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Preparation and Characterization of New Optically Active Poly(amide imide)s Derived from *N,N'*-(4,4'-Sulphonediphthaloyl)-bis-(s)-(+)-valine Diacid Chloride and Aromatic Diamines under Microwave Irradiation

Shadpour Mallakpour*[1], Elaheh Kowsari

Organic Polymer Chemistry Research Laboratory, College of Chemistry, Isfahan University of Technology, Isfahan, 84156-8311, I.R. Iran. 1-Previous Name: Shadpour E. Mallakpour
E- mail: MALLAK@CC.IUT.AC.IR

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Summary

The present paper is an extension of microwave method describing the synthesis of the new optically active poly(amide-imide)s. The main focus of this work is the design of new effective microwave method for preparing optically active poly(amide-imide)s. Imide-acid (**3**) was synthesized by the reaction of 3,3',4,4'-diphenylsulfonetetracarboxylic dianhydride (**1**) with (s)-(+)-valine (**2**) in acetic acid. The compound **3** was coverted to diacid chloride **4** by reaction with excess amount of thionyl chloride. Polycondensetion reaction of diacid chloride **4** with several aromatic diamines such as 4,4'-sulfonyldianiline (**5a**), 4,4'-diaminodiphenyl methane (**5b**), 4,4'-diaminodiphenylether (**5c**), *p*-phenylenediamine (**5d**), *m*-phenylenediamine (**5e**), 2,4-diaminotoluene (**5f**) and 4,4'-diaminobiphenyl (**5g**) was carried out in the presence of small amount of *o*-cresol under microwave irradiation as well as conventional heating method. We obtained a series of optically active poly(amide-imide)s with high yield and inherent viscosity ranging from 0.22-0.35 dL/g. These new polymers were characterized by FT IR, ^{1}H NMR, elemental analyses and specific rotation techniques.

Introduction

Application of microwave irradiation in polymerization reaction is a new and interested field. As a peculiar source of energy, microwaves have been widely used to enhance chemical reactions [1-6]. Experienced synthetic chemists, particularly in the pharmaceutical industry, have come to recognize the ease of scalability associated with microwave methods as a principal benefit just as important as the speed of reactions. With traditional methods, a great deal of time-consuming and experimental techniques are required to convert milligram-scale reactions to larger volumes. However, in the microwave, the same conditions that worked at the smallest scale can be applied at a much larger scale, to generate a proportional yield of equal purity. One important advantage, which led to the initial adoption of microwave-enhanced polymerization, is simply the speed with which synthetic reactions can be carried out. Reactions that would take days with conventional methods can be completed in few minutes in the microwave oven, eliminating that bottleneck. Recently we have used microwave irradiation for the synthesis of organic compounds as well as polymeric matireals [7-11].

Poly(amide-imide)s (PAI)s are an attractive polymer family which have received increased attention due to its natural biodegradability. These polymers are a type of chemicals materials, which could become one of the new sources of a family of environmentally friendly.

Polymers with optically active properties have been used for the separation of racemic mixture in pharmaceutical applications [12-16]. Aromatic polyimides have excellent physical properties and thermal stability, but their applications are limited because of their poor solubility. To solve these problems, amide groups were introduced into polyimides to form PAIs [17]. Sulfone groups were also introduced into polymer backbone which can effectively improve solubility. Recently, we have shown the synthesis of novel optically active polymers by different methods [18-25] such as modification of polybutadiene with an optically active substituted urazole group [18], Diels-Alder-ene reactions, [19-20] and reaction of an optically active monomer with several diamines via solution polymerization [21-25]. In polycondensation reactions, we use amino acids as chiral inducting agents. These materials are naturally occurring compounds therefore synthetic polymers based on amino acids are expected to be biodegradable and biocompatible. Biodegradable polymers have received considerable attention due to their potential applications in the fields related to human life such as environmental protection and the maintenance of physical health in the last two decades.

This paper reports the microwave assisted synthesis of optically new active PAIs containing 3,3',4,4'-diphenylsulfonetetracarboxylic dianhydride and (s)-(+)-valine moieties and compare this method with classical heating solution polymerization.

Experimental

Materials

4,4'-Diaminodiphenylmethane **5b** and benzidine **5g** was purified by recrystallization from water. 4,4'-Diaminodiphenylether **5c** was purified by a mixture of acetone and water. 1,4-Phenylenediamine **5d**, 1,3-phenylenediamine **5e**, 2,4-diaminotoluene **5f** and **5g** were purified by sublimation. 3,3'4,4'-Diphenylsulfonetetracarboxylic dianhydride was supplied from T.C.I Chemical Co. *N,N'*-Dimethylacetamide (DMAc) was dried over BaO, then distilled under vacuum. The other chemicals from Fluka Chemical CO., Aldrich Chemical CO. and Riedel-deHaen AG were used as obtained without further purification.

Instruments

We used a Samsung microwave oven (2450 MHz, 900W). All of the polymerization reactions carried out in a hood with strong ventilation.^{1}H NMR spectra were taken on a Bruker (Germany) 500-MHz and a Varian EM-390 (Varian Associates, Palo Alto, CA) spectrometer using $CDCl_3$ or DMSO-d_6 as the solvent and TMS as the internal standard. Multiplicities of proton resonance are designated as singlet (s), doublet (d), and multiplet (m).

Infrared spectra were tracked by the Shimadzu 435 IR spectrophotometer (Japan) and FT IR Unicom Mattson 1000 (England). Spectra of solids were carried out using KBr pellets. Vibrational transition frequencies are reported in wave number (cm^{-1}). Band intensities are assigned as weak (w), medium (m), shoulder (sh), strong (s) and broad (br). Inherent viscosities were measured by a standard procedure using a Cannon Fenske Routine Viscometer (Germany). Specific Rotations were measured by a Perkin Elmer-241 polarimeter (Germany). Thermal gravimetric analysis (TGA), and differential scanning calorimetry (DSC) data for polymers were taken on a STA 1640 (England) under nitrogen atmosphere at a rate of 10 ^{o}C/min. Elemental analysis were performed by Malek-Ashtar University of Technology, (Tehran, Iran).

Synthesis of monomer

N,N'-(4,4'-sulphonediphthaloyl)-bis-(s)-(+)-valine (Diacid 3)

Into a 50-mL, round-bottom flask (1.00 g, 2.80 × 10^{-3} mol) of 3,3',4,4'-diphenylsulfonetetracarboxylic dianhydride **1** (0.72 g , 6.14 × 10^{-3} mol) of (s)-(+)-valine **2**, 30 mL of acetic acid and a stirring bar were placed. The mixture was stirred at room temperature for 3 h and then was refluxed for 8 h. The solvent was removed under reduced pressure and 5 mL of cold concentrated HCl was added to the residue. A pale yellow precipitate was formed, filtered off, washed with cold concentrated HCl, to give 1.45 g (0.93%) of compound **3**.

mp 134-136 ^{o}C. $[\alpha]_{D}^{25}$: 102^{o} (0.050 g in 10 mL of dimethylformamide (DMF). FT-IR (KBr, cm^{-1}): 2969.23(w), 1730 (s),), 1384 (s), 1153 (m), 745.1 (m), 676.9 (w), 569.2 (w); ^{1}H NMR (90 MHz, $CDCl_3$, TMS, δ, ppm): 0.90 (d, 6H, $J = 6.0$ Hz), 1.20 (d, 6H, $J = 6.0$ Hz), 2.75 (m, 2H), 4.65 (d, 2H, $J = 9.0$ Hz), 7.80 (s, br, 2H), 8.20 (d, 2H, $J =$ 9.0 Hz), 8.50 (d, overlapped with singlet 2H), 8.55 (s, overlapped with doublet 2H). Elemental analysis $C_{26}H_{24}N_2O_{10}S$ (556): Cal. C, 56.11; H, 4.31; N, 5.03, Found: C, 55.93, H, 4.51, N, 4.93.

N,N'-(4,4'-sulphonediphthaloyl)-bis-(s)-(+)-valine(Diacid chloride 4)

Into a 25 mL, round-bottomed flask were placed (1.00 g, 1.79 × 10^{-3} mol) of compound **3** and 3 mL (an excess amount) of thionyl chloride. The mixture was heated with an oil bath up to 50 ^{o}C until the suspension mixture was converted to a clear solution and was heated for 2h. Unreacted thionyl chloride was removed under reduced pressure and was washed with fresh dry ether three times, to leave 1.04 g (97%) of pale yellow solid.

mp 92-95 ^{o}C, $[\alpha]_{D}^{25}$: −135^{o} (0.050 g in 10 mL DMF); FT IR (KBr, cm^{-1}): 3492.3 (w), 2969.2 (w), 1807.6 (s), 1730.7 (s), 1384.6 (s), 1153.8 (m), 1092.3 (m), 1007.6 (w), 915.3 (w), 753.8 (w), 675.9 (w), 569.2 (w);.^{1}H NMR (90 MHz, $CDCl_3$, TMS, δ, ppm): 0.90 (d, 6H, $J = 7.5$ Hz,), 1.20 (d, 6H, $J = 6.0$ Hz), 2.80 (m, 2H), 4.90 (d, 2H, $J = 7.5$ Hz), 8.30 (d, 2H, $J = 7.5$ Hz), 8.60 (d, overlapped with singlet, 2H), 8.70 (s, overlapped with doublet, 2H).

Polymerization

Different methods were applied to obtain polymers.

Method I: Polymerization under Microwave Irradiation

The PAIs were prepared by the following general procedure (using polymer **6aI** as an example). Into a porcelain dish were placed (0.10 g, 1.68 × 10^{-4} mol) of diacid chloride **4** and (0.0418 g, 1.68 × 10^{-4}mol) of diamine **5a**. After the reagents were completely ground, 0.10 mL of *o*-cresol was added, the mixture was ground for 3 min, then the reaction mixture was irradiated in the microwave oven for 6 min with 100% of the power. The resulting product was isolated by adding methanol and triturating, following by filtration and was dried at 80 ^{o}C for 10 h under vacuum to leave 0.124 g (96.0%) of solid **6aI**.
FT IR (KBr, cm^{-1}): 3616.7 (w), 3350.5 (w), 2966.7 (w), 1724.4 (s), 1591.3 (s), 1527.7 (s), 1379.1 (s), 1321.3 (m), 1149.6 (s), 1105.2 (m), 742.6 (w), 673.2 (W), 565.1 (w). ^{1}H NMR (500 MHz, DMSO-d_6, δ, ppm): 0.85 (d, distorted), 1.05 (d, distorted), 2.70 (m), 4.95 (d, distorted), 7.70 (d, J = 7.75 Hz), 7.85 (d, J = 8.45 Hz), 8.45 (d, J = 12 Hz), 8.5-8.6 (d, overlapped with singlet), 8.50-8.60 (s, overlapped with doublet), 10.30 (s)

Method II: Low temperature solution polycondensation with trimethylsilyl chloride (TMSCl)

Using polymer **6aII** as an example, the general procedure consisted of adding (0.100 g, 1.68 × 10^{-4} mol) of diacid chloride **4** to a cooled (-5.0 ^{o}C) and stirring solution of (0.0418 g, 1.68 × 10^{-4} mol) of diamine **5a** in 0.20 mL of NMP. After the reagents dissolved completely, 0.05 mL of TMSCl was added and reaction was allowed to proceed for 2h under a blanket of nitrogen. Then the temperature was raised to room temperature. The reaction mixture was stirred for 8h. The viscous solution was poured into 40 ml of methanol and the precipitated solid was filtered off and dried at 80 ^{o}C for 10 h under vacuum to leave 0.124 g (96 %) of solid polymer **6aII**.

Method III: High temperature solution polycondensation

Polymer **6aIII** is used as an example. Into a 5 mL round-bottom flask were placed (0.100 g, 1.68 × 10^{-4}mol) of diacid chloride **4,** (0.0418 g ,1.68 × 10^{-4}mol) of diamine **5a** and 0.10 mL of DMAc and then 0.05 mL of TMSCl was added. The mixture was refluxed for one min then the reaction mixture was cooled to room temperature. The viscous solution was poured into 40 mL of methanol and the precipitated solid was filtered off and dried at 80 ^{o}C for 10 h under vacuum to leave 0.121 g (94.0 %) of polymer **6aIII**.

Polymer **6bI.** FT IR (cm^{-1}): 3337.0 (w), 2968.5 (w), 1722.5 (s), 1670.4 (m), 1514.2 (m), 1379.2 (m), 1321.3 (m), 1147.7(m), 1078.2 (w), 748.0 (w), 673.2 (w), 565.1(w).

Polymer **6cI.** FT IR (cm^{-1}): 3443.1 (w), 3387.2 (w), 3323.5 (w), 2968.6 (w), 1724.4 (s) 1498.7 (s), 1381.1 (s), 1325.1 (w), 1222.9 (s), 1147.7 (m), 1078.2 (m), 827.5 (w), 673.2 (w), 503.4 (w) cm^{-1}. ^{1}H NMR (500 MHz, DMSO- d_6, δ, ppm): 0.85 (d, J = 5.15 Hz), 1.05 (d, J = 5.26 Hz), 2.80 (m), 4.60 (d, distorted), 6.90 (s), 7.50 (d, J = 7.70 Hz), 8.15 (d, J = 7.34 Hz), 8.65 (d, J = 9.75 Hz), 9.90 (s, distorted).

Polymer **6dI.** FT IR (cm^{-1}): 3348.6 (w), 2968.6 (w), 1724.4 (s), 1516.1 (s), 1379.2 (s), 1321.1 (m), 1147.72 (m), 1078.3 (w), 746.5 (w), 673.2 (w), 565.2 (w) cm^{-1}. ^{1}H NMR (500 MHz, DMSO-d_6, δ, ppm): 0.80 (d, J = 5.26 Hz), 1.05 (d, J = 4.13 Hz), 2.80 (m), 4.40-4.60 (d, distorted), 7.40 (s), 8.15 (d, J = 7.59 Hz), 8.50-8.70 (d, overlapped with singlet), 8.50-8.70 (s, overlapped with doublet), 9.90 (s).

Polymer **6eI.** FT IR (cm^{-1}): 3607.1 (w), 3348.6 (w), 2968.6 (w), 1780.4 (m), 1722.5 (s), 1608.7 (m), 1541.2 (w), 1489.1 (w), 1377.2 (m), 1147.7 (m), 1078 .0(w), 920.1 (w), 856.4 (w), 744.0 (w), 673.2 (w), 565.1 (w).

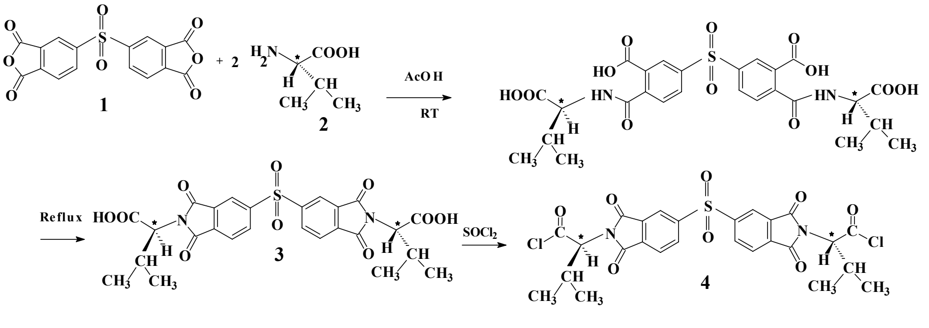
Polymer **6fI.** FT IR (cm^{-1}): 3350.5 (w), 2968.6 (w), 1724.4 (s), 1604.8 (w), 1531.5 (w), 1379.1 (s), 1323.2 (m), 1147.7 (m), 1078 (m), 922.0 (w), 746.5 (w), 673.2 (m), 565.1 (w).

Polymer **6gI.** FT IR (cm^{-1}): 3607.1 (w), 2968.6 (w), 1722.5 (s), 1504.6 (m), 1379.2 (m), 1323.3 (m), 1147.7 (w), 1078.2 (w), 746.5 (w), 673.2(w), 565.2 (w).

Results and discussion

Monomer Synthesis

A novel type of (s)-(+)-valine based monomer 4 was prepared, according to our earlier works, [16-19] by the three step process as shown in Scheme 1. Thus, asymmetric diacid compound **3** was synthesized by the condensation reaction of dianhydride **1** with two moles of (s)-(+)-valine **2**. In this reaction the intermediate amic acid was not isolated and ring closure for the formation of imide ring was performed under refluxing conditions.



Scheme 1. Synthesis of monomer 4.

The diacid **3** was converted to diacid chloride **4** by reaction with thionyl chloride. The chemical structure and purity of the compounds **3** and **4** were proved using elemental analysis, IR and ^{1}H NMR spectroscopic techniques. The IR spectrum of compound **3** showed a broad and strong peak at 3500-2500 cm^{-1}, which was assigned to the COOH groups.

The ^{1}H NMR spectrum (90 MHz) of compound **3** is shown in Figure 1. The doublet in 4.90 ppm is assigned to the protons of the chiral center. The ^{1}H NMR spectrum

(90 MHz) of diacid chloride **4** is similar to that of diacid derivative. A singlet to carboxylic acid proton was disappeared.

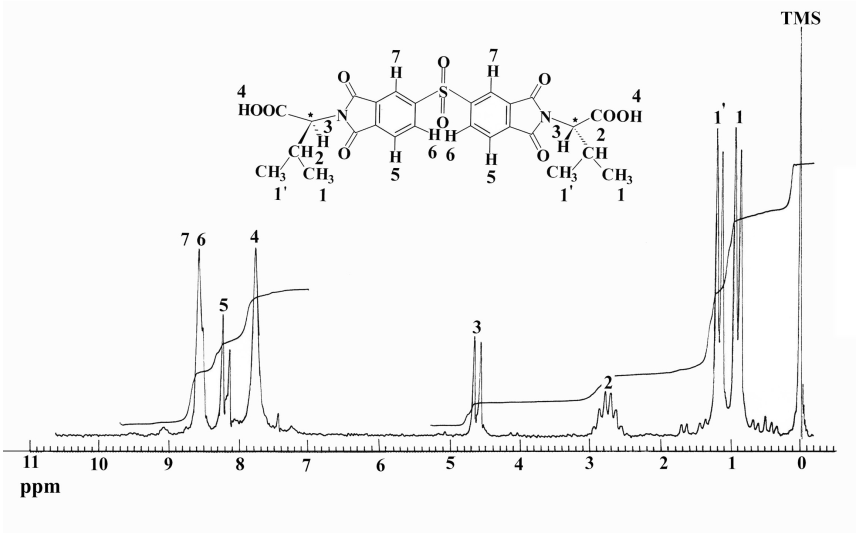
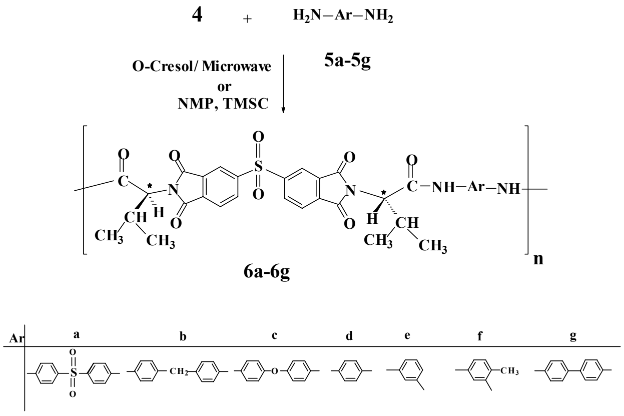


Figure 1. 1H-NMR (90 MHz) Spectrum of diacid (3) in $CDCl_3$, TMS at rt.

Polymer Synthesis

Microwave-assisted polycondensation as well as solution polycondensation reactions of an equimolar mixture of monomer **4** with seven different aromatic diamines (**5a-5g)** were carried out to produce PAIs **6a-6g** as shown in Scheme 2. The conventional solution polymerization reactions were performed under low temperature as well as high temperature conditions.



Scheme 2. Ploycondensation reactions of monomer **4** with aromatic diamines.

The microwave assisted polycondensation reactions were performed in the presence of small amount of *o*-cresol that acts as a primary microwave absorber, then the reaction mixture was irradiated for 6 min with 100% of radiation power. At higher radiation time dark products were obtained, on the other hand under low radiation time or power, reaction gave low yield. One important advantage, which led to the initial adoption of microwave-enhanced polymerization, is simply the speed with which synthetic reactions can be performed, We did not use catalyst in this method too. Microwave-enhanced synthesis actually generates large yields and purer products than conventional techniques. The reaction yields and some physical data for PAIs **6aI-6gI** are listed in Table 1.

Table 1. Some Physical Properties of PAIs **6aI-6gI** Prepared by Method I.

diamine	Polymer			
	Polymer code	Yield (%)	η_{inh}(dL/g)	$[\alpha]_D^{25}$
5a	**6aI**	96.0	0.22	-36.0
5b	**6bI**	86.4	0.35	-33.8
5c	**6cI**	95.3	0.27	-37.0
5d	**6dI**	70.0	0.26	-81.2
5e	**6eI**	79.0	0.28	-91.6
5f	**6fI**	65.2	0.29	-3.4
5g	**6gI**	78.6	0.31	-117.6

In order to compare microwave-assisted polycondensation method with conventional solution polycondensation methods, the polymerization of diacid chloride **4** with aromatic diamines **5a-5g** was performed under low temperature (Method II) and reflux condition (Method III). In method II polycondensation reactions were carried out in cold NMP solution (–5 °C) in the presence of TMSCl. In this method TMSCl activates the diamine monomers [26] and polymerization reactions occurred at lower temperature in a period of 5h. In method III polycondensation reactions proceeded rapidly at the reflux temperature of the used solvent for a period of 1 min. in this method we did not use any catalyst. We obtained comparable yield and viscosity of the PAIs from methods II and III compare to microwave assisted polymerizations. The reaction yields and some physical data of the solution polycondensations are listed in Tables 2 and 3.

Table 2. Some Physical Properties of PAIs **6aII-6gII** Prepared by Method II.

Diamine	Polymer			
	Polymer code	Yield (%)	η_{inh}(dL/g)	$[\alpha]_D^{25}$
5a	**6aII**	96.0	0.22	-43.0
5b	**6bII**	83.0	0.32	-21.2
5c	**6cII**	77.5	0.27	-21.6
5d	**6dII**	76.0	0.28	-54.8
5e	**6eII**	84.3	0.25	-58.0
5f	**6fII**	92.5	0.31	-75.2
5g	**6gII**	63.5	0.35	-18.6

Table 3. Some Physical Properties of PAIs **6aIII-6gIII** Prepared by Method III.

Diamine	Polymer			
	Polymer code	Yield (%)	η_{inh}(dL/g)	$[\alpha]_D^{25}$
5a	**6aIII**	94.2	0.22	-186.6
5b	**6bIII**	90.0	0.34	-34.4
5c	**6cIII**	85.7	0.25	-32.2
5d	**6dIII**	79.0	0.23	-28.8
5e	**6eIII**	85.0	0.26	-87.8
5f	**6fIII**	83.0	0.30	-14.0
5g	**6gIII**	87.3	0.31	-83.7

Structural Characterization of Polymers

The polymers obtained with the aromatic diamines are readily soluble in many organic solvents and show excellent thermal stability, The structures of these polymers were confirmed as PAIs by means of elemental analysis, IR and ^{1}H NMR spectroscopy. Elemental analysis data of the resulting polymers are listed in Table 4.

The FT IR data show the characteristic absorption peaks for the imide ring at 1724 and 1780 cm^{-1} due to the symmetrical and asymmetrical carbonyl stretching vibrations. The bands at 3337-3607cm^{-1} was assigned to the stretching vibration absorption of N-H in –CONH (hydrogen band) and 1498-1591 cm^{-1} (amide II band) for all of the polymers. All of them exhibited strong absorptions at 1379-1381 cm^{-1}, that show the presence of the imide heterocycle ring in these polymers. The polymer **6aI** showed characteristic absorptions at 1321.3 and 1149.6 cm^{-1} due to the sulfone moiety (SO_2 stretching). Figure 2 shows a typical IR spectrum.

Table 4. Elemental Analysis of PAIs **6aI–6gI**.

Polymer	Formula		Elemental Analysis (%)			Moisture Intake (%)
			C	H	N	
6aI	$(C_{38}H_{32}N_4O_{10}S_2)_n$	Calcd	59.37	4.20	7.29	0.50
	$(768)_n$	Found	58.93	4.66	7.01	
		Corr	59.22	4.63	7.04	
6bI	$(C_{39} H_{34}N_4O_8S)_n$	Calcd	65.18	4.73	7.80	0.70
	$(718)_n$	Found	64.50	5.06	7.61	
		Corr	64.95	5.02	7.67	
6cI	$(C_{38}H_{32}N_4O_9S)_n$	Calcd	63.33	4.44	7.77	1.60
	$(720)_n$	Found	62.00	4.72	7.54	
		Corr	63.00	4.64	7.66	
6dI	$(C_{32}H_{28}N_4O_8S)_n$	Calcd	61.14	4.45	8.91	0.10
	$(628)_n$	Found	61.00	4.80	8.90	
		Corr	61.05	4.79	8.91	
6eI	$(C_{32}H_{28}N_4O_8S)_n$	Calcd	61.14	4.45	8.91	0.15
	$(628)_n$	Found	60.92	4.84	8.79	
		Corr	61.01	4.83	8.80	
6fI	$(C_{33}H_{30}N_4O_8S)_n$	Calcd	61.68	4.67	8.72	0.20
	$(642)_n$	Found	61.33	4.94	8.79	
		Corr	61.44	4.92	8.81	
6gI	$(C_{38}H_{32}N_4O_8S)_n$	Calcd	64.77	4.54	7.95	2.90
	$(704)_n$	Found	62.02	4.54	7.28	
		Corr	63.82	4.40	7.49	

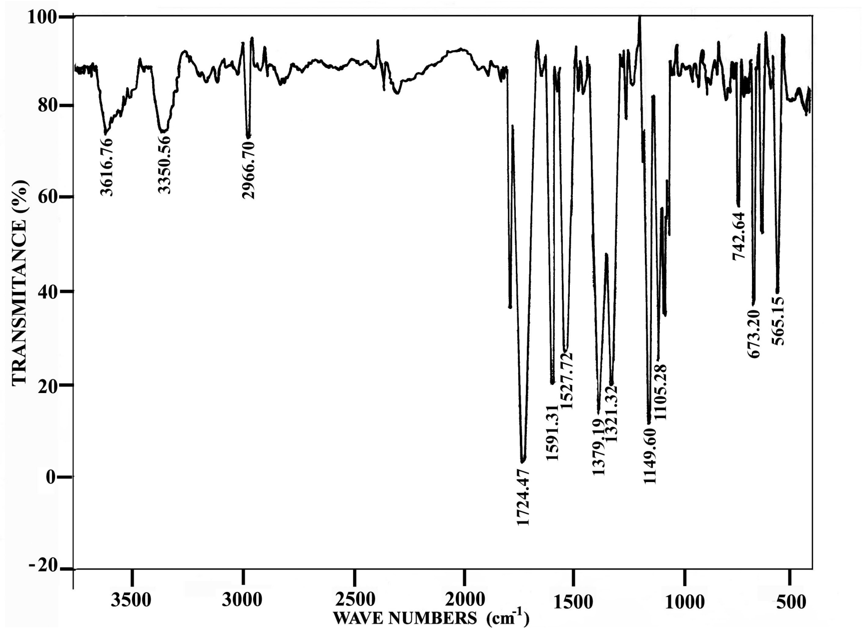


Figure 2. FT-IR (KBr) Spectrum of PAI-**6aI**.

The ^{1}H NMR spectra of PAI-**6cI** and PAI-**6dI** are shown in Figures 3 and 4 Appearance of the N-H proton of amide group around 10.00 ppm indicates that the

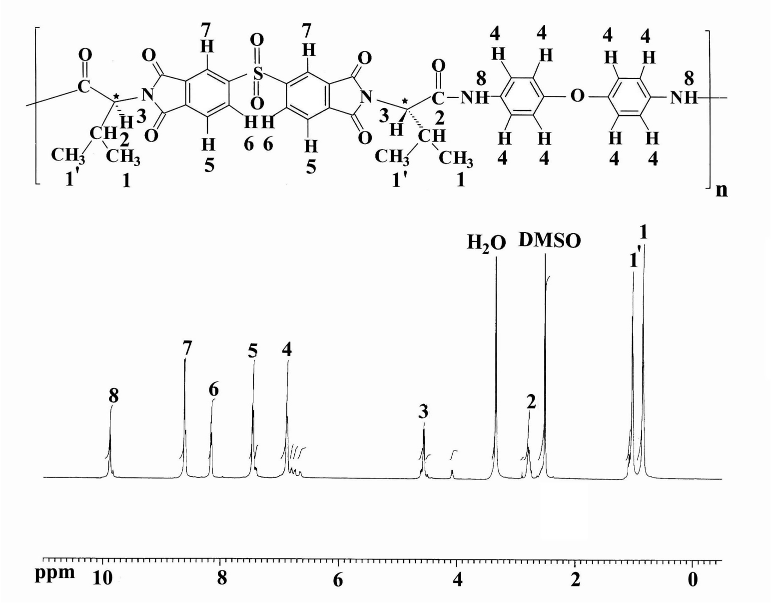


Figure 3. ^{1}H-NMR (500 MHz) Spectrum of PAI-**6cI** in DMSO-d_6 at rt.

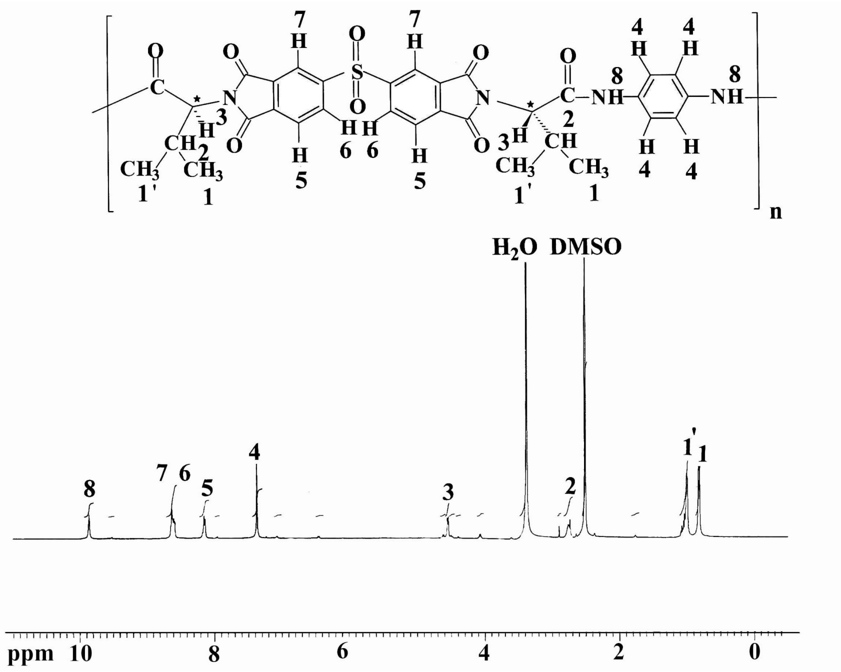


Figure 4. ^{1}H-NMR (500 MHz) Spectrum of PAI-**6dI** in DMSO-d_6 at rt.

amide group has been formed successfully by the reaction of diacid chloride **4** with diamines **5a-5g** .

PAIs are readily soluble in many organic solvents. solubilities are similar with different structures, all polymers are soluble in organic polar solvents such as DMAc, DMF, NMP and even in less polar solvents like *o*-cresol and THF. These polymers are insoluble in solvents such as chloroform, acetonitrile, cyclohexane and water.

Thermal Properties

The thermal stability of PAI-**6eI** was investigated by TGA measurement. TGA of PAI-**6eI** showed 5% and 10% weight loss (T_5, T_{10}) around 375.6 °C and 400.0°C under nitrogen, respectively and its char yields at 600°C was 53.75. It indicates that the resulting polymer has excellent heat resistance. A typical TGA curve of representative polymers is shown in Figure 5.

DSC measurement was performed on PAI-**6eI** between 50-1000°C. The glass transition temperatures (*Tg*) could be easily observed at 295.2°C. Figure 6 shows the STA curve for PAI-**6eI**.

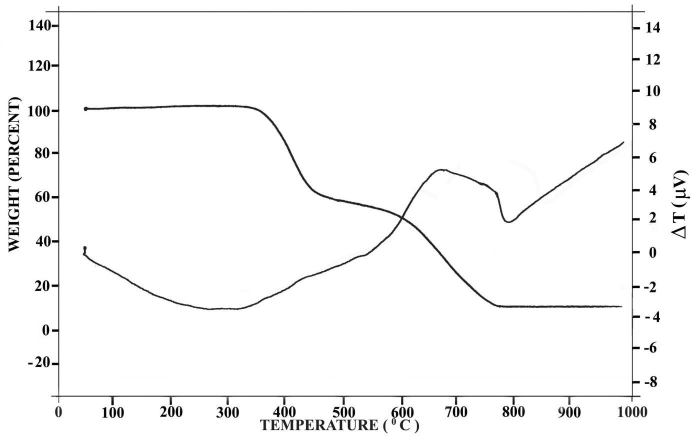


Figure 5. TGA/DSC of PAI-**6eI** with a heating rate of 10 °C/min in nitrogen atmosphere.

Conclusion

Several new optically active aliphatic-aromatic PAIs having *N,N'*-(4,4'-sulphonediphthaloyl)-bis-(s)-(+)-valine moieties were synthesized by using a microwave oven from polycondensation of optically active diacid chloride **4** with several diamines in the presence of a small amount of *o*-cresol. The use of such an organic medium was necessary to induce effective homogeneous heating of the monomers and thereby subsequent polycondensation leading to the formation of the polymers having inherent viscosity ranging from 0.22-0.35 dL/g. In order to compare this method with conventional solution polycondensation methods, PAIs were also synthesized by both low and high temperature solution polycondensation. We obtained comparable results from these methods with microwave assisted polymerization. These polymers are expected ideally to have potential to be used as packing materials in column chromatography.

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