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Synthesis and characterization of new optically active and photolabile poly (amide-imide)s from *N,N'*-(3,3',4,4'-benzophenonetetracarboxylic)-3,3',4,4'-diimido-di-L-methionine and different diamines

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Summery

N,N'-(*3,3',4,4'*-Benzophenonetetracarboxylic)-3,3',4,4'-diimido-di-L-methionine (**3**) was prepared from the reaction of *3,3',4,4'*-benzophenonetetracarboxylic-*3,3',4,4'*-dianhydride (**1**) with L-methionine (**2**) in a solution of (glacial acetic acid/pyridine) at refluxing temperature. The phosphorylation polycondensation of the diimide-diacid monomer (**3**) with 1,3-phenylenediamine (**4a**), 1,4-phenylenediamine (**4b**), 2,6-diaminopyridine (**4c**), 3,5-diaminopyridine (**4d**), 4,4′-diaminobiphenyl (**4e**) and 4,4′-diaminodiphenylsulfone (**4f**) was carried out in *N*-methyl-2-pyrolidone (NMP). The resulting poly (amide-imide)s showed admirable moderate inherent viscosities (0.23-0.48 dl g^{-1}), good thermal stability and improved optical activity. All of the above compounds were fully characterized by IR spectroscopy, elemental analysis and specific rotation. Some structural characterization and physical properties of these new poly (amide-imide)s are presented.

Introduction

Due to the increasing demands for high-performance polymers as a replacement for ceramics or metals in the microelectronic, aerospace and automotive industries, thermally stable polymers have received much interest over the past decade. Polyimides and their copolymers are certainly one of the most useful classes of high-performance polymers, which have found many applications in industries [1-2]. Aromatic polyimides are an important class of heterocyclic polymers with remarkable heat resistance and superior mechanical and electrical properties, and also durability [3-5]. Poor thermoplastic fluidity and solubility are the major problems in wide application of polyimides. But polyamide-imide (PAI) is the most successful material, which combines the advantages of high-temperature stability and processability [6]. A notable example is Torlon (Amoco Co.), a commercial PAI obtained from trimellitic anhydride chloride and 4,4’-diaminodiphenylmethane [7]. The synthesis and application of optically active polymers are the newly considerable topics which have been paid more attention recently [8]. Most of the natural polymers are optically active and have special chemical activities, such as catalytic properties that exist in genes, proteins and enzymes. Some other applications are construction of chiral media for asymmetric synthesis, chiral stationary phases for resolution of enantiomers in chromatographic techniques [9-13], chiral liquid crystals in ferroelectrics and nonlinear optical devices [14-17]. These synthetic polymers based on optically pure aminoacids can induce crystallinity with their ability to form higher ordered structures that exhibit enhanced solubility characteristics [18]. These properties have caused them to be good candidate for drug delivery systems, biomimetic systems, biodegradable macromolecules, biomaterials, and also as chiral purification media [19]. So, more considerations to improve different synthetic procedures of optically active polymers exist. Recently, we have synthesized optically active polymers by different methods [20-23]. The triphenyl phosphite activated polycondensation (phosphorylation reaction) technique for the synthesis of polyamides has been reported by Yamazaki and coworkers [24]. In this research, we report the synthesis and characterization of six new photolabile, optically active and thermally stable PAIs from aromatic diamines and *N,N’*-(3,3’,4,4’-benzophenonetetracarboxylic)- 3,3’,4,4’-diimido-di-L-methionine, a monomer performed imide groups, by phosphorylation polycondensation method. We have chosen L-methionine, since its corresponding monomer (**3**) has a high optical rotation ($[\alpha]_D^{25} = -115.5^{\circ}$), and also we had expected that the sulfide group in the pendant moiety would improve the solubility of the corresponding polymers. Here we have also investigated the effect of different reaction conditions such as reaction temperature, reaction time and activating agent ratio on optical activity and viscosity of polymers.

Experimental

Materials

N-Methyl-2-pyrolidone (NMP, Merck) and pyridine (Py, Merck) were purified by distillation under reduced pressure over calcium hydride and stored over $4\overset{\circ}{\mathrm{A}}$ molecular sieve. Triphenyl phosphate (TPP, Merck) was purified by fractional distillation under vacuum. LiCl (Merck) was dried for 10h at 180ºC under vacuum. 3,3’,4,4’-Benzophenonetetracarboxylic-3,3’,4,4’-dianhydride (**1**) (Merck) was recrystalized from acetic anhydride. Reagent grade aromatic diamine (Aldrich) including 1,3-phenylenediamine (**4a**), 1,4-phenylenediamine (**4b**), 2,6-diamino-pyridine (**4c**) and 3,5-diaminopyridine (**4d**) were purified by sublimation. 4,4′-Diaminobiphenyl (**4e**) and 4,4′-diaminodiphenylsulfone (**4f**) were recrystalized from water/ethanol (1:1).

Instruments

IR spectra were recorded in potassium bromide pellets on a JASCO FT-IR_680plus over the range 400-4000 cm^{-1}. Band intensities are assigned as weak (w), medium (m), strong (s) and band shapes as shoulder (sh), sharp (s) and broad (br). Elemental analysis was performed by a CHN-O-Rapid Heraeus elemental analyzer (Wellesley, MA). Inherent viscosities (η_{inh} at a concentration of 0.5 dl g^{-1}) were measured by a standard procedure using a Cannon Fenske routine viscometer at 25ºC using DMF as solvent. Specific rotations were measured by a JASCO P-1030 Polarimeter. Thermogravimetric analyses (TGA) were recorded on a Mettler TGA-50 under air atmosphere at a heating rate of 10 °C min^{-1}.

*Synthesis of N,N'-(3,3',4,4'-benzophenonetetracarboxylic)-3,3',4,4'-diimido-di-L-methionine (**3**)*

Into a 25 ml round-bottomed flask, 2.523 g (7.83x10^{-3} mol) of 3,3',4,4'-benzophenonetetracarboxylic-3,3',4,4'- dianhydride (**1**), 2.345 g (1.57x10^{-2} mol) of L-methionine (**2**), 10 ml of a mixture of acetic acid and pyridine (3:2) and a stirring bar were placed. The mixture was stirred at r.t. for 2 h and then refluxed for 15 h. The solvents were removed under reduced pressure and then 5 ml of cold concentrated HCl was added. A pale yellow precipitate was formed, filtered off, and washed with hot water. The solid was recrystallized from DMF-H_2O mixture and dried to leave 4.545 g (99.4%) of a white solid (**3**). M.p.>300°C. $[\alpha]_D^{25}$ = -115.5°. IR (KBr): 3477-2650 (m, br.), 2920 (m, s), 1777 (s, s), 1718 (s, br), 1383 (s, s), 1249 (w, s), 1105 (m, s), 727 (m, s) cm^{-1}. $C_{27}H_{24}N_2O_9S_2$: Calc. C 55.4, H 4.2, N 4.7. Found C 56.2, H 5.0, N 3.9.

*Synthesis of model Compound (**5**)*

The model compound was prepared by phosphorylation polycondensation method. Into a 10 ml round-bottomed flask fitted with water cooled condenser and a magnetic stirrer, 0.292 g (5.0x10^{-4} mol) of diimide-diacid (**3**), 0.1 g of calcium chloride, 0.15 g of LiCl, 0.436 g (1.34x10^{-3} mol) of triphenyl phosphite, 0.106 g of pyridine, 0.093 g (1.0x10^{-3} mol) of aniline and 1.0 ml of NMP was placed. The stirrer was operated and the mixture was heated under nitrogen atmosphere in an oil bath at 60 °C for 1 h then at 90 °C for 0.5h and finally 4h at 100°C. The reaction mixture was poured into 50 ml of MeOH. The solid powder was recrystalized from EtOH to leave 0.348 g (95%) of pale yellow solid, mp>300 °C. IR (KBr): 3383 (m, br), 2916 (w, s), 1762 (w, s), 1717 (s, br), 1510 (s, s), 1382 (s, s), 1350 (s, s), 728 (w, s) cm^{-1}.$C_{39}H_{34}N_4O_7S_2$: Calc. C 63.7, H 4.7, N 7.6. Found C 61.0, H 4.9%, N 7.2%. After moisture correction (2.1% moisture intake): C 62.2, H 4.8, N 7.3.

*Polymerization: synthesis of polymer (**6a**)*

The PAIs were prepared by the following procedure (taking polymer **6a** as an example). Into a 10 ml round-bottomed flask fitted with water cooled condenser and a magnetic stirrer, 0.292 g (5.0x10^{-4} mol) of diimide-diacid **3**, 0.1 g of calcium chloride, 0.15 g of LiCl, 0.436 g (1.34x10^{-3} mol) of triphenyl phosphite, 0.106 g of pyridine, 0.054 g (5.0x10^{-4} mol) of diamine (**4a**) and 1.0 ml of NMP was placed. The stirrer was operated and the mixture was heated under nitrogen atmosphere in an oil bath for 1 h at 60 °C then 0.5 h at 90 °C and finally 10 h at 100 °C. The viscous reaction mixture was poured into 50 ml of MeOH. The obtained polymer was washed thoroughly with MeOH, collected by filtration, and dried at 80 °C for 15 h under vacuum to leave 0.311 g (95%) of dark yellow polymer (**6a**). IR (KBr): 3425 (m, br), 2915 (w, s), 1771 (m, s), 1720 (s, s), 1513 (m, s), 1383 (m, s), 1358 (m, s), 832 (w, s), 722 (m, s) cm^{-1}. $C_{33}H_{28}N_4O_7S_2$: Calc. C 60.3, H 4.3, N 8.5. Found C 53.6, H 5.1%, N 7.4%. After moisture correction (10.7% moisture intake): C 59.3, H 4.5, N 8.2.

The other polymers (**6b-6f**) were prepared in a similar way.

*Polymer (**6b**)*
Yellow; IR (KBr): 3427 (m, br), 2917 (w, s), 1775 (m, s), 1722 (s, s), 1514 (m, s), 1383 (m, s), 1358 (m, s), 835 (w, s), 727 (m, s) cm^{-1}. $C_{33}H_{28}N_4O_7S_2$: Calc. C 60.3, H 4.3, N 8.5. Found C 53.6, H 5.1%, N 7.4%. After moisture correction (10.5% moisture intake): C 59.2, H 4.5, N 8.2.

*Polymer (**6c**)*
Dark yellow; IR (KBr): 3433 (m, br), 2910 (w, s), 1775 (w, s), 1717 (s, s), 1591 (m, br), 1384 (s, s.), 1108(w, s), 830 (w, s), 721 (m, s) cm^{-1}. $C_{32}H_{27}N_5O_7S_2$: Calc. C 58.4, H 4.2, N 10.5. Found C 53.9, H 4.9%, N 8.9%. After moisture correction (8.1% moisture intake): C 58.3, H 4.5, N 9.6.

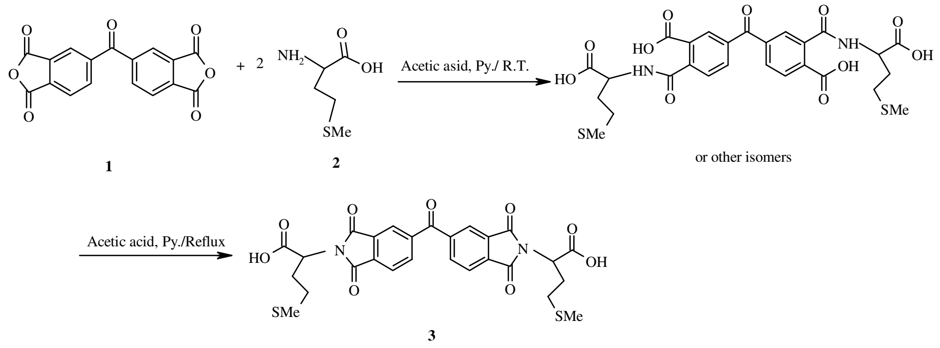
*Polymer (**6d**)*
Dark yellow; IR (KBr): 3431 (m, br), 2912 (w, s), 1776 (w, s), 1718 (s, s), 1590 (m, br), 1380 (s, s.), 1106(w, s), 831 (w, s), 723 (m, s) cm^{-1}. $C_{32}H_{27}N_5O_7S_2$: Calc. C 58.4, H 4.2, N 10.5. Found C 53.9, H 4.9%, N 8.9%. After moisture correction (8.3% moisture intake): C 58.4, H 4.5, N 9.6.

*Polymer (**6e**)*
Pale yellow; IR (KBr): 3433 (m, br), 2919 (w, s), 1775 (m, s), 1718 (s, br), 1590 (m, s), 1500 (w, s),1383 (m, s.), 1356 (m, s), 1246 (w, s), 1151 (w, s), 1100 (w, s), 822 (w, s),725 (w, s) cm^{-1}. $C_{39}H_{32}N_4O_7S_2$: Calc. C 63.9, H 4.1, N 7.6. Found C 61.2, H 5.2%, N 6.2%. After moisture correction (5.1% moisture intake): C 64.2, H 4.9, N 6.5.

*Polymer (**6f**)*
Pale yellow; IR (KBr): 3436 (m, br), 2917 (w, s), 1775 (m, s), 1723 (s, s), 1590 (m, s), 1526 (w, s),1383 (m, s.), 1356 (m, s) , 1317 (m, s), 1251 (w, s), 1151 (m, s), 1105 (m, s),835 (w, s),726 (m, s) cm^{-1}. $C_{39}H_{32}N_4O_9S_2$: Calc. C 58.7, H 4.0, N 7.0. Found C 52.3, H 4.4%, N 5.9%. After moisture correction (9.1% moisture intake): C 57.1, H 4.0, N 6.4.

Results and discussion

N,N'-(3,3',4,4'-Benzophenonetetracarboxylic)-3,3',4,4'-diimido-di-L-methionine (**3**) was prepared from condensation of dianhydride (**1**) with L-methionine (**2**) in 1:2 molar ratio in refluxing acetic acid:pyridine (3:2) (Scheme 1).The IR spectrum of the monomer (**3**) is represented in Figure. 1. The chemical structure and purity of the photolabile, and optically active monomer (**3**) was proved by using elemental analysis.



Scheme 1. Preparation of monomer **3**

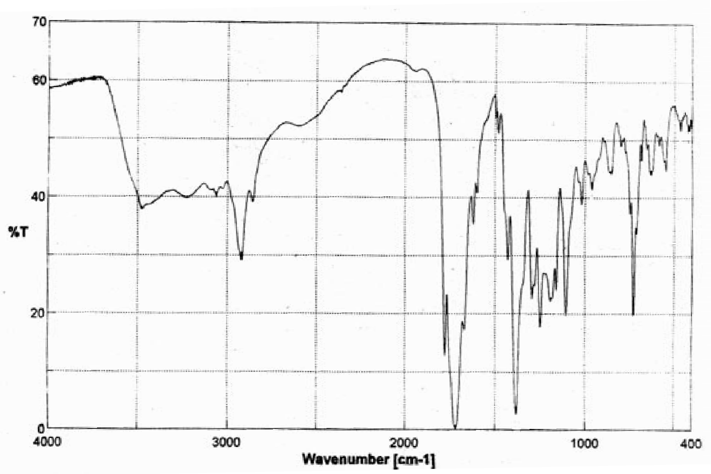
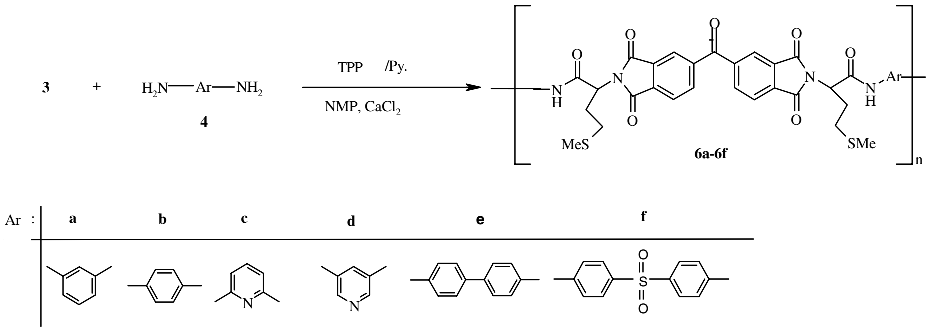


Fig. 1: IR spectrum of monomer **3**

The phosphorylation polycondensation method was used to prepare the polyamides (**6a-6f**) (Scheme 2).The optimum TPP/monomer ratio found to be 2-3 and more ratio cause lower viscosity. The synthesis conditions and some physical properties of these novel photolabile and optically active PAIs are given in Table 1. The entire polycondensation readily proceeded in a homogeneous solution. When the viscous PAIs solutions were trickled into the stirred methanol tough and stringy precipitates formed. All polymers were obtained in quantitative yields with moderate inherent viscosities (0.23-0.48 dl g^{-1}) which are admirable for these types of polymers. In compare to our last project on optically active polymers [23], these polymers showed improved optical activity (-12.5 to -16.6) and also because of the benzophenone moiety presence, these polymers can potentially be photolabile [25]. These photolabile polymers may be potentially able to be used as affinity column for protein purification [25]. The outstanding characteristics of these polymers include thermal stability, improved optical activity and being photolabile.



Scheme 2. Phosphorylation polycondensation

Table 1 Synthesis conditions and some physical properties of PAIs (**6a-6f**)

Diamine	Polymer				
	PAIs	Yield (%)	η_{inh} (dl g^{-1})[a]	$[\alpha]_D^{25}$ [b]	Color[c]
4a	**6a**	95	0.36	-13.3	DY
4b	**6b**	94	0.30	-12.5	Y
4c	**6c**	88	0.23	-13.2	DY
4d	**6d**	94	0.33	-16.1	DY
4e	**6e**	91	0.48	-14.1	PY
4f	**6f**	90	0.38	-16.6	PY

[a] Measured at a concentration of 0.5 g (dl^{-1}) in DMF at 25 °C

[b] Measured at a concentration of 0.050 g in 10 ml DMF at 25 °C

[c] PY=pale yellow, Y=yellow, DY=dark yellow

To optimize the polymerization conditions, we did six experiments to prepare polymer **6a** at different temperatures and time; the results are given in Table 2. PAIs formation was confirmed by the IR spectroscopy and CHN analysis. As an example, the IR of PAI (**6a**) showed the characteristic absorptions of amide and imide groups around 3434, 1766, 1716 and 1520 cm^{-1}, peculiar to N-H stretching and carbonyls stretching of imide and amide groups respectively. All of these PAIs exhibited strong absorption at 1388 and 720 cm^{-1}, due to the presence of the heterocyclic imide group in these polymers. IR spectrum of PAI (**6a**) is represented in Fig. 2. Polymer **6f** showed characteristic absorptions at 1317 and 1151 cm^{-1} due to the sulfone moiety (SO_2 stretching) (Fig. 3). The values of elemental analysis of the polymers are listed in Table 3.

Table 2 Optimization of phosphorylation on polymer **6a**

Time program	Temperature program	η_{inh} (dl g^{-1})	$[\alpha]_D^{25}$ [b]
1-0.5-5	60-90-130	0.25	-13.0
1-0.5-7	60-90-130	0.30	-12.8
1-0.5-10	60-90-130	0.40	-11.3
1-0.5-10	60-90-120	0.41	-12.1
1-0.5-10	60-90-110	0.39	-12.7
1-0.5-10	60-90-100	0.36	-13.3

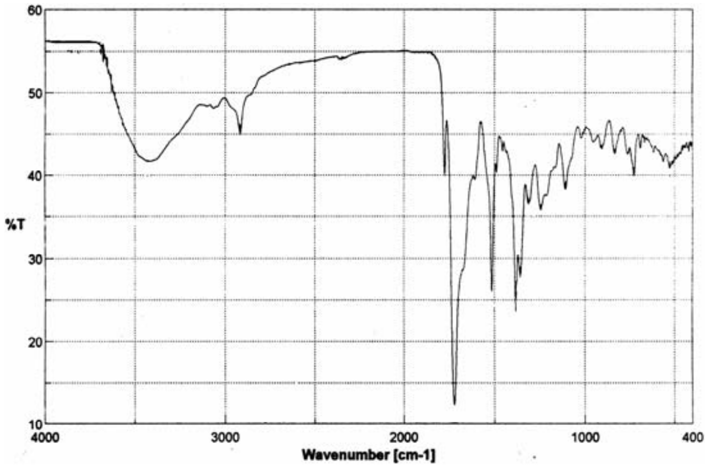


Fig. 2: IR spectrum of polymer **6a**

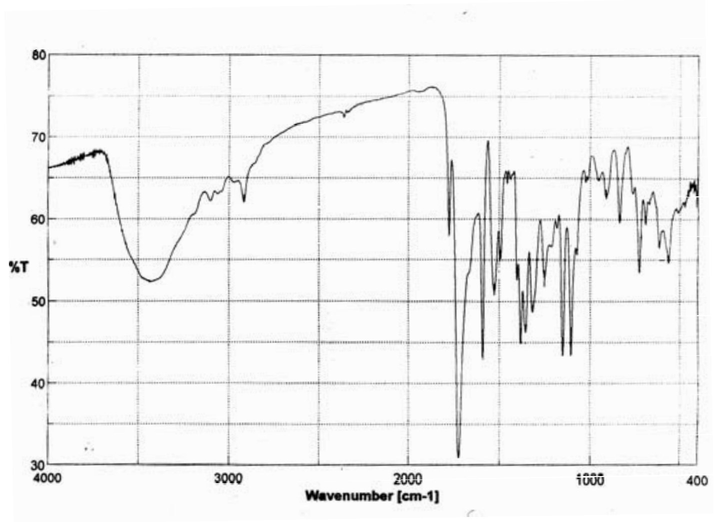


Fig. 3: IR spectrum of polymer **6f**

Since the monomer (**3**) does not dissolve in $CDCl_3$ and contains the aliphatic parts of the L-methionine which overlap with its solvent peaks (DMSO_d_6) in ^{1}HNMR, the spectrum is very complex and its interpretation is difficult, therefore we did not bring its ^{1}HNMR and also the polymers are highly hydrophilic and contain aliphatic and aromatic parts that overlap with the solvent peaks (DMSO_d_6) in ^{1}HNMR, the interpretation is very difficult and so we did not bring their ^{1}HNMR. The color of PAIs derived from monomer **3** may range from pale yellow to dark yellow. The solubility of PAIs was tested qualitatively in various solvents and the results are given in Table 4. All of the PAIs are soluble in solvents such as DMF, DMAc, DMSO, NMP and H_2SO_4 at room temperature, and are insoluble in solvents, such as choloroform, methylene chloride, methanol, ethanol and water. The thermal properties of PAIs (**6a-6f**) were evaluated by means of TGA/DTG under air atmosphere at a heating rate of

10 °C min^{-1}. All of these polymers showed similar decomposition behavior. Table 5 summarizes the thermal properties of PAIs (**6a-6f**). These PAIs exhibited good resistance to thermal decomposition. The temperature of 10% weight loss for the polymers (**6a-6f**) ranged from 220 °C to 290 °C and the residual weight for these polymers at 600 °C ranged from 5.5% to 35.8% under air atmosphere. TGA/DTG thermograms of PAIs **6a** and **6f** under air atmosphere are presented in Figs. 4 and 5 respectively.

Table 3 Elemental Analysis of PAIs **(6a-6f)**

			Elemental Analysis (%)			Moisture Intake(%)[a]
Polymer	Formula		C	H	N	
6a	$C_{33}H_{28}N_4O_7S_2$	Calcd	60.3	4.3	8.5	10.7
		Found	53.6	5.1	7.4	
		Corr[b]	59.3	4.5	8.2	
6b	$C_{33}H_{28}N_4O_7S_2$	Calcd	60.3	4.3	8.5	10.5
		Found	53.6	5.1	7.4	
		Corr[b]	59.2	4.5	8.2	
6c	$C_{32}H_{27}N_5O_7S_2$	Calcd	58.4	4.2	10.5	8.1
		Found	53.9	4.9	8.9	
		Corr[b]	58.3	4.5	9.6	
6d	$C_{32}H_{27}N_5O_7S_2$	Calcd	58.4	4.2	10.5	8.3
		Found	53.9	4.9	8.9	
		Corr[b]	58.4	4.5	9.6	
6e	$C_{39}H_{32}N_4O_7S_2$	Calcd	63.9	4.1	7.6	5.1
		Found	61.2	5.2	6.2	
		Corr[b]	64.2	4.9	6.5	
6f	$C_{39}H_{32}N_4O_9S_3$	Calcd	58.7	4.0	7.0	9.1
		Found	52.3	4.4	5.9	
		Corr[b]	57.1	4.0	6.4	

[a] Moisture Intake (%)=$(W-W_o)/W_o$x100, W=weight of polymer sample after standing at room temperature and W_o= weight of polymer sample after dring in vaccum at 100°C for 10 h.
[b] Corrected value for C and N= found value x (100+moisture_intake)/100, and corrected value for H= found value x (100-moisture_intake)/100.

Table 4 Solubility of PAIs **(6a-6f)**[a]

Solvents	**6a**	**6b**	**6c**	**6d**	**6e**	**6f**
NMP	+	+	+	+	+	+
DMSO	+	+	+	+	+	+
DMAc	+	+	+	+	+	+
DMF	+	+	+	+	+	+
H_2SO_4	+	+	+	+	+	+
CH_2Cl_2	-	-	-	-	-	-
$CHCl_3$	-	-	-	-	-	-
EtOH	-	-	-	-	-	-
MeOH	-	-	-	-	-	-
H_2O	-	-	-	-	-	-

[a] Concentration: 5 mg ml^{-1}: Soluble at room temperature (+);Insoluble (-).

Table 5 Thermal behavior of PAIs **(6a-6f)**

Polymer	Decomposition temperature (^{o}C) T_{10}[a]	Char yield (%)[b]
6a	250	20.0
6b	255	20.5
6c	250	32.9
6d	245	32.1
6e	220	35.8
6f	290	5.5

[a] Temperature at which 10% weight loss was recorded by TGA at a heating rate of 10^{o}C/min under air atmosphere.

[b] Percentage weight of material left after TGA analysis at maximum temperature 600^{o}C under air atmosphere.

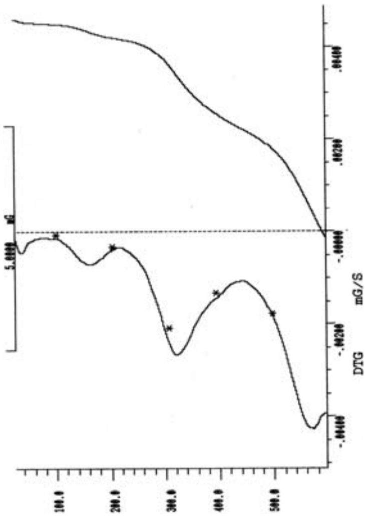


Fig. 4: TGA/DTG thermograms of **PAI** (**6a**) under air atmosphere at a heating rate of 10 ^{o}C min^{-1}

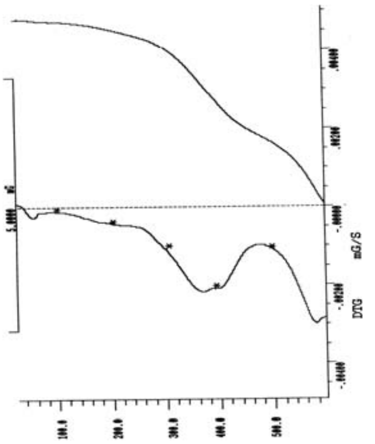


Fig. 5: TGA/DTG thermograms of **PAI** (**6f**) under air atmosphere at a heating rate of 10 ^{o}C min^{-1}

Conclusions

Six new photolabile, thermally stable and optically active PAIs with admirable inherent viscosities (0.23–0.48 dl g^{-1}) and improved optical activity (-12.5 to -16.6) in compare to our paper [23]. These aromatic PAIs show optical rotation, are readily soluble in various organic solvents, have a moderate thermal stability, and also because of the benzophenone moiety presence can potentially be photolabile [25].

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