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ORIGINAL PAPER

Synthesis, characterization, and properties of novel aromatic polyamides containing phthalazinone moiety

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Abstract A unsymmetrical and kink non-coplanar heterocyclic dicarboxylic acid monomer, 4-[4-(4-carboxy phenoxy)-naphthyl]-2-(4-carboxyphenyl)phthalazin-1-one (**3**) was successfully synthesized with high purity and high yields. A series of novel polyamides containing phthalazinone were prepared from the newly synthesized dicarboxylic acid with various aromatic diamines by means of the phosphorylation polycondensation reaction. Molecular weights of the obtained polyamides were evaluated viscometrically, and the inherent viscosities (η_{inh}) measured were in the range 0.54–0.69 dL/g. These polyamides were amorphous and readily soluble in many organic solvents, such as *N*-methyl-2-pyrrolidone, *N*,*N*-dimethylacetamide, *N*,*N*-dimetheylformamide, dimethyl sulfoxide, pyridine, and m-cresol etc., and they could easily be solution-cast into transparent, flexible films with good mechanical properties, with tensile strength ranging from 63.9 to 81.6 MPa and elongation at break from 7.2 to 11.4%. These polymers still kept good thermal stability with high-glass transition temperatures in the range of 283–338 °C, and the decomposition temperature in nitrogen for a 10% weight-loss temperatures in excess of 490 °C, and char yield at 800 °C in nitrogen ranged from 56 to 63%. Furthermore, the polyamides films were essentially colorless; their cut-off wavelengths were between 365 and 379 nm.

Introduction

As a class of high-performance materials, wholly aromatic polyamides exhibit superior properties, such as very high thermal stability, good mechanical and

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electrical behavior, as well as low flammability, so they have been widely used in fields of electrical materials, adhesives, composites and membranes [1–4]. However, aromatic polyamides are generally difficult to process due to their poor solubility in organic solvents and high flowing or melting temperature, which are caused by the rigidity of the backbone and present strong hydrogen bonding in macromolecules [5, 6]. Therefore, a great deal of efforts have been made to improve processability and solubility of polyamides without sacrificing their excellent properties via structure modification, typically by introduction of bulky substituents or cardo groups [7–10], asymmetric units [11, 12], and flexible links or bridging functional groups to the main polymer chains [13–15].

Since Hay et al. first found that 1,2-dihydro-4-(4-hydroxyphenyl)(2H) phthalazinones were bisphenol-like monomers [16], then a number of polymers such as poly(phthalazinone ether)s, polyamides, polyimides, and polyesters has been developed from the series of monomer [17–21]. The incorporation of the twisted, non-coplanar structure into the polymer backbone would disrupt inter-chain hydrogen bonding and reduce the chain packing efficiency and crystallinity which could promote solubility. Moreover, introducing heterocyclic aromatic rings into the backbone can retain acceptable thermal properties with high T_g through controlled segmental mobility. Furthermore, it has been demonstrated that incorporation both ether and naphthyl units into the polymer backbones is a successful route to improve the solubility of polyamides while retaining high thermal stability too [22, 23]. The combination of the two above effective factors with each other may be proposed as suitable choice for increasing the processability of aromatic polymers without extreme loss of thermal resistance.

In the present article we report the successful synthesis of a new dicarboxylic acid containing phthalazinone (DHPZ) moiety in the main chain. The new dicarboxylic acid was directly polycondensed with various aromatic diamines to produce a series of novel organosoluble polyamides. The properties of these polymers, such as crystallinity, solubility, optical transparency, thermal and mechanical properties of these polymers are also discussed herein.

Experimental

Materials

4-(4-hydroxynaphthalenyl)phthalazin-1-one **1** was synthesized in our laboratory, details of the synthesis and characterization data of the new bisphenol-like compound were described in a separate article [24]. According to a well-established procedure [25], 1,4-bis(4-aminophenoxy)benzene (**4c**, m.p. 171–172 °C) and 1,4-bis(4-amino-2-trifluoromethylphenoxy)benzene (**4d**, m.p. 133–134 °C) were prepared by the aromatic nucleophilic substitution reaction of hydroquinone with p-chloronitrobenzene and 2-chloro-5-nitrobenzotrifluoride, respectively, in the presence of potassium carbonate, and subsequent reduction of the intermediate dinitro compounds using hydrazine as the reducing agent and palladium as the catalyst. p-Phenylenediamine (**4a**) were purified by distillation under reduced pressure before use, 4.4′-oxydianiline (**4b**) were purified by recrystallization from ethanol. N-methyl-2- pyrrolidone (NMP) and pyridine (Py) were purified by distillation under reduced pressure over calcium hydride and stored over 4Å molecular sieves. Triphenyl phosphite (TPP) was purified by distillation under reduced pressure. Commercially obtained anhydrous calcium chloride ($CaCl_2$) was dried under vacuum at 180 °C for 10 h. All other materials were reagent-grade and used without further purification unless otherwise noted.

Measurement

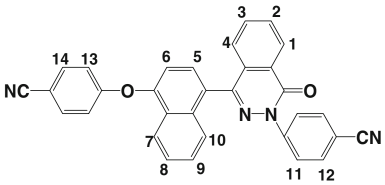
IR spectra were recorded in the range 4,000–500 cm^{-1} by Nicolet Magna 470 spectrometer. Elemental analyses were made on a Carlo-Erba1106 instrument. The inherent viscosities of all polymers were determined at 0.5 g/dL concentration with Ubbelohde viscometer at 30 °C. NMR spectra were recorded using Bruker AM 400 MHz instrument. Differential scanning calorimetric analysis was performed on differential scanning calorimeter (TA 2900M) at a heating rate of 20 °C/min. Thermogravimetric data were obtained on a DuPont 2000 SDT-2960 under nitrogen flowing condition at a rate of 50 cm^3/min and a heating rate of 20 °C/min. Dynamic mechanical analysis (DMA) was performed on thin films (30-mm long, 10-mm wide, 50–60 μm thick) on a DMTA-4 Analyzer (America Rheogoniometer Co.) in the tensile mode at frequency of 1 Hz over the temperature range 30–400 °C at a heating rate of 5 °C/min in nitrogen. Ultraviolet-visible (UV-vis) spectra of the polymer films and the dilute *N*,*N*-dimetheylformamide (DMF) solution were recorded on a Lambda 35 (Perkin Elmer) spectrophotometer. The emission spectra were measured with a Perkin Elmer LS50B luminescence spectrometer in a dilute DMF solution. Wide-angle X-ray diffraction (WAXD) patterns were obtained at room temperature on a Rigaku D/MAX 2500 powder diffractometer with a scanning speed of 4°/min, and the patterns were recorded in the 2θ range of 5°–40°. Mechanical properties of the films were measured on a Instron model 1,130 tensile tester with a 5 kg load cell at a crosshead speed of 5 cm/min on strips approximately 35–60 μm thick and 0.5 cm wide with a 2 cm gauge length.

Monomer synthesis

*4-[4-(4-cyano phenoxy)-naphthyl]-2-(4-cyano phenyl)phthalazin -1-one (**2**)*

In a 250 mL round-bottomed flask equipped with a nitrogen inlet, a Dean-Stark trap and a condenser, 13.8 g (0.1 mol) of potassium carbonate were suspended in a solution of bisphenol-like compounds **1** 11.53 g (0.04 mol) in a mixture of 70 mL of *N*,*N*-dimethylacetamide (DMAc) and 50 mL toluene, then the reactive system was heated with stirring at reflux temperature for 5 h. After the toluene has been removed completely, the mixture was cooled and then 11.01 g (0.08 mol) of p-chlorobenzonitrile was added. The reaction was carried out at 150 °C for 15 h, and then the reaction mixture was poured into 800 mL of cold water. The products was filtered and recrystallized from ethanol/DMAc (V/V = 1:3) and was dried to afford 13.10 g (yield 69%) of yellowish powder, mp. 197–198 °C (by DSC). IR

Scheme 1 Chemical structure of dinitrile (**2**)

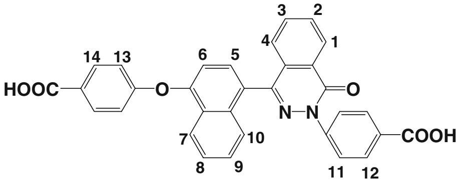


(KBr, cm^{-1}): 2,226 (C$\equiv$N), 1,675 (C=O), 1,243 (C–O–C).^{1}H NMR(DMSO-d$_6$, ppm) (Scheme 1): δ 8.50 (d, 1H, 1-H), 8.07 (d, 1H, 7-H), 7.99 (m,4H, 12-H,14-H), 7.95 (t, 1H, 2-H), 7.88 (m, 4H, 3-H,10-H,11-H), 7.78 (d, 1H, 5-H), 7.62 (t, 1H, 8-H), 7.55 (t, 1H, 9-H), 7.41 (d, 1H, 6-H), 7.32 (d, 1H, 4-H), 7.27 (d, 2H, 13-H), ^{13}C NMR(DMSO-d$_6$, ppm): 161.26, 158.30, 151.05, 146.51, 145.31, 134.71, 134.10, 133.42, 132.64, 132.45, 129.87, 128.97, 128.55, 127.96, 127.75, 127.06, 127.03, 126.83, 126.48, 126.17, 126.03, 121.55, 118.58, 118.42, 118.08, 115.50, 109.70, 105.46. Anal. Calcd for $C_{32}H_{18}N_4O_2$: C, 78.36%; H, 3.70%; N, 11.42%. Found: C, 78.11%; H, 3.68%; N, 11.57%.

*4-[4-(4-carboxy phenoxy)-naphthyl]-2-(4-carboxy phenyl)phthalazin-1-one (**3**)*

The dicarboxylic acid **3** was obtained by alkaline hydrolysis of the dinitrile compound **2**. In a 250 mL round-bottomed flask, a suspension of dinitrile **2** (9.80 g, 0.02 mol) in an water/ethanol (75 mL/75 mL) containing dissolved 22.40 g (0.4 mol) of potassium hydroxide was boiled under reflux with nitrogen atmosphere. Reflux was continued until the evolution of ammonia had ceased. The result clear solution was cooled, and the PH value was adjusted by dilute hydrochloric acid to near 3. The offwhite precipitate was collected by filtration and washed thoroughly with distillated water and methanol, then dried in vacuum, to give 10.25 g (yield 97%) offwhite powder without further purification, mp. 166–168 °C (by DSC). IR (KBr, cm^{-1}): 3,070 (broad, C(O)O–H), 1,692 (C=O), 1,239 (C–O–C). ^{1}H NMR(DMSO-d$_6$, ppm) (Scheme 2): δ 12.93 (s, 2H, COOH), 8.51 (d, 1H, 1-H), 8.14 (d, 1H, 7-H), 8.07 (d, 2H, 12-H), 8.00 (d, 2H, 14-H), 7.96 (t, 1H, 2-H), 7.88 (m, 4H, 3-H,10-H,11-H), 7.77 (d, 1H, 5-H), 7.60 (t, 1H, 8-H), 7.56 (t, 1H, 9-H), 7.34 (d, 1H, 6-H), 7.31 (d, 1H, 4-H), 7.21 (d, 2H, 13-H), ^{13}C NMR (DMSO-d$_6$, ppm): 166.68, 166.64, 161.05, 158.25, 151.82, 146.26, 145.13, 133.97, 133.38, 132.33, 131.71, 129.89, 129.59, 129.54, 128,56, 128.47, 127.99, 127.69, 126.95, 126.86, 126.78, 126.18, 125.91, 125.73, 125.71, 121.69, 117.30, 114.75. Anal. Calcd for $C_{32}H_{18}N_4O_2$: C, 72.72%; H, 3.81%; N, 5.30%. Found: C, 72.49%; H, 3.77%; N, 5.45%.

Scheme 2 Chemical structure of diacid (**3**)



Polymers synthesis

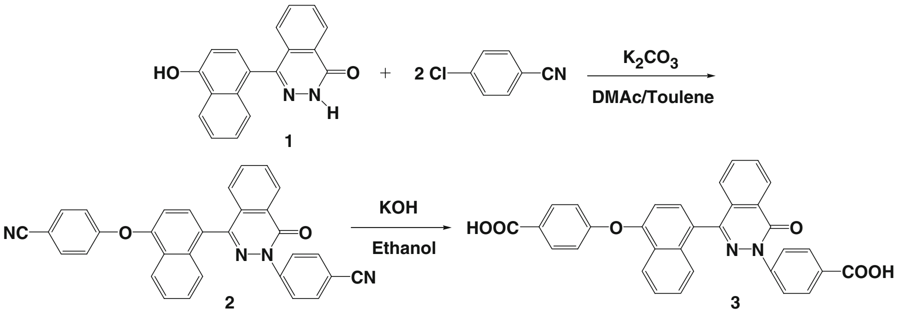
The synthesis of 5d is given as a typical example for the preparation of the polyamides. A mixture of 0.7927 g (1.5 mmol) of diacid **3**, 0.6425 g (1.5 mmol) of 1,4-bis(4-amino-2-trifluoromethylphenoxy)benzene (**4d**), 0.30 g dried calcium chloride, 1.2 mL of Py, 1.8 mL of triphenyl phosphite, and 3.0 mL of NMP was heated with stirring at 110 °C until the magnetic stirrer could no longer work. The polymer solution was poured slowly into 300 mL of stirring methanol giving rise to a fiber-like precipitate which was washed thoroughly with hot water and methanol, collected by filtration, and dried under vacuum at 120 °C. The yield (99.6%) was quantitative. Other polyamides were synthesized by an analogous method. The film preparation was performed via the procedure of Hsiao's experience [14], then the obtained films were used for X-ray diffraction measurements, solubility test, tensile test, and thermal analyses.

Results and discussion

Monomer synthesis

As shown in Scheme 3, the new phthalazione-containing aromatic dicarboxylic acid was synthesized by alkaline hydrolysis of the dinitrile compound resulting from the nucleophilic substitution reaction of 4-(4-hydroxynaphthalenyl)phthalazin-1-one **1** with p-chlorobenzonitrile. All synthesized compounds were characterized by elemental analysis, FT-IR and NMR spectroscopy with satisfied results. Figure 1 shows the IR spectra of dinitrile and diacid compound. The characteristic bands representative of the cyano functionality were identified in the IR spectrum at 2,228 cm^{-1}, where it disappeared in IR spectrum of diacid **3**, and instead a broad O–H absorption appeared in the region of 2,600–3,500 cm^{-1}. Furthermore, in the IR spectrum of diacid, the characteristic absorption band at 1,689 cm^{-1} signed to C=O stretching became wider than that of dinitrile compound, implying more presence of carbonyl group existing in diacid.

The chemical structures compound **2** and monomer **3** were also confirmed by ^{1}H NMR spectroscopy. Figure 2 shows ^{1}H NMR spectrum of monomer **3**, in which all



Scheme 3 Synthesis of the unsymmetrical diacid monomer containing the phthalazinone moiety

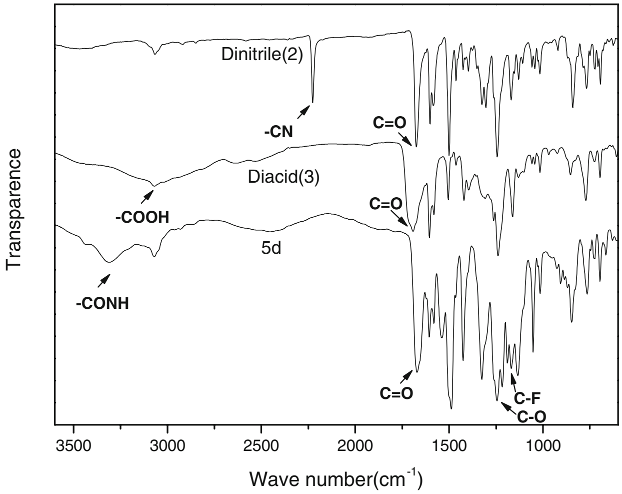


Fig. 1 FTIR spectra of compound **2**, monomer **3** and polymer **5d**

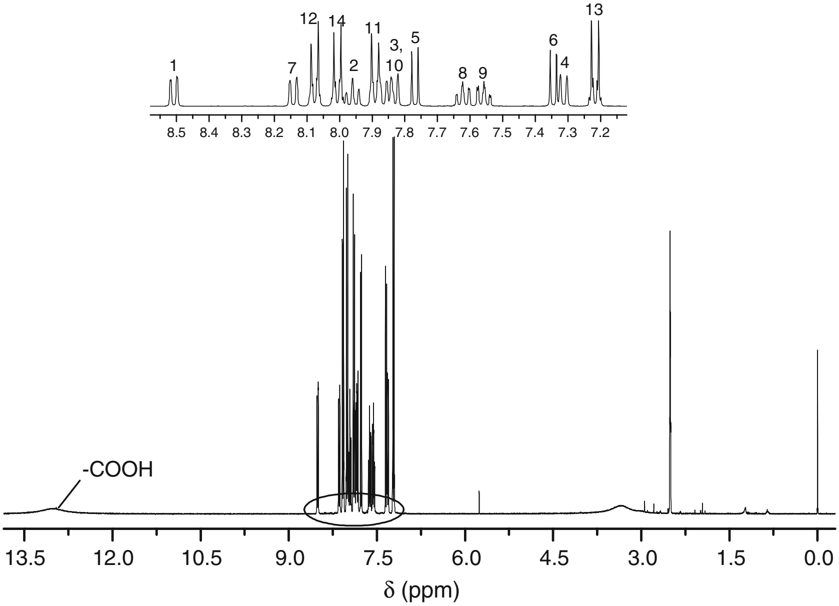
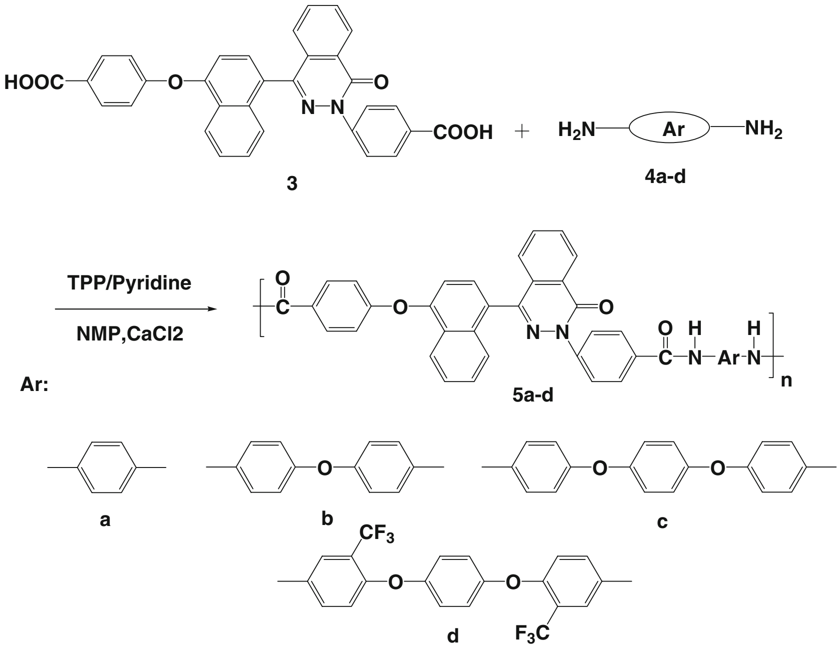


Fig. 2 ^{1}H NMR spectrum of monomer **2** in DMSO-d_6

the protons were assigned as shown in scheme 2. In addition, the corrected elemental analysis values were identical with the calculated ones.

Polymer synthesis

Polymerization of diacid **3** with stoichiometric amount of different aromatic diamines was carried out in dry freshly distilled NMP and Py outlined in Scheme 4.



Scheme 4 Synthesis of polyamides containing the phthalazinone moiety

The direct polycondensation of aromatic dicarboxylic acid with diamines using a mixture of triphenyl phosphite and Py as condensing agent is a convenient method on a laboratory scale [26]. All polyamidations proceeded in a homogeneous system throughout the reaction, and the polyamides were isolated as tough fibers in quantitative yields. The synthesis conditions and inherent viscosities of the resulting polyamides are summarized in Table 1. Inherent viscosities of the polyamides ranged from 0.54–0.69 dL/g, indicative of the formation of high molecular weights. Among these polymers, the one (**5c**) showed the highest inherent viscosity maybe

Table 1 Synthesis condition, inherent viscosity, and film quality of polyamides

Polymer code	Amount of reagents used[a]				Yield (%)	η_{inh}^{b} (dL/g)	Film quality[c]
	NMP (mL)	Py (mL)	TPP (mL)	$CaCl_2$ (g)			
5a	2.8	1.2	1.8	0.25	99.1	0.63	Flexible
5b	2.8	1.2	1.8	0.28	98.4	0.58	Flexible
5c	3.0	1.2	1.8	0.28	99.4	0.69	Flexible
5d	3.0	1.2	1.8	0.30	99.6	0.54	Flexible

[a] Reaction runs in NMP with equal equivalent (1.5 mmol) of diacid and diamine in nitrogen at 110 °C. Reaction time = 3.0 h

[b] Measured in DMAc with a concentration of 0.5 g/dL at 30 °C

[c] Films were casted by slow evaporation of polymer solution in DMAc

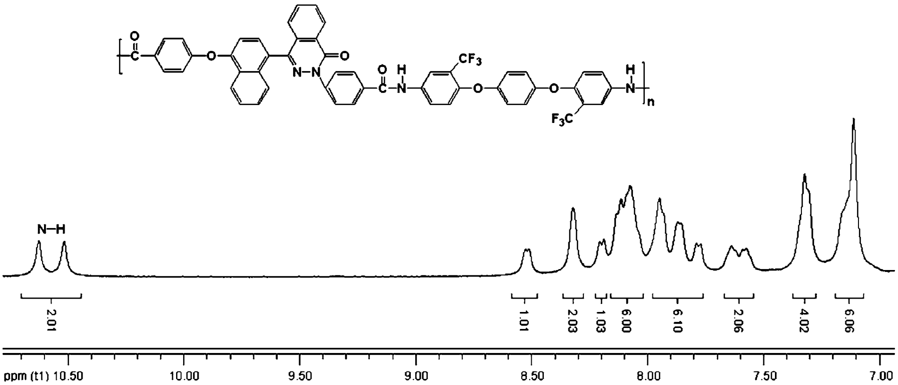


Fig. 3 ^{1}H NMR spectrum of polymer **5d** in DMSO-d_6

the highest reactivity of **4c** during the diamines. The structures of these polyamides were confirmed by IR spectroscopy. For example, polymer **5d** (Fig. 1) showed characteristic absorptions of amide group occurred around 3,310 and 1,668 cm^{-1}, peculiar to N–H stretching and carbonyl stretching, respectively. The ^{1}H NMR spectrum of **5d** was showed in Fig. 3. As it can be seen, the absorption peak at 10.6 ppm was assigned to the N–H protons. Meanwhile, no absorption was detected in the range greater than 10.6 ppm, and this indicated the completely polymerization. The total protons in the spectrum are consistent with the proposed chemical structure of polymer **5d**.

Thermal properties

The thermal behavior and glass transitions temperature (T_g) of the polymers were evaluated with TGA and DSC, respectively (Fig. 4), and the characteristic data are summarized in Table 2. The T_g value of **5a–d** were in the range of 283–338 °C. As anticipated, the T_g values of these polyamides depended on the structure of the diamine component and decreased with increasing flexibility of diamine employed,

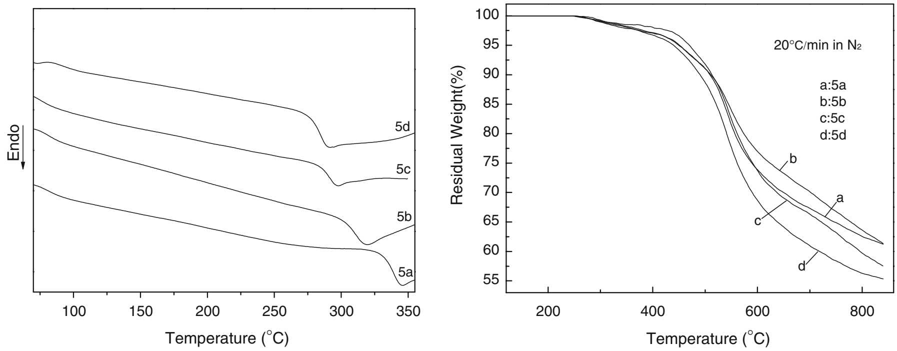


Fig. 4 DSC and TGA curves of polyamides **5a–d**

Table 2 The thermal properties of polyamides

Polymer	T_g(°C)		$T_{d5\%}$ (°C)[c]	$T_{d10\%}$ (°C)[c]	Char yield[d] (%)
	DSC[a]	DMA[b]			
5a	338	357	448	509	62
5b	311	324	451	511	63
5c	290	289	470	513	59
5d	283	280	440	490	56

[a] Measured by second scan of DSC with a heating rate of 20 °C/min in nitrogen

[b] The temperature of tan δ peak at 1 Hz by DMA with a heating rate of 5 °C/min in nitrogen

[c] Decomposition temperature at which 5 or 10% weight loss was recorded by thermogravimetry conducted at a heating rate of 20 °C/min in nitrogen

[d] Residual weight in % at 800 °C in nitrogen

the **5a** show the highest T_g up to 338 °C because of the highest rigidity of diamine used. We also measured all polyamides by DMA, the tan δ peak at 1 Hz of these polymers was used to calculate the T_g, and the result followed the same trend as the DSC. All polyamides showed high thermal stability. The 5 and 10% weight-loss temperature in nitrogen were in the range of 440–470 °C and 490–513 °C, respectively. The amount of residue of all polyamides at 800 °C in nitrogen was higher than 56%. Their excellent thermal stabilities may be due to the presence of very rigid aromatic heterocyclic backbone.

Solubility and crystallinity

The solubility behavior of the new aromatic polyamides was determined at concentration of 5% (W/V) in a number of solvents and the results were tabulated in Table 3. Almost all prepared polyamides exhibited excellent solubility in polar aprotic solvents such as NMP, DMF, DMAc, dimethyl sulfoxide (DMSO), and even in less polar solvents like Py and m-cresol, but showed poorer solubility in THF and chloroform due to the low value of dielectric constant of the solvent. Their high solubility is attributed to the presence of twist and noncoplanar aromatic heterocyclic moiety in the polymer backbone, which interrupt the regular packing of polymer chains. In addition, the bulky CF_3 side group and more flexible ether

Table 3 The solubility of polyamides

Polymer	NMP	DMAc	DMF	DMSO	THF	m-Cresol	$CHCl_3$	H_2SO_4	Py
5a	++	++	++	++	−	++	−	++	++
5b	++	++	++	++	+	++	−	++	++
5c	++	++	++	++	++	++	+	++	++
5d	++	++	++	++	++	++	+	++	++

Qualitative solubility was tested by using 50 mg of sample in 1 mL of solvent

++, Fully soluble at room temperature within 24 h; +, partially soluble; −, insoluble

Table 4 Mechanical properties of polyamides

Polymer	Tensile strength (MPa)	Elongation at break (%)	Tensile modulus (GPa)
5a	63.9	7.2	2.27
5b	75.3	9.9	1.93
5c	77.5	11.4	1.71
5d	81.6	9.0	2.08

group were introduced in the polymer backbone are especially effective for the high solubility. As expected, the WAXD studies of polyamides **5a–d** indicated that all of these polymers were essentially amorphous. It appears that the kink noncoplanar together with ether unit in the polymer backbone lead to loose chain packing.

Mechanical properties

As mentioned above, all polyamides could be readily processed into flexible, creasable films by solution casting from DMAc. The mechanical properties of polyamide films are listed in Table 4. These films showed tensile strengths of 63.9–81.6 MPa, elongations at break of 7.2–11.4%, and tensile modulus of 1.71–2.27 GPa. It was noted that the tensile strength became higher values as more ether units were introduced in the polymer chains. These results indicated that the series of polyamides are strong and tough materials, which could be applied practically.

Optical properties

Our previous study about poly(arylene ether)s containing kink noncoplanar heterocyclic structure exhibited interesting optical properties [24], therefore the absorption and the emission features of polyamides in the DMF solution were investigated. The typical UV-visible spectra and emission spectra of polyamides in DMF solution were given in Fig. 5, the UV-visible absorption showed maxima in the range of 293–312 nm, and the peak absorption showed a slight blue shift from

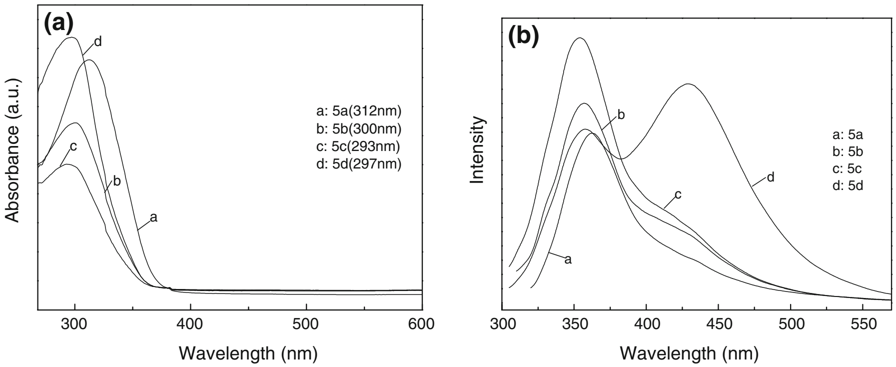


Fig. 5 UV–vis absorption spectra (**a**) and emission spectra (**b**) for polyamides **5a–d** in DMF solution

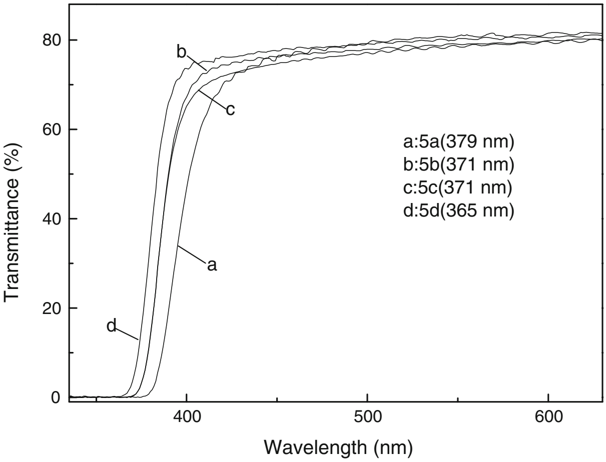


Fig. 6 UV–visible spectra of polyamide films

5a to **5c**. It's probably due to the introduction of ether unit along the chains, which could disrupt the conjugation, and subsequently increase the electronic bandgap of the polymers. The fluorescence spectra of polyamides showed emission maxima around 355 nm, whereas **5d** exhibited an additional peak at 430 nm, when excited at corresponding maximum absorption wavelength. Moreover, the optical transparency of polyamide films were also measured using UV-vis spectroscopy and the UV-visible spectra of the polyamide films are showed in Fig. 6. The cutoff wavelengths (λ_0) of all polyamides ranged in 365–379 nm. It revealed that the polymers were light colored with high transparency in visible light region, and it was noticed that the **5d** with CF_3 group revealed a shorter λ_0 than **5c**, which is accorded with the Yang's results [27, 28].

Conclusions

A novel unsymmetrical diacid from bisphenol-like 4-(4-hydroxy-naphthalenyl) phthalazin-1-one **1** was obtained in two steps. And a series of polyamides **5a–d** containing phthalazione were synthesized from prepared diacid with various aromatic diamines by the Yamazaki phosphorylation method. The results indicated that adding asymmetrical and noncoplanar heterocyclic unit into polymer backbone improved the solubility of polyamides with the retention of the thermostability. The polyamide film showed good mechanical properties and excellent transparency. The solution in DMF of all polyamides were UV-fluorescent. These properties make these polyamides attractive for practical applications such as processable high-performance polymeric materials.

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