

SYNTHESIS AND CHARACTERIZATION OF ORGANOSOLUBLE POLYAMIDES FROM QUINOXALINE BASED DIAMINE

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Abstract Aromatic/aliphatic polyamides were synthesized from a diamine monomer, 2,3-bis-*p*-aminophenylquinoxaline (IV), based on quinoxaline and various dicarboxylic acids of aliphatic, aromatic and heterocyclic. The diamine and polyamides were characterized by elemental analysis, FTIR and ¹H-NMR. The solubility of the polyamides was affected by the quinoxaline and heterocyclic groups in the polymer chain. They were all soluble in common organic solvents such as dimethylsulfoxide (DMSO), *N,N*-dimethylformamide (DMF) and *N*-methylpyrrolidone (NMP). The polyamides showed inherent viscosity in the range of 0.25–0.3 dL/g in DMSO at 25°C and good thermal stability with the char yields in the range of 65%–82% at 600°C in nitrogen.

Keywords: Quinoxaline; Polyamides; Solubility; Thermal stability.

INTRODUCTION

Aromatic polyamides are well known as high performance engineering thermoplastics and have been widely used in various industries because of their good thermal stability, chemical resistance and mechanical properties^[1]. However, their poor solubility in many organic solvents and also high softening or melting temperatures caused by their rigid structures and high stiffness of the polymer backbone or high crystallinity lead their process ability difficult. With the ever increasing demand for new processable engineering plastics, attempts have been oriented to the introduction of substituents having various chemical structures onto the benzene rings of monomers, the use of monomers containing several benzene rings separated by flexible bridges, or the use of heterocyclic monomers^[2–6]. Another approach to increasing the processability is the introduction of a bulky pendant group^[7] or a polar, rigid and zigzag group (heterocyclic monomer) into the main chains of polyamide^[8–10]. Among various heterocyclic units which have been introduced into the main chain of aromatic polyamides, the phenylquinoxaline rings proved to be very beneficial^[11, 12]. Such modifications in the aromatic backbone of polymers cause a decrease in the crystallinity and improve solubility in organic solvents and therefore better capability to be processed from solutions, while maintaining a high thermal resistance^[13, 14]. Preparation of polyamides based on quinoxaline ring and its related derivatives has been under investigation by many researchers. Akutsu *et al.* has prepared aromatic polyamides containing quinoxaline ring by transformation of α -diketone moieties in the main chain^[15] and by polycondensation of 4,4'-(2,3-quinoxalinediyl)dibenzoyl chloride and 4,4'-(2,3-benzo[g]quinoxalinediyl)benzoic acid with aromatic diamines^[16–19]. They have also reported the preparation of polyamides and polyimides from 2,3-bis(4-aminophenyl)quinoxalines and aromatic dicarboxylic acids and tetracarboxylic dianhydrides^[20, 21]. Hedrick *et al.*^[22] reported preparation of polyimide

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copolymers containing imide-aryl ether phenylquinoxaline by polycondensation of aryl ether phenyl-quinoxaline diamines with dianhydrides. There are reports of preparation of copolymers containing phenylquinoxaline rings in the main chain^[23, 24]. Jenekhe *et al.*^[25] reported the preparation of copolymers of 9,9-dioctylfluorene and 2,3-bis(*p*-phenylene)quinoxaline. They used 2,3-bis(4-bromophenyl)quinoxaline as a monomer which was prepared by the reaction of *o*-phenylene diamine with 4,4'-dibromobenzil. Direct polycondensation methods have been developed by several investigators and especially by Higashi^[26, 27]. In direct polycondensation, preparation and separation of acid chlorides are not necessary and the reactions proceed as one pot reaction using the free carboxylic acid, diamines and compounds such as CaCl₂, LiCl, triphenylphosphite, phosphorousoxychloride, *etc*^[27, 28].

In our study, the reaction of *o*-phenylene diamine with 4,4'-dinitrobenzil, the same method as Ref. [25], was used for the preparation of 2,3-bis-(*p*-nitrophenyl)-quinoxaline. The total yield for the preparation of 2,3-bis-*p*-aminophenylquinoxaline monomer by this method was about 10%. The present work describes the synthesis and characterization of quinoxaline based polyamides by direct polycondensation reaction of this diamine with different aliphatic, aromatic and heterocyclic dicarboxylic acids containing pyridine and imidazole rings. To the best of our knowledge, however, no paper has dealt with the preparation of polyamide from 2,3-bis-*p*-aminophenylquinoxaline with pyridine-2,6-dicarboxylic acid and 2-phenyl-1H-imidazole-4,5-dicarboxylic acid. The prepared polyamides were completely characterized by elemental analysis, FTIR, ¹H-NMR, solubility, inherent viscosity, thermal properties and thermal stability.

EXPRIMENTAL

Materials and Instruments

Benzoin, urea, *o*-phenylene diamine and all other chemicals were purchased from Fluka (Switzerland). ¹H-NMR spectra were recorded on a Bruker advance DRX 500 MHz instrument, using DMSO-d₆ as solvent and tetramethyl silane as an internal standard. FTIR spectra were recorded on a Bruker, Vector 22 spectrometer, using KBr pellets. The CHN-600 Leco analyzer was used for elemental analysis. Thermal gravimetric analysis (TGA) and differential scanning calorimetry (DSC) were performed at 10 K/min under N₂ atmosphere by using a Perkin-Elmer pyres and a Metler Toledo 822°, respectively. Inherent viscosity ($\eta_{inh} = \ln \eta_{rel}/c$) of polymers was determined for solutions of 0.5 g/dL in DMSO at 25°C using an Ubbelohde viscometer.

Synthesis

The reaction steps for the synthesis of compounds 4,5-diphenylglyoxalone (I), 4,4'-dinitrobenzil (II) and 2,3-bis(*p*-nitrophenyl)quinoxaline (III) are shown in Scheme 1. Compounds (I) and (II) were synthesized according to the procedures given in the literature^[29]. The yields for the compounds (I) and (II) were 71.5% and 21% with the melting points of 330°C and 213°C, respectively. Compound (III) was synthesized according to the procedure given in the literature^[30], and the yield for this compound was 77% with the melting point of 201°C.

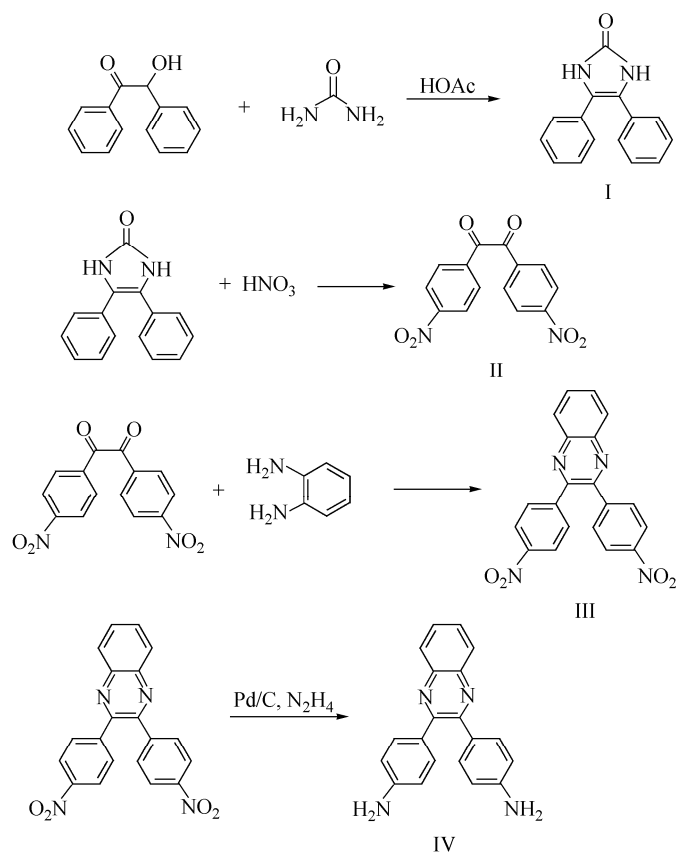
*Synthesis of 2,3-Bis-*p*-aminophenylquinoxaline (IV)*

A solution of 4.46 g (12 mmol) of dinitro compound (III) and 0.4 g of Pd/C (10% Pd) in 200 mL of ethanol in a flask equipped with dropping funnel, reflux condenser and thermometer was heated for 2 h, and a solution of 10 mL hydrazine hydrate 85% in 20 mL ethanol was added drop wise in 1 h. The reaction was refluxed for 2 h and the hot solution was filtered. The precipitate was formed during cooling the filtrate which was filtered and dried in a vacuum oven. The powder of 2,3-bis-*p*-aminophenylquinoxaline was obtained in a yield of 85% with melting at about 265°C.

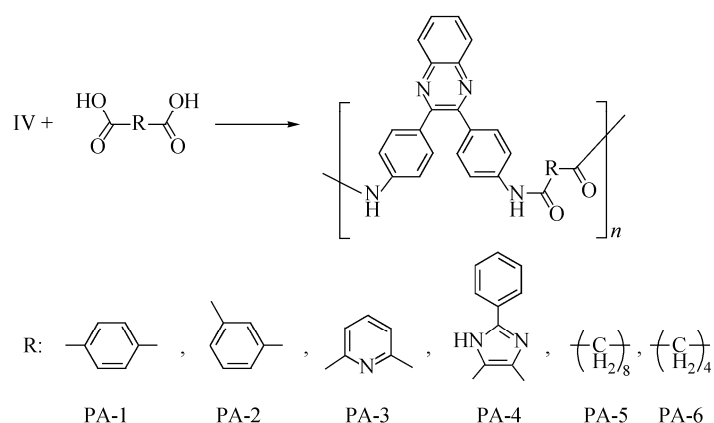
Synthesis of Model Compound (V)

Model diamide (V) was prepared from 2,3-bis-*p*-aminophenylquinoxaline and benzoic acid by using a conventional method (Scheme 2). A two-necked flask equipped with a magnetic stirrer, a condenser and a nitrogen inlet/outlet tube was charged with the diamine (IV) (0.312 g, 1 mmol), benzoic acid (0.244 g, 2 mmol), a mixture of 30 mL NMP and 6 mL pyridine, triphenyl phosphite (4.1 mmol, 1.3 mL), 0.6 g LiCl, and 1.8 g

CaCl_2 . The reaction was heated at 110°C for 10 h under a stream of N_2 , and the extent of reaction was followed by thin layer chromatography (TLC). The solution was then poured into methanol. A precipitate was formed which was filtered off and washed with refluxing methanol for 24 h. The solid product was then dried in a vacuum oven at 100°C , mp 350°C .



Scheme 1 Synthesis reactions for preparation of diamine compound



Scheme 2 Synthesis reactions for preparation of polyamides

Synthesis of Polyamides

Synthesis of polyamides is shown in Scheme 2, and a typical procedure for the preparation of polyamides is given below: A two-necked flask equipped with a magnetic stirrer, a condenser and a nitrogen inlet/outlet tube

was charged with 0.624 g (2 mmol) diamine (IV) and 2 mmol dicarboxylic acid such as terephthalic acid, isophthalic acid, pyridine-2,6-dicarboxylic acid, 2-phenyl-1H-imidazole-4,5-dicarboxylic acid, adipic acid, or sebacic acid in a mixture of 30 mL NMP and 6 mL pyridine containing 1.3 mL triphenyl phosphite, 0.6 g LiCl, and 1.8 g CaCl_2 . The reaction mixture was heated at 110°C for 10 h under N_2 . The reaction solution was poured into methanol; the precipitate was filtered off and washed with refluxing methanol for 24 h. The solid product was dried in a vacuum oven at 100°C .

RESULTS AND DISCUSSION

2,3-Bis-4-nitrophenylquinoxaline (III) was synthesized in three steps from benzoin, urea and 1,2-phenylenediamine according to the procedures given in the literatures^[18, 19]. Data from FTIR and ^1H -NMR spectra and melting point were used to confirm the structures of the compounds I, II, and III which were in good agreement with those given in the literatures. Scheme 1 displays the structures of starting materials and the products I, II, III and IV. FTIR spectra of dinitro compound III showed characteristic peaks (cm^{-1}) at: 1517–1678 ($\text{C}=\text{C}$ and $\text{C}=\text{N}$ stretching), 1523 and 1339 (cm^{-1} asymmetrical and symmetrical NO_2 stretching), 3100 cm^{-1} (aromatic $\text{C}-\text{H}$), and peak in the region 2250–2460 cm^{-1} was related to the water vapor and carbon dioxide. The reduction of compound III to 2,3-bis-*p*-aminophenylquinoxaline (IV) by hydrazine hydrate was carried out in ethanol in the presence of Pd/activated carbon as catalyst. ^1H -NMR and ^{13}C -NMR of the diamino compound (IV) are shown in Figs. 1 and 2, respectively.

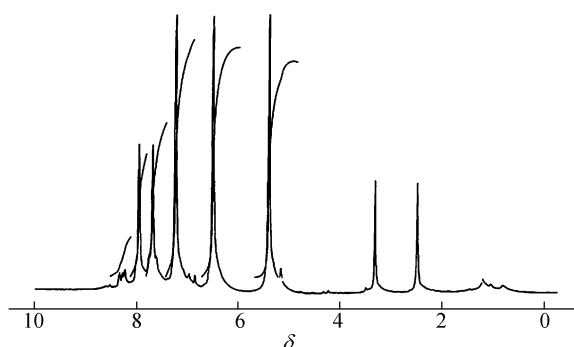


Fig. 1 ^1H -NMR spectrum of diamine compound (IV)

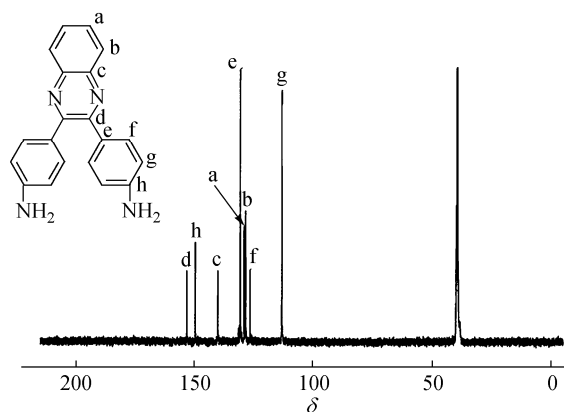


Fig. 2 ^{13}C -NMR spectrum of diamine compound (IV)

FTIR spectrum of compound (IV) showed characteristic absorption bands at (cm^{-1}): 3337, 3217 ($\text{N}-\text{H}$ stretching) and 1585–1607 ($\text{C}=\text{C}$ and $\text{C}=\text{N}$ stretching). ^1H -NMR spectrum showed signals at δ : 7.96, 7.69 (4H, quinoxaline ring), 7.25, 7.23 (4H, phenyl ring), 6.52, 6.50 (4H, phenyl ring), 5.4 (4H, NH_2). ^{13}C -NMR spectra showed characteristic bands at δ = 153 and 140 for carbons of quinoxaline ring and δ = 149 for carbons next to NH_2 groups. The detailed descriptions of ^{13}C -NMR spectra of the diamine compound (IV) are shown in Fig. 2 with the designated carbon atoms. 2,3-Bis-*p*-aminophenylquinoxaline (IV) was used as a starting material for the preparation of model compound in reaction with benzoic acid. The reaction of IV (1 mmol) with the benzoic acid (2 mmol) was conducted in a mixture of 30 mL NMP and 6 mL pyridine in the presence of triphenyl phosphite, LiCl and 1.8 g CaCl_2 at 110°C for 10 h under a stream of nitrogen. The structure of model compound was confirmed by using FTIR, and the spectrum is shown in Fig. 3. The FTIR spectra showed the characteristic peaks at (cm^{-1}): 3363 ($\text{N}-\text{H}$, amid), 2890 ($\text{C}-\text{H}$, aromatic) and 1658 ($\text{C}=\text{O}$).

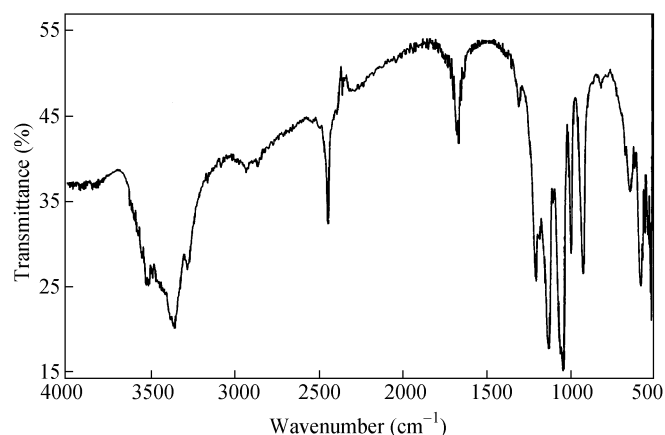


Fig. 3 FTIR spectrum of model compound (V)

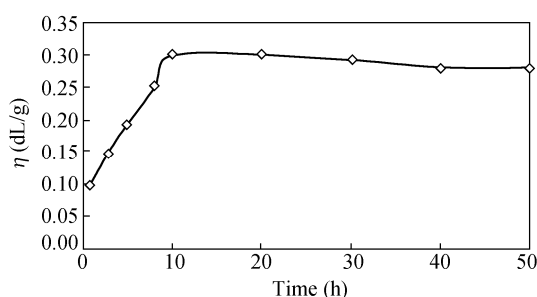


Fig. 4 Viscosity versus reaction time

Polyamides were prepared in good yields by reaction of 2 mmol of 2,3-bis-*p*-aminophenylquinoxaline (IV) with 2 mmol of dicarboxylic acid such as terephthalic acid, isophthalic acid, pyridine-2,6-dicarboxylic acid, 2-phenyl-1H-imidazole-4,5-dicarboxylic acid, adipic acid, or sebacic acid in NMP containing (PhO)₃P, LiCl and CaCl₂. Pyridine was also added to the mixture which acted as a complexation agent. The polycondensation reaction and polyamide designations are shown in Scheme 2. The structure of the synthesized polyamides was confirmed by elemental analysis and spectroscopic methods. The presence of amidic bands at *ca.* 3320 cm⁻¹ and *ca.* 1655 cm⁻¹ related to N—H and C=O stretching, respectively, in the FTIR spectra and amidic proton signal at *δ ca.* 10.5 in the ¹H-NMR spectra confirmed the amidic structure of the polyamides. The inherent viscosity of the polyamides was also measured at a concentration of 0.5 g/dL in DMSO at 25°C, and the results are given in Table 1. The inherent viscosity of the polymerization mixture was measured with the reaction time, as shown in Fig. 4. Viscosity increased to a maximum of 0.3 dL/g after 12 h and remained almost constant up to 50 h of reaction time. The yields, elemental analysis and characteristic peaks of the spectroscopic methods are listed in Table 2. Typical FTIR and ¹H-NMR spectra of the representative polyamides are shown in Figs. 5 and 6, respectively. The intense ¹H-NMR signal at *δ* = 6 in Fig. 6 can probably be related to tautomerism of protons between nitrogen atoms inside the aromatic ring of pyrazine and the amide group along the polymer chain as it is illustrated below:

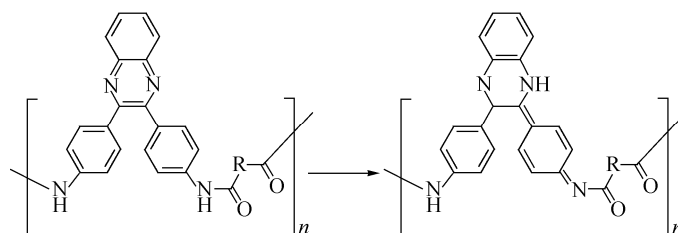


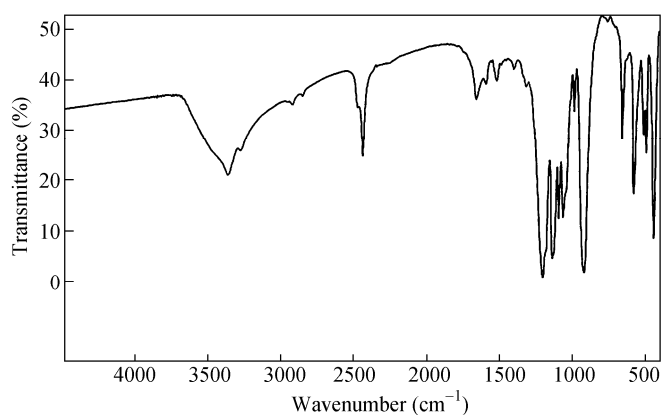
Table 1. Solubility and inherent viscosity of polyamides

Polymer code	NMP	DMF	DMSO	TCE	Py	THF	H ₂ SO ₄	HMPA	DMAC	Aceton	η_{inh} (dL/g)
PA-1	+	+	+	+	+	±	+	+	±	+	0.27
PA-2	+	+	+	+	+	±	+	+	±	+	0.30
PA-3	+	+	+	±	±	±	+	+	±	+	0.25
PA-4	+	+	+	±	±	±	+	+	±	+	0.25
PA-5	+	+	+	±	±	±	+	+	±	±	0.30
PA-6	+	+	+	±	±	±	+	+	±	±	0.27

+ Soluble, ± Partially soluble

Table 2. Spectra data, elemental analysis results and the yields of model compound and polyamides

Substrate	IR (KBr, cm ⁻¹)	¹ H-NMR (δ, DMSO-d ₆)	Elemental analysis						Yield (%)
			Calc.			Found			
			C	H	N	C	H	N	
PA-1	3362 (N—H, amid),	8.12, 7.85, 7.52, 7.48,							
	2880 (C—H, arom.), 1664 (C=O)	7.25 (16H, Aro), 10.54 (2H, Amid)	76.0	4.07	12.6	75.9	4.1	12.4	95
PA-2	3360 (N—H, amide),	7.48, 7.47, 7.27, 7.10,							
	2860 (C—H, arom.), 1666 (C=O)	7.06 (16H, Aro), 10.5 (2H, Amid)	76.0	4.07	12.6	75.9	4.1	12.6	95
PA-3	3385 (NH-amide),	7.93, 7.90, 7.86, 7.84,							
	3050 (CH-Arom.), 1620 (C=O)	7.27, 7.05 (15H, Aro), 10.5 (2H, Amid)	73.1	3.8	15.8	73.5	3.9	15.5	94
PA-4	3331 (NH-amide),	7.93, 7.90, 7.86, 7.84,							
	2950 (CH-Arom.), 1666 (C=O)	7.27, 7.05 (17H, Aro), 10.5 (2H, Amid)	73.2	3.9	16.5	73.1	3.8	16.6	92
PA-5	3360 (NH-amide),	7.48, 7.29, 7.10, 7.09							
	2980 (CH-Arom.), 2880 (methylen group), 1666 (C=O)	(12H, Aro), 10.5 (2H, Amid), 1.65–0.78 (16H, Aliph)	75.3	6.2	11.7	75.1	6.3	11.9	94
PA-6	3357 (NH-amide),	7.48, 7.29, 7.10, 7.09							
	2920 (CH-Arom.), 2820 (methylen group), 1666 (C=O)	(12H, Aro), 10.5 (2H, Amid), 1.48–0.77 (8H, Aliph)	73.9	5.2	13.2	73.8	5.1	12.9	94

**Fig. 5** FTIR spectrum of polyamide (PA-1)

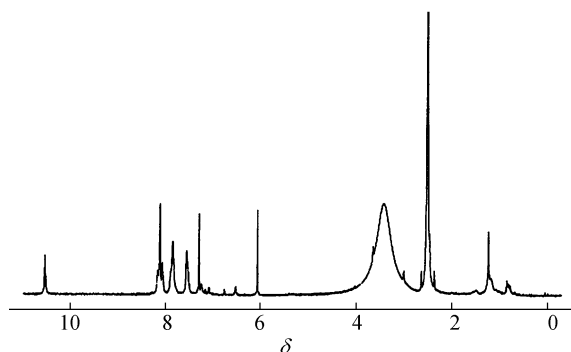


Fig. 6 ^1H -NMR spectrum of polyamide (PA-1)

The solubility of polyamides was tested in different organic solvents at room temperature, and the results are shown in Table 1. All aromatic and aliphatic polyamides were soluble in common organic solvents such as DMF, NMP, DMSO and HMPA. The aromatic polyamides with heterocyclic rings in the backbone such as PA-3 and PA-4 were also soluble in the above mentioned solvents and the polyamides containing aliphatic units in the backbone such as PA-5 and PA-6 were not soluble in solvents such as TCE, pyridine, THF and DMAC.

DSC thermograms of model compound and the representative polyamides are shown in Figs. 7–10. DSC of model compound showed an endothermic peak in the temperature range of 200–300°C (Fig. 7, curve a) during first scan which was not observed during second run (curve b) after cooled to room temperature. The DSC thermograms of polyamides similar to those of the model compound showed, Figs. 8–10, endothermic peak in the temperature region of 200–300°C which was not observed during second scans. Samples of the model compound and polyamides were solvent extracted for removing any possible impurities. The extracted samples were dried in the oven and used for DSC analysis. Thermograms of the extracted samples also showed the endothermic peak only during first scan in the same temperature region. We do not have any reasonable explanation for this transitional behavior. The polyamides did not show crystallization and melting peak during cooling and heating processes. Thermal stability of the polyamides was evaluated by thermogravimetric analysis (TGA) in the atmosphere of nitrogen and at a heating rate of 10 K/min. TGA data are summarized in Table 3. TGA curves of the representative polyamides are shown in Fig. 11. The relative thermal stability of the polyamides was determined by noting the temperatures of 10% and 20% weight losses and percent char yield at 600°C. The initial weight loss of about 10% which started in the temperature range of 50–100°C can be due to the loss of absorbed moisture. A major weight loss of about 20% occurred in the temperature range of 350–450°C for aromatic-aromatic polyamides and 200–250°C for aliphatic-aromatic polyamides indicating high thermal stability of the polymers. There is no significant weight loss beyond these temperatures. The char yields of the polyamides, which correlates to the ability to sustain combustion, were high (about 72% for aromatic-aromatic and about 65% for aromatic-aliphatic polyamides) at 600°C. The char yield tended to depend on the structure of the repeating unit of the polymer chains, and it was high for aromatic-aromatic based polyamides which can be due to the stiffness of the polymer chain. Similar and higher thermal stability and percentage of the char yields were reported for different thermal stable polymers by other researchers^[31–34]. Char yield can be used as criteria for evaluating limiting oxygen index (LOI) of the polyamides in accordance with the Van Krevelen and Hoftyzer equation^[35].

$$\text{LOI} = 17.5 + 0.4 \text{ CR}$$

where CR = char yield. All polyamides had LOI values calculated based on their char yield higher than 28. On the basis of LOI values, such materials can be classified as self-extinguishing and flame resistant polymers.

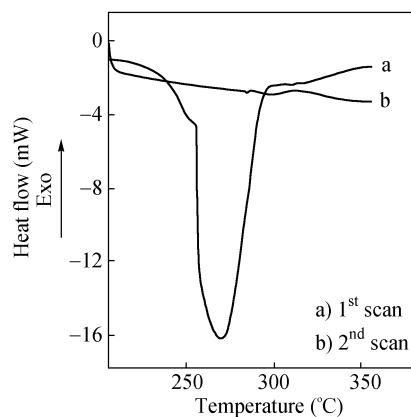


Fig. 7 DSC thermograms of model compound

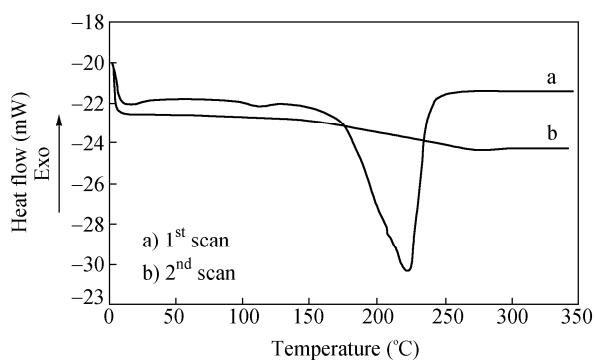


Fig. 8 DSC thermograms of polyamide PA-1

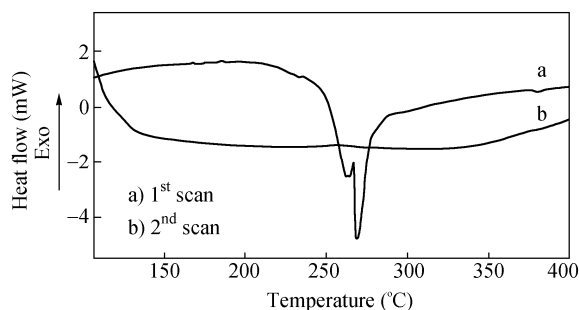


Fig. 9 DSC thermograms of polyamide PA-2

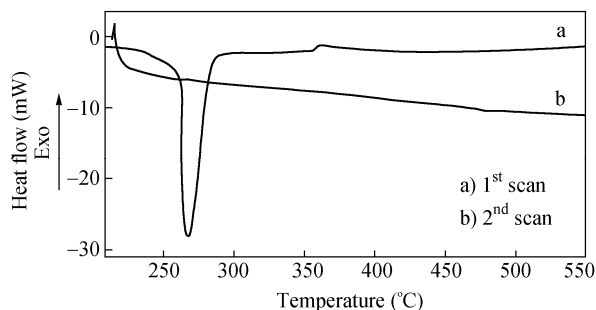


Fig. 10 DSC thermograms of polyamide PA-4

Table 3. Thermal analysis data

Compound code	$T_{10\%}$ (°C)	$T_{20\%}$ (°C)	Char yield	LOI
PA-1	< 100	350	70	45.5
PA-3	< 100	420	82	50.3
PA-6	—	200	65	43.5

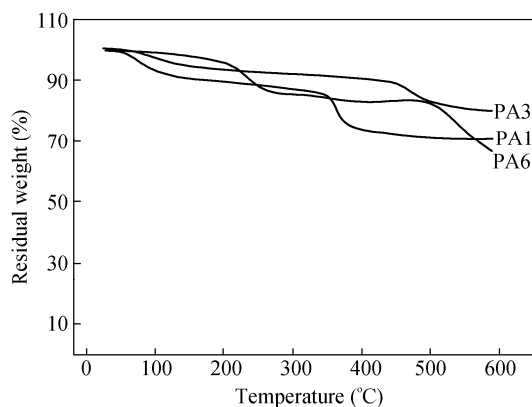


Fig. 11 TGA curves of polyamides PA-1, PA-3 and PA-6

CONCLUSIONS

A series of polyamides containing quinoxaline ring in the main chain were prepared from the reaction of six different dicarboxylic acids with the prepared diamine. The polyamides were fully characterized and their thermal properties were studied. All the polyamides were soluble in common aprotic organic solvents such as

DMF, NMP and DMSO. The aromatic polyamides having pyridine and imidazole moieties in the backbone showed a nice balance of solubility and thermal stability.

The prepared polyamides did not show melting or crystallization temperatures and thus are amorphous polymers. DSC thermograms of the model compound and the polyamides showed an unexplainable endothermic transitional peak above 200°C. TGA traces of aromatic-aromatic polyamides showed a minor weight loss of about 20% above 400°C, and there was no significant weight loss beyond this decrease. The char yields were 82% for heterocyclic polyamides, 70% for aromatic polyamides and 65% for aliphatic-aromatic polyamides at 600°C. On the basis of LOI values, the prepared polyamides can be classified as self-extinguishing and flame resistant polymers.

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