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New Thermally Stable Aromatic Polyimides Based on Aromatic Diamine 2,5-Bis(3-amino-4-methyl benzene)-1,3,4-oxadiazole (BAMO): Synthesis and Characterization

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In current study, the synthesis and characterization of novel thermally stable polyimides (PIs) containing 1,3,4-oxadiazole moiety based on a diamine, *i.e.* 2,5-bis(3-amino-4-methyl benzene)-1,3,4-oxadiazole (BAMO), have been reported. The polymers were characterized using FT-IR and elemental analysis (CHN). Thermal and mechanical behaviours of the prepared PIs were studied by thermo-gravimetric analysis/differential thermal analysis TGA/DTA, and dynamic mechanical thermal analysis (DMTA). PI (4) obtained from BAMO and 4,4'-(hexafluoroisopropylidene)diphthalic anhydride (6FDA) exhibited the best thermal and mechanical properties.

Keywords: Polyimide, 1,3,4-Oxadiazole, Mechanical properties, Thermal properties

INTRODUCTION

The synthesis of aromatic polyimides (PIs) was firstly reported by Marston Bogert in 1908. In 1955, high molecular weight aromatic PIs were synthesized by a two-stage polycondensation of pyromellitic dianhydride with diamines [1]. Polyimides were then developed in the early 1960s and have since been of great technological importance due to their outstanding thermal and electrical properties [2,3]. One of the directions in the development of new PIs is the synthesis and study of extended-rod or rigid rod-like aromatic PIs [4]. Linear aromatic PI film is a material of choice for use in the areas of modern industries [5]. Aromatic PIs are well-known as highly thermally and thermo-oxidatively stable polymers with a desirable balance of physical and chemical properties. This means that they can be used at high continuous temperatures [6]. They also possess outstanding mechanical, optical and good insulation properties with low dielectric constant as well as good chemical resistance [7-9]. Because of their inherent toughness and flexibility, low density, remarkable thermal stability, radiation resistance and mechanical strength, aromatic PIs are the most useful super engineering plastics with wide industrial applications [10]. They have found their way into cell processing [11], aerospace [12,13], optoelectronic applications [14], gas separation [15,16], coating [17], superhydrophobic surfaces [18], photocatalyst for hydrogen generation [19] and composites [20,21].

As a main drawback, rigidity of the stiff backbone structure leads to glass transition (T_g) and melting temperatures ranging from 200 to above 400 °C and limits solubility in most organic solvents. For this reason, a two-step procedure is widely used in which the soluble polyamic acid resulting from the reaction of an aromatic dianhydride with an aromatic diamine is processed from solution into thin materials such as films, coatings or impregnated fabrics. These materials then undergo a prolonged thermal treatment at high temperatures (300 °C) to afford the fully imidized chemical structure of the polymer [22].

It was shown that the incorporation of 1,3,4-oxadiazole moieties into the polymer structure raises the thermal stability [23]. Aromatic poly(1,3,4-oxadiazole)s are a class of heterocyclic polymers possessing chemical and thermal stability. These polymers are classified as high-performance

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polymers with excellent mechanical strength and stiffness. The outstanding thermal stability is ascribed to the electronic equivalency of the oxadiazole ring to the phenylene ring structure, which has high thermal-resistance [24]. These features cause polymers containing 1,3,4-oxadiazole moieties to act as alternatives in the development of heat flame-resistant, semi-conducting, fiber-forming and thermally stable membranes for gas separation [24-27].

As a part of our continuing efforts on synthesis of new polymers with high thermal stability, we have previously reported the synthesis of a new aromatic diamine, *i.e.* 5-(5-pyridin-2-yl)-1,3,4-oxadiazole-2-yl)-benzene-1,3-diamine (POBD), based on 1,3,4-oxadiaxole ring. It has been used for preparing of new thermally stable poly(amide-imide)s [28], polyamides [28], polyimides [29] and polyimide/clay nanocomposites [30]. Recently, we have also reported the synthesis and using of 5-bis-(aminopyridine-2-yl)-1,3,4-oxadiazole (BAPO) and 1,4-bis((4-aminophenyl)-1,3,4-oxadiazolyl)benzene (BAOB), other aromatic oxadiazole-containing diamines [31-33]. The present article aims at the synthesis of PIs based on an aromatic diamine (BAMO) with enhanced solubility with respect to our previously reported PIs [32]. The obtained polymers were characterized by spectroscopic methods and elemental analysis (CHN). Thermal and mechanical behaviors of the PIs were investigated by TGA/DTA and DMTA methods.

EXPERIMENTAL

Instruments

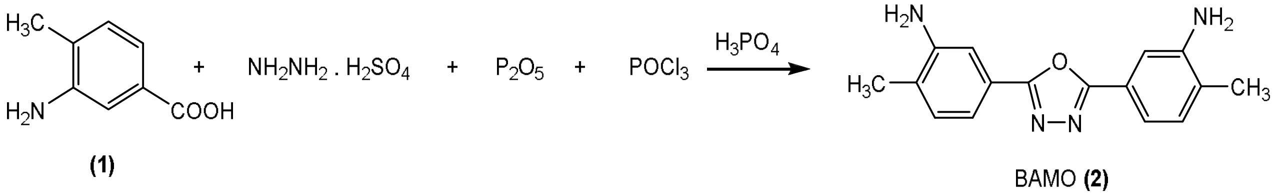
NMR spectra were obtained on a Bruker 300 MHz spectrometer. The FT-IR (KBr) spectra were recorded on a PerkinElmer RXI spectrophotometer (2w/w% in KBr, resolution 4 cm^{-1}, scan no. 6). The mass spectrum (electron impact, 70 eV) was obtained on an Agilent Technologies 5973Network MSD mass spectrometer. DMTA and TGA-DTA thermograms were measured on Triton Tritec 2000 DMN and Linseis STA PT 1000 instruments under nitrogen atmosphere and a ramping rate of 10.0 °C min^{-1}, respectively. Elemental analyses (CHN) of the samples were performed on an Elemental Vario EL III instrument. Inherent viscosities were measured by an Ostwald viscometer at 25 °C in DMSO.

Materials

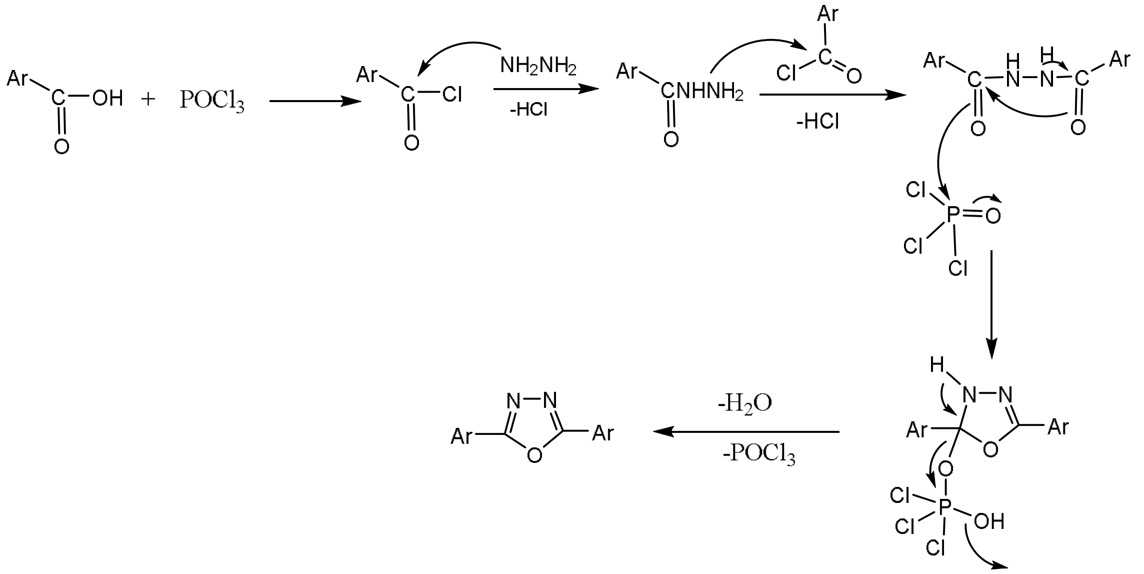
3-Amino-4-methylbenzoic acid (1) and all other chemicals were of laboratory grade and obtained from Merck (Germany). All solvents were of laboratory grade and dried according to procedures described in the literature [34].

Synthesis of 2,5-bis(3-amino-4-methyl benzene)-1,3,4-oxadiazole, BAMO (2). The diamine, BAMO, was synthesized under anhydrous condition according to the method described in the literature [31,35]. To the stirred solution of 3-amino-4-methylbenzoic acid (1) (1.510 g, 0.01 mol) and hydrazine sulfate (0.650 g, 0.005 mol) in an aqueous solution of phosphoric acid (85%, 2.7 ml), phosphorus pentoxide (P_2O_5) (4.260 g, 0.03 mol) was added slowly followed by addition of phosphoryl chloride ($POCl_3$) (0.9 ml, 0.01 mol). The viscous solution was heated at 170 °C for 2 h while magnetic stirring. After cooling to room temperature, the reaction mixture was poured over crushed ice with vigorous stirring. The mixture was neutralized with 10% sodium hydroxide solution to pH 6, and then completely neutralized with 5% solution of sodium hydrogen carbonate and filtered in vacuum. The precipitate was then stirred in 10 ml of distilled water for 30 min, filtered in vacuum and then dried to give 2.550 g (91.0% yield) of a pale yellow solid product. m.p.: 273-275 °C (273 °C; measured by DTA). FT-IR (KBr, cm^{-1}): 3405 (s), 3341 (s), 3231 (s), 3022 (w), 2970 (w), 1643 (s), 1584 (s), 1493 (s), 1002 (m), 801 (m), 734 (s). ^1H NMR (300 MHz, DMSO-d_6, δ, ppm): 7.31 (s, 2H, Ar-H); 7.11 (s, 4H, Ar-H); 5.19 (br, s, NH_2, 4H, exchanged with D_2O); 2.09 (s, 6H, Me). ^{13}C NMR (75 MHz, DMSO-d_6, δ, ppm): 164.6, 147.8, 131.2, 125.9, 122.0, 114.6, 111.5, 18.0. Mass: $(m/e)^+$: 280 (28.6%).

General procedure for the preparation of PI films (3-7). To a solution of BAMO (0.1752 g, 0.625 mmol) in 2.5 ml of N,N'-dimethylacetamide (DMAc) was added gradually dianhydride (0.625 mmol). The mixture was stirred at room temperature for 6 h under argon. The obtained DMAc solution of poly(amic acid) was poured onto a glass plates, and then heated from 60 to 270 ºC (at a heating rate of 1 ºC min^{-1}) in a furnace. The films were then



Scheme 1. Synthesis of 2,5-bis(3-amino-4-methyl benzene)-1,3,4-oxadiazole, BAMO



Scheme 2. The proposed mechanism for the cyclo-dehydration of carboxylicacids

peeled off from the glass plates.

RESULTS AND DISCUSSION

The diamine, BAMO, was synthesized in one-step through cyclo-dehydration of 3-amino-4-methylbenzoic acid (1) with hydrazine sulfate. The reaction was carried out in the mixture of P_2O_5, $POCl_3$ and concentrated phosphoric acid as dehydration medium, Scheme 1.

The proposed mechanism for cyclo-dehydration reaction is shown in Scheme 2 [35]. The chemical structure of BAMO was confirmed by FT-IR, ^{1}H NMR, ^{13}C NMR and mass spectrometry techniques. In the FT-IR spectrum (Fig. 1), amino stretching vibrations were observed at 3405, 3341 and 3231 cm^{-1}. The stretching vibrations of aromatic and aliphatic C-H bonds were observed at 3022 and 2970, respectively. Vibration of C=N bond of oxadiazole ring was appeared at 1643 cm^{-1}.

^{1}H NMR and ^{13}C NMR spectra of BAMO are shown in Fig. 2. The amino protons were merged to appear as a broad singlet peak centered at 5.19 ppm in the ^{1}H NMR spectrum, (this peak was disappeared upon addition of D_2O and a new peak related to HOD was appeared at 3.90 ppm). In Figure 2b, the eight signals related to eight non-symmetrical carbons of BAMO are in accordance with its structure. In the mass spectrum, the molecular ion peak with 28.6% abundance appears at m/e = 280, Fig. 3.

Poly(amic acid) is the requisite intermediate for preparing PIs [36]. In this work, the new diamine BAMO reacted with aromatic dianhydrides in DMAc at room temperature to give the corresponding poly(amic acid)s. PI films were then obtained via thermal imidization of poly(amic acid) solutions, Scheme 3.

In order to find the optimum time to reach the maximum

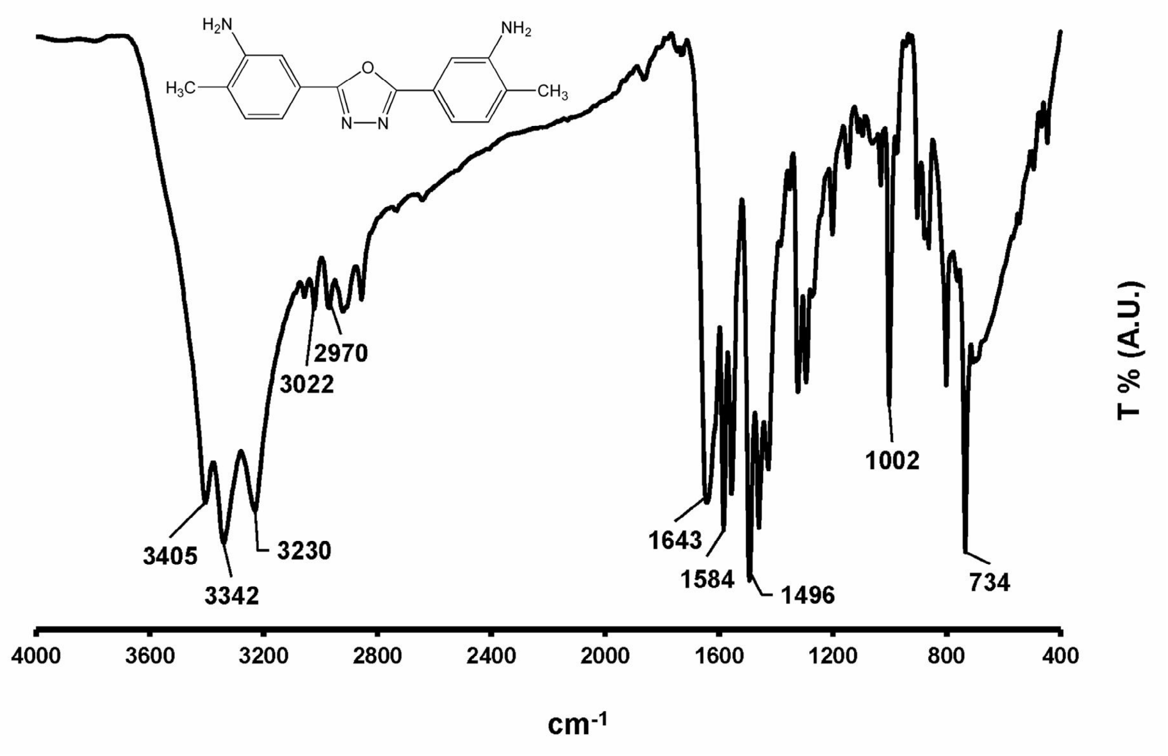


Fig. 1. FT-IR spectrum (KBr) of BAMO.

viscosity, inherent viscosity of the polyamic acid solution, obtained from BAMO and benzophenone-3,3′,4,4′-tetracarboxylic dianhydride (BTDA), was measured in N,N'-dimethylformamide (DMF) at different reaction times (with concentrations of 0.125 g dl^{-1} and 0.25 g dl^{-1} at 25 ± 0.5 °C). The results are shown in Fig. 4. As seen, the maximum viscosity was achieved within 6 h, and this time was chosen for all polymerization reactions.

The FT-IR absorptions appearing at approximately 1778-1789 cm^{-1}, 1717-1726 cm^{-1}, 1351-1374 cm^{-1}, 1092-1108 cm^{-1} and 724-747 cm^{-1} indicated the presence of imide functional groups in the PI films, Fig. 5. The conversion from poly(amic acid) to PI was confirmed by the presence of these strong imide absorption bands and the absence of a broad absorption band due to the amino (N-H) and hydroxyl (OH) groups [37]. Infrared spectroscopy data, inherent viscosities, and reaction yields of the obtained PIs are listed in Table 1.

Solubility test results (Table 2) showed that the obtained PIs are soluble in concentrated sulfuric acid at both ambient and elevated temperatures. Some of them are also soluble in polar organic solvents such as dimethylsulfoxide (DMSO), N-methylpyrrolidone (NMP), DMAc, and DMF at boiling temperatures. For this experiment, about 0.020 g of the polymer sample was examined in 1 ml of the solvent at room temperature and at boiling temperature. As can be seen, PIs (3) and (4) show the best solubility among the prepared polymers. The enhanced solubility of PI (4) can be attributed to the increased polarity coming from very polar C-F bonds [38].

The inherent viscosities of the PI solutions were in the range of 0.79-0.96 dl g^{-1} and 0.37-0.42 dl g^{-1} as measured in concentrated sulfuric acid with a concentration of 0.125 g dl^{-1} and 0.25 g dl^{-1} at 25 ± 0.5 °C, respectively. The highest viscosity was noted with PI (7), obtained from the reaction of BAMO with pyromellitic dianhydride.

The elemental analysis (CHN) data are presented in Table 3. The calculated and found values for the CHN analyses were in agreement after applying a correction for moisture intake.

The results of thermal and mechanic analysis of the polymers are summarized in Table 4. TGA-DTA and DMTA

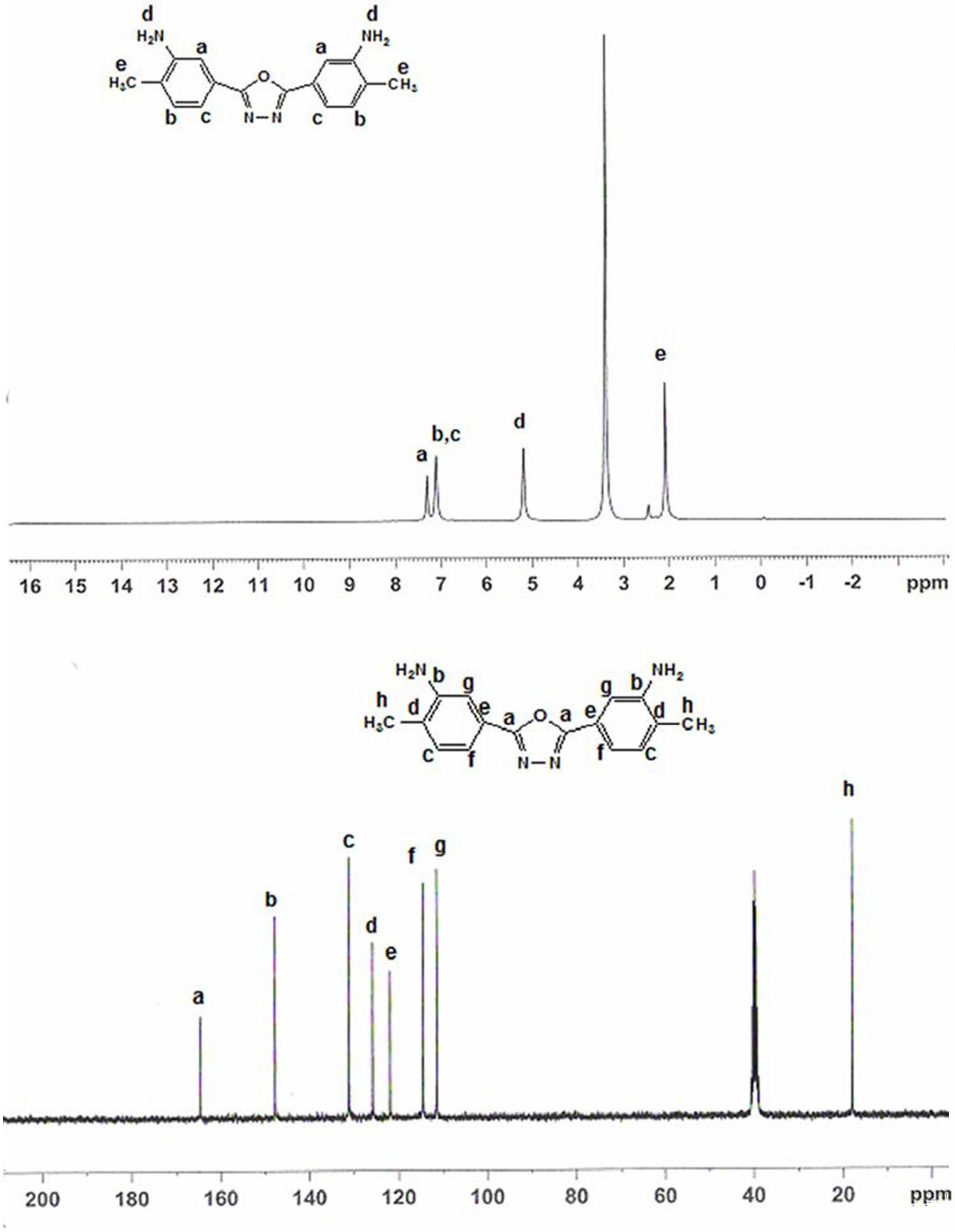


Fig. 2. ^{1}H NMR (300 MHz) and ^{13}C NMR spectra (75 MHz) of BAMO in DMSO-d$_6$.

techniques were used to study the thermal and mechanical behaviors of the obtained PIs. As seen, PI (4), obtained from BAMO and 6FDA, shows T_g at 339.4 °C as measured by DMTA. Based on the thermal stability parameters, the polymer has more improved thermal properties than those of PI (3), obtained from BAMO and BTDA. The high

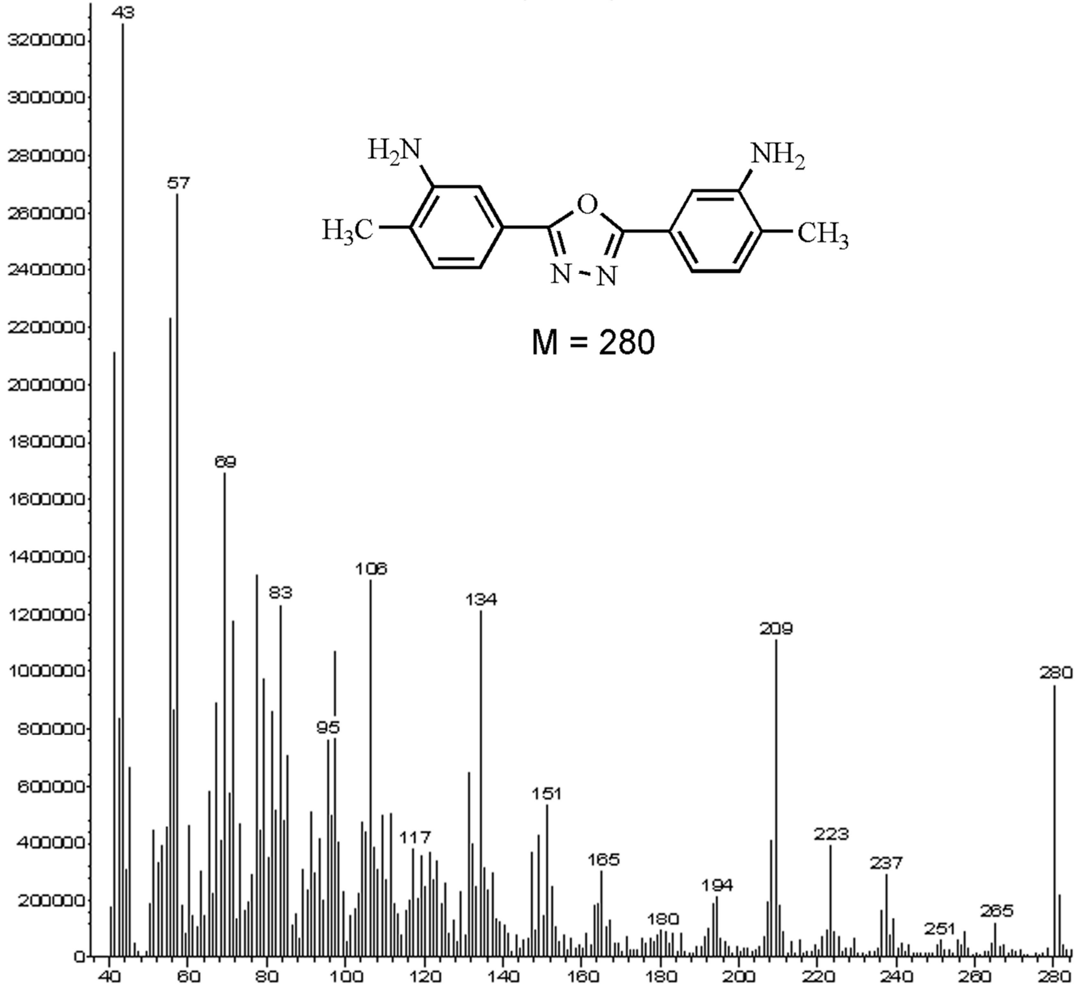
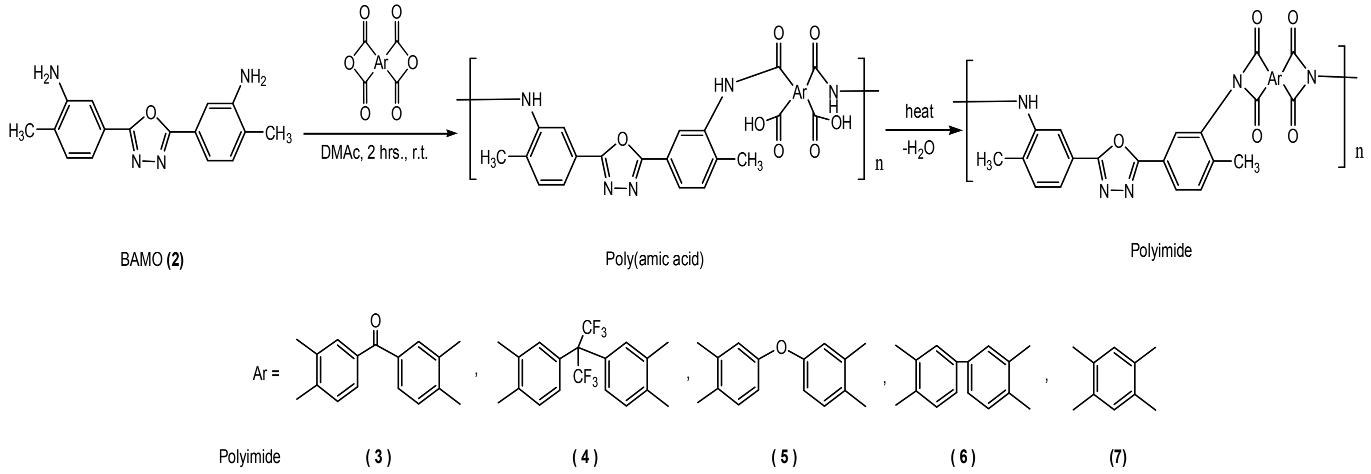


Fig. 3. Mass Spectrum of BAMO.



Scheme 3. Synthesis of polyimides using BAMO

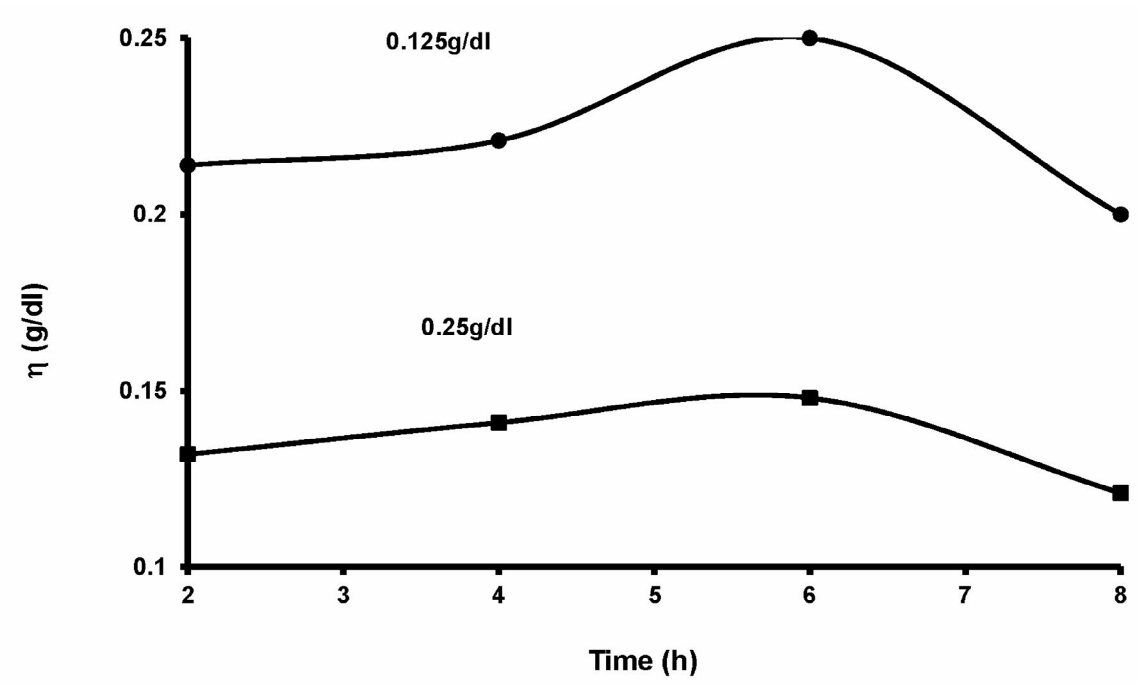


Fig. 4. Time dependence of the poly(amic acid) solution obtained from BAMO and BTDA.

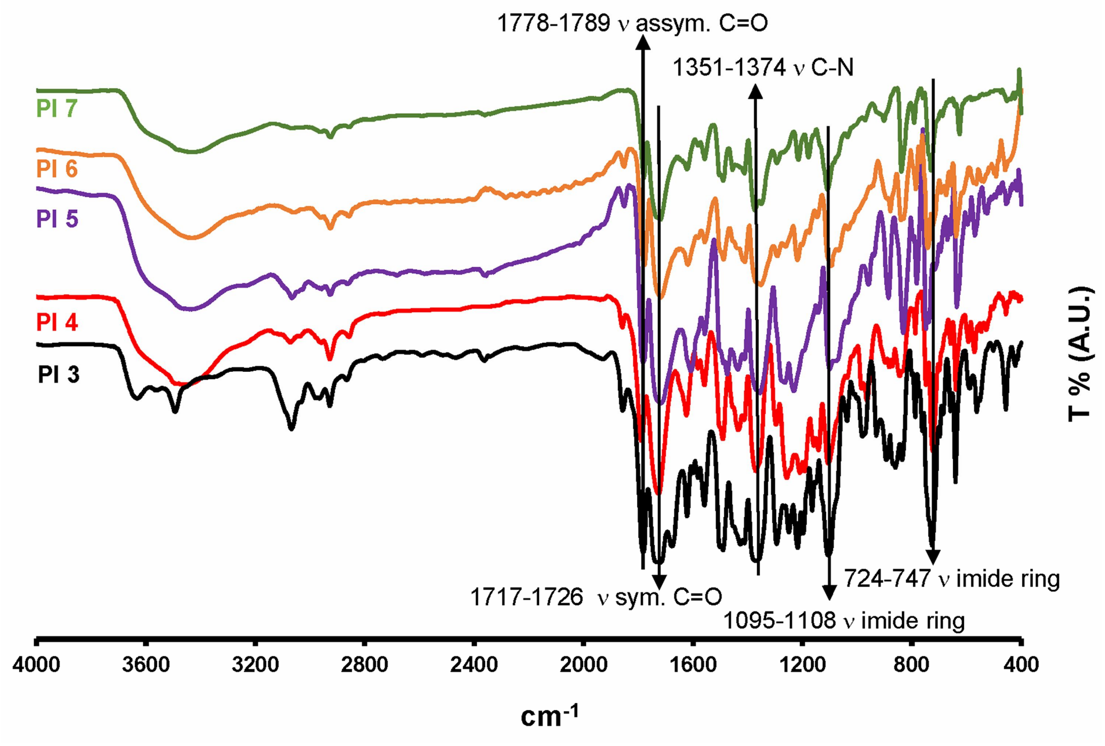


Fig. 5. FT-IR spectra (thin film) of the synthesized PIs.

Table 1. Infrared Spectroscopy, Inherent Viscosity Results, and Yields of the Polyimides

Polymer	FT-IR data (cm^{-1})	Inherent viscosity η_{Inh} ($dl\ g^{-1}$)[a]	 η_{Inh} ($dl\ g^{-1}$)[b]	Yields (%)
3	3067 (w[c]), 2926 (m[d]), 1782 (m), 1725 (s[e]), 1671 (w), 1359 (m), 1105 (m), 724 (m)	0.76	0.37	91.2
4	3072 (w), 2927 (m), 1789 (m), 1726 (s), 1624 (m), 1370 (s), 1105 (m), 724 (s)	0.77	0.39	91.5
5	3067 (w), 2927 (w), 1782 (m), 1717 (s), 1608 (m), 1355 (m), 1100 (m), 747 (m)	0.85	0.38	90.8
6	3063 (w), 2926 (m), 1778 (m), 1717 (s), 1618 (m), 1351 (s), 1092 (m), 742 (m)	0.90	0.43	92.1
7	2924 (w), 1779 (m), 1719 (s), 1621 (w), 1374 (s), 1108 (m), 730 (m)	0.96	0.42	91.3

[a] Measured in concentrated H_2SO_4 with a concentration of 0.125 dl g^{-1} at 25 ± 0.5 °C. [b] Measured in concentrated H_2SO_4 with a concentration of 0.25 dl g^{-1} at 25 ± 0.5 °C. [c] Peak with weak intensity. [d] Peak with medium intensity. [e] Peak with strong intensity.

Table 2. Solubility of the Prepared Polyimides in Common Organic Solvents

Polymer	3		4		5		6		7	
Solvent	R.T.	Δ	R.T.	Δ	R.T.	Δ	R.T.	Δ	R.T.	Δ
DMSO	--	-+	-+	++	-+	-+	--	-+	--	-+
DMF	-+	++	-+	++	-+	++	--	-+	--	-+
DMAc	-+	++	-+	++	--	-+	-+	++	--	-+
NMP	-+	++	-+	++	-+	++	-+	++	-+	++
MeOH	--	--	--	--	--	--	--	--	--	--
$CHCl_3$	--	--	--	--	--	--	--	--	--	--
Acetone	--	--	--	--	--	--	--	--	--	--
H_2SO_4	++	++	++	++	++	++	++	++	++	++

++ Soluble. -+ Low solubility. – Insoluble.

Table 3. Elemental Analysis of the PIs

Polymer	Compound	Calcd.			Found		
		%C	%H	%N	%C	%H	%N
3	$(C_{33}H_{18}N_4O_6)_n$	69.96	3.20	9.89	69.56	3.10	9.92
4	$(C_{35}H_{18}F_6N_4O_5)_n$	59.75	2.72	9.95	60.03	2.52	8.24
5	$(C_{32}H_{18}N_4O_6)_n$	66.31	3.27	10.10	66.85	3.14	9.73
6	$(C_{32}H_{18}N_4O_5)_n$	73.37	3.37	10.40	74.84	3.35	10.34
7	$(C_{26}H_{14}N_4O_5)_n$	67.53	3.05	12.12	67.51	2.97	12.00

Table 4. Thermal and Mechanical Properties of the Polyimide Films

Polymer	T_g (°C)[a]	10% Mass loss (°C)	T_D (°C)[b]	T_p[c]	Char yield (%)[d]	Storage modulus (GPa)				
						50 °C	100 °C	200 °C	250 °C	300 °C
3	294.9 (340.5)	482.91	441.45	481.9	76.93	2.66	2.61	2.55	2.50	2.46
4	N.O. (339.4)[e]	494.09	435.77	532.3	65.65	4.85	4.74	4.41	4.16	3.82
5	286.0	468.28	410.90	491.5	72.71	N. A.[f]	N. A.	N. A.	N. A.	N. A.
6	N.O. (362.7)	484.49	440.13	471.2	77.63	4.34	4.26	3.98	3.82	3.58
7	N.O.	485.43	437.65	493.4	75.44	N. A.	N. A.	N. A.	N. A.	N. A.

[a] Obtained based on DTA measurements. The values in the parenthesis were obtained based on DMTA measurements. [b] Onset decomposition temperature. [c] The 1st derivative peak temperature. [d] At 596 °C. [e] Not observed. Glass transition temperature tentatively overlapped with melting endotherm. [f] Not analyzed.

thermal stability of PI (4) can be attributed to the presence of CF_3 groups in the main chain. PIs containing trifluoromethyl groups are of special interest due to enhanced solubility and optical transparency together with a lower dielectric constant, which were attributed to low polarizability of the C-F bond and increasing free volume. The trifluoromethylated PIs also provided other merits such as good thermal and low moisture absorption [39]. PI (5), obtained from BAMO and ODPA, has the lowest T_g value (T_g = 286.0 °C, measured by DTA). Attachment of the phenylene rings by a flexible ether linkage in the ODPA units can be responsible for observing low T_g. It must be noted that, observing of T_g for a polymer, especially at lower temperatures, can be considered as an advantage, because, it shows that the polymer is tractable and can be processed. Nevertheless, this advantage is slightly sacrificed by a drop in the thermal stability parameters (10% mass loss, T_D and T_p, as seen in Table 4). PI (6), obtained from BAMO and 3,3',4,4'-biphenyltetracarboxylic acid dianhydride, showed the maximum glass transition temperature (T_g = 362.7 °C, measured by DMTA). Both DTA and DMTA methods did not show T_g value for PI (7), obtained from BAMO and pyromellitic dianhydride. The high T_g of PI (6) and absence of T_g in PI (7) can be

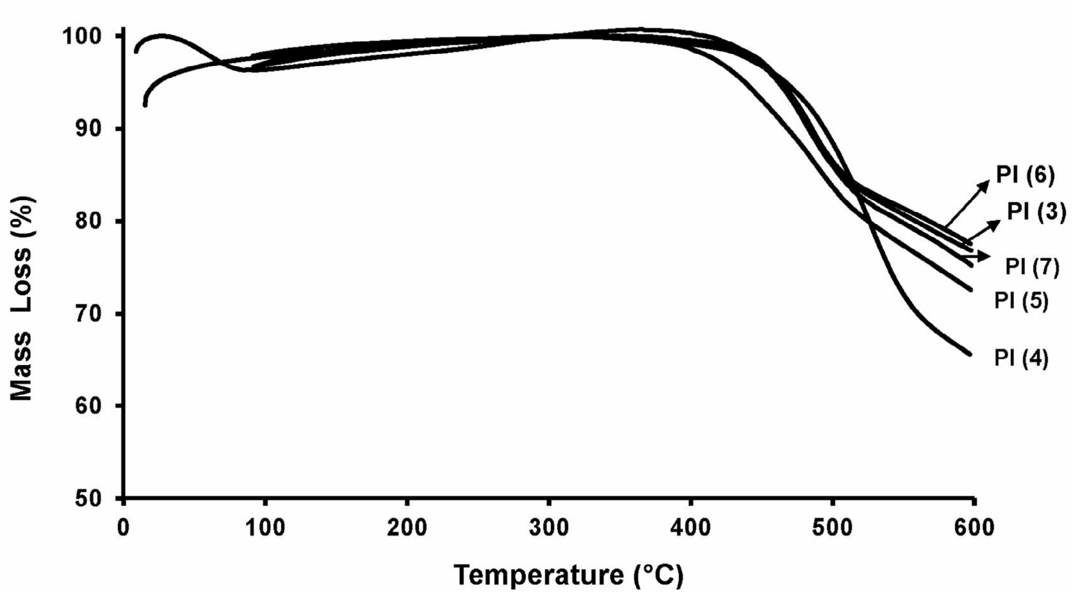


Fig. 6. TGA curves (nitrogen atmosphere, scan rate 10 °C min^{-1}) of the polyimides.

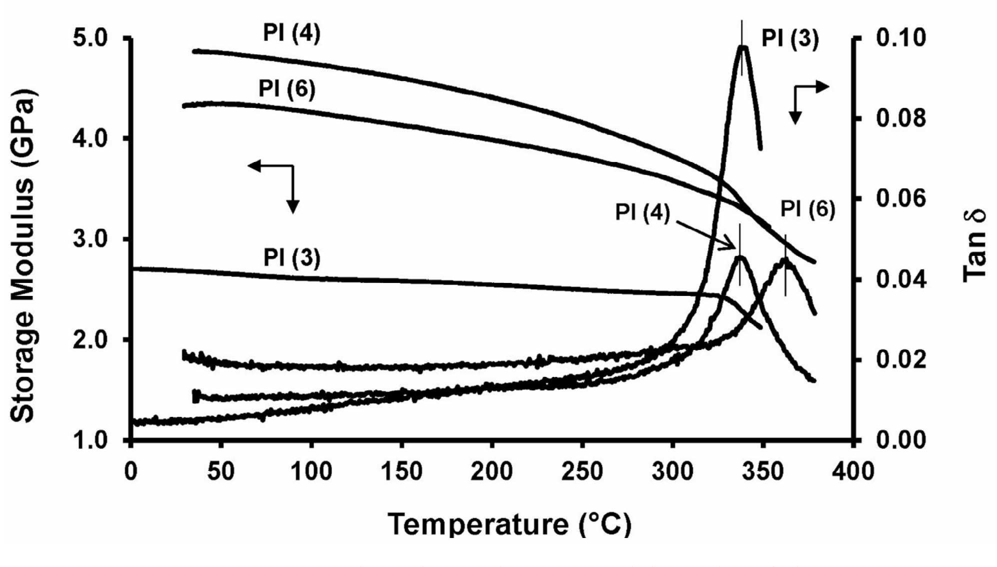
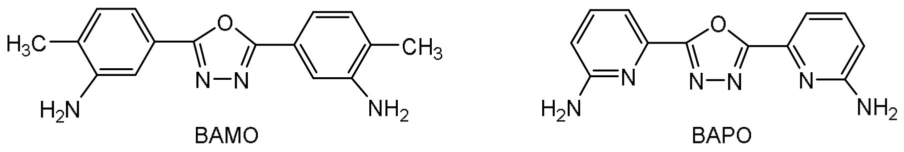


Fig. 7. Temperature dependence of storage modulus and tan δ the PIs.



Scheme 4. Comparison of the structure of BAMO and BAPO [32]

attributed the their rod-like structure and absence of any flexible linkage between the aromatic rings. TGA curves of the prepared PIs are shown in Fig. 6.

Based on our previous reports, BAPO [32] may be the nearest structurally aromatic diamine that can be compared with BAMO. The structure of both monomer is almost the same, Scheme 4.

The PIs obtained in this study have enhanced solubility along with slightly low thermal stability than those obtained from BAPO. The enhanced solubility of the PIs obtained from BAMO can be attributed to the presence of methyl groups. At the same time, methyl groups cause to lowering the thermal stability of the PIs obtained from BAMO. The presence of pyridine rings is responsible for the improved thermal stability parameters of the PIs obtained from BAPO.

Variations of storage modulus and tan δ *versus* temperature for PI (3), PI (4) and PI (6) have been shown in Fig. 7 and the results are summarized in Table 4. PI (4), obtained from BAMO and 6FDA, showed the highest storage modulus within room temperature to 300 °C. Therefore, based on the obtained results from TGA-DTA and DMTA measurements, it can be concluded that among the prepared polymers PIs (4) and (5) can be distinguished for their improved thermal/mechanical properties and low T_g, respectively.

CONCLUSIONS

New thermally stable PIs containing 1,3,4-oxadiazole ring in the main chain based on BAMO were synthesized. Solubility tests showed the polymers are soluble in polar organic solvents such as DMSO, NMP and DMAc at elevated temperatures. The thermal and mechanical behaviors of the polymers were studied by TGA/DTA and DMTA methods. PI (4) exhibited the best thermal and mechanical properties, while PI (5) is noted for its low T_g value.

ACKNOWLEDGEMENTS

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