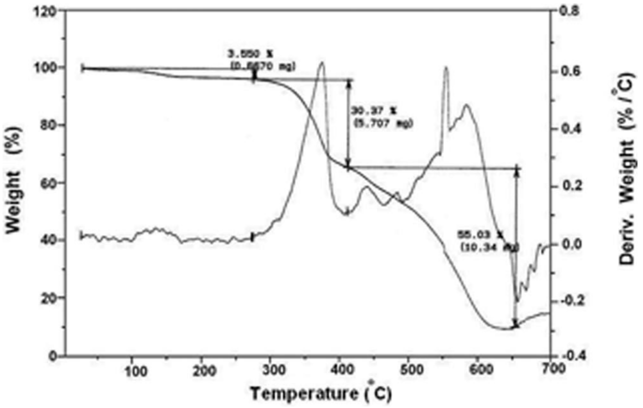


Figure 4 TGA and DTG thermogram of PAI 9a.

TABLE V
Thermal Behavior of PAIs (9a, 9d, and 9h)

Polymer	T_g	T_5 (°C)[a]	T_{10} (°C)[b]	Char yield[c]
9a	145	330–335	350–355	18.0
9d	156	290–295	315–320	39.0
9h	150	320–325	355–360	24.0

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

[c] Weight percentage of material left after TGA analysis at maximum temperature 600°C in N_2.

CONCLUSIONS

A new series of PAIS **9a–h** containing phosphine oxide moieties were synthesized by two different methods such as microwave-assisted polycondensation and solution polycondensation reactions of an equimolar mixture of monomer **7** with eight different derivatives of aromatic diamine **8a–h**. These polymers showed excellent solubilities and thermal stability characterized by TGA and DTG and DSC. The introduction of phosphine oxide moieties into the backbone of these polymers increased flame-retardancy properties. These properties can make these polymers attractive for practical applications such as processable high-performance engineering plastics. In addition, the earlier results demonstrate that microwave irradiation is a valuable optimization method, since it dramatically reduces reaction time and increases inherent viscosities of obtained polymers. Resulting polymers are soluble in various organic solvents. We are currently using this method for the synthesis of novel polymers and modification of polymers.

APPENDIX

Polymer 9a

FTIR (KBr): 3180 (w), 3051 (w), 1776 (w), 1724 (s), 1662 (s), 1590 (w), 1489 (s), 1371 (m), 1224 (s), 1168 (m), 850 (w), 725 (m), 628 (m) cm^{-1}.

Polymer 9b

FTIR (KBr): 3175 (w), 3067 (m), 1778 (w), 1724 (s, br), 1682 (m), 1593 (m), 1511 (s), 1475 (m), 1371 (sm), 1290 (m), 1140 (m), 750 (w) cm^{-1}.

Polymer 9c

FTIR (KBr): 3149 (w), 30,654 (w), 17,802 (wm), 17,224 (s, br), 1662 (m), 1593 (w), 1511 (m), 1483 (m), 1423 (s), 1371 (s), 1225 (m), 1160 (w), 725 (m) cm^{-1}.

Polymer 9d

FTIR (KBr): 3170 (w), 3067 (w), 1782 (w), 1724 (s, br), 1665 (m), 1591 (m), 1485 (s), 1425 (m), 1373 (s), 1215 (m), 1190 (w), 725 (m) cm^{-1}.

Polymer 9e

FTIR (KBr): 3180 (w), 3061 (w), 1776 (w), 1724 (s), 1662 (s), 1593 (w), 1498 (s), 1371 (m), 1224 (s), 1169 (m), 720 (w), 628 (m) cm^{-1}.

Polymer 9f

FTIR (KBr): 3088 (w), 3014 (w), 1782 (m), 1724 (s), 1682 (m, br), 1591 (s), 1510 (m), 1498 (m), 1371 (m), 1290 (m), 1147 (m), 752 (w) cm^{-1}.

Polymer 9g

FTIR (KBr): 3140 (w), 3059 (w), 1778 (w), 1724 (s), 1664 (s), 1593 (s), 1510 (s), 1479 (s), 1371 (s), 1303 (s), 1238 (m), 1153 (m), 725 (m), 690 (m) cm^{-1}.

Polymer 9h

FTIR (KBr): 3200 (w), 30,714 (w), 1778 (wm), 1725 (s, br), 1660 (sbr), 1590 (w), 1485 (s), 1436 (m), 1370 (m), 1223 (s), 1165 (m), 725 (m) cm^{-1}.

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