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High glass transitions and fluorescence of novel organosoluble poly(arylene ether)s containing kink noncoplanar heterocyclic structures

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Summary

A novel unsymmetrical and kink noncoplanar heterocyclic bisphenol-like monomer, 4-(4-hydroxynaphthalenyl)phthalazin-1-one (**2**), was synthesized in a two-step reaction starting from 1-naphthol and phthalic anhydride via Friedel-Crafts acylation reaction and cyclization with hydrazine hydrate. Two heterocyclic poly(arylene ether)s were prepared conveniently from monomer **2** and two activated dihalide monomers by a N-C coupling reaction. The polycondensation proceeded quantitatively in *N,N*-dimethylacetamide in the presence of anhydrous potassium carbonate and afford the polymers with inherent viscosities of 0.46 and 0.34 dL/g. Thermal analysis showed that the polymers had excellent thermal properties. The glass transition temperatures were 277°C and 316°C respectively, and the temperatures of 5% weight loss were all above 500°C in nitrogen atmosphere. The unsymmetrical and kink noncoplanar structures of the polymers chains could disturb interchain and intrachain interactions and make them amorphous and readily soluble in polar, aprotic solvents. The dilute solution of the polymers in chloroform showed strong optical absorption in ultraviolet region, and exhibited violet-blue fluorescence with emission maxima at 400-470 nm.

Introduction

Heterocyclic polymers have attracted much attention in both academic and commercial fields due to their outstanding performance, especially very high temperature stability [1-6]. There are different kinds of these polymers which have been used in aerospace and military industries as high performance engineering plastics or matrix resins. Generally, the incorporation of aromatic heterocyclic rings to the macromolecular chains could remarkably improve the heat resistance of polymers. Based on this idea, numerous efforts have been made to synthesize novel heterocyclic polymers. Some representative heterocyclic groups that were usually employed to incorporate into polymers include imide [7], benzoxazole [8], benzimidazole [9], benzothiazole [10], 1,3,4-oxadiazole [11,12], pyridine [13,14], etc. Among them, phthalazinone-based poly(arylene ether)s families have received much interest due to their good solubility and thermal stability since the first report of a class of poly(arylene ether ketone)s and poly(arylene ether sulfone)s derived from 4-(4-hydroxylphenyl)-2,3-phthalazin-1-one(DHPZ) [15-21]. Because of the structurally unsymmetrical nature and noncoplanar conformation of the phthalazinone moiety, these polymers show good solubility in common organic solvents which is an important advantage to process and find final applications. In addition, they also exhibit very high glass transition temperatures and excellent thermooxidative stability. 4-(4-hydroxylphenyl)-2,3-phthalazin-1-one prepared from phenol and phthalic anhydride is the bisphenol-like monomer which was usually employed to react with activated dihalide monomers such as 4,4'-difluorobenzophenone or/and bis(4-chlorophenyl)sulfone to obtain high molecular weight polymers [22–24]. Side groups such as methyl and halogen groups could be introduced into this monomer to obtain phthalazinone derivatives and their corresponding polymers [25–28]. In general, such a structural modification can further improve the solubility, but also reduce the glass transition temperature or decrease the heat decomposition temperature.

In this study, a novel phthalazinone derivative, 4-(4-hydroxynaphthalenyl)phthalazin-1-one (**2**), was successfully synthesized in a two-step reaction. Meanwhile, its corresponding polymers containing kink noncoplanar rigid heterocyclic structures were prepared from monomer **2** with 4,4'-difluorobenzophenone and bis(4-chlorophenyl)sulfone through N-C coupling reactions. The 1,4-naphthyl unit has a structure similar to the 1,4-phenylene but with another benzene ring fused in the 2,3-position. The fused benzene ring can be regarded as a bulky pendent group as it is incorporated into macromolecular chains, so the chain-packing efficiency and intermolecular interactions could be reduced in this way. In the meantime, the bulky pendent group gives more restricted rotation around the ether linkage. Therefore, organosoluble poly(phthalazinone ether)s with moderate *T*g's and high thermal stability could be achieved by the incorporation of the naphthalene and phthalazinone unit in the backbones of macromolecular chains. Based on the successful synthesis of the novel bisphenol-like monomer **2**, two new polymer **3a** and **3b** were prepared. The solubility and thermal properties of these polymers were measured and investigated. Furthermore, the absorption and fluorescent properties of the polymers in dilute solution were also studied.

Experimental

Materials

All solvents were reagent-grade and desiccated with anhydrous calcium chloride. 1-naphthol (from Alfa) and hydrazine hydrate (85 wt%) were used as received. 4,4'-difluorobenzophenone (DFBP) and bis(4-chlorophenyl)sulfone (BCS) were purified from recrystallization in ethanol and dried in a vacuum oven for 24 h. All other chemicals were reagent-grade and used without further purification unless otherwise noted.

Instrument

The NMR spectra were measured on a Bruker AM 400 MHz instrument with dimethyl sulfoxide-d_6 (DMSO-d_6) or chloroform ($CDCl_3$) as a solvent and tetramethylsilane (TMS) as an internal standard. FTIR spectra were recorded on a Nicolet Magna 470 spectrometer. Ultraviolet-visible (UV-vis) spectra were obtained after measurement of the dilute chloroform solution on a Lambda 35 (Perkin Elmer) spectrophotometer. The emission spectra were measured with a Perkin Elmer LS50B luminence spectrometer in a dilute chloroform solution. Elemental analysis was carried out on a Carlo-Erba1106 system. Differential scanning calorimetry (DSC) analysis was performed on a TA 2900 M DSC instrument at a heating rate of 20°C/min in nitrogen atmosphere. Glass transition temperatures (T_g) were read at the middle of the transition in the heat capacity from the second heating scan after quick cooling from 330°C at a cooling rate of 20°C/min. Thermogravimetric analysis (TGA) of the polymer samples was measured on a DuPont 2000 SDT-2960 instrument at a heating rate of 20°C/min in nitrogen atmosphere, and T_{d5} and T_{d10} are reported as the temperatures where 5 and 10% weight losses were observed, respectively. Inherent viscosities ($\eta_{inh} = \ln \eta_r/c$) were measured at a concentration of 0.5 g/dL in chloroform at 25°C with an Ubbelohde viscometer. The solubility of the polymers was determined with 10 mg of a polymer in 1 mL of a solvent. 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°.

Monomer synthesis

*2-(1-hydroxy-1-naphthoyl)benzoic acid (**1**)*

In a 500 mL, four-necked, round-bottom flask equipped with a mechanical stirrer, a condenser, and a thermometer, 28.84 g (0.2 mol) of 1-naphthol was dissolved in 200 mL of 1,2-dichloroethane in an ice-water bath. Subsequently, 80.0 g (0.6 mol) of anhydrous $AlCl_3$ and 29.63 g (0.2 mol) of phthalic anhydride were added to the solution slowly in several portions. The resulting mixture was stirred intensively at room temperature for 4 h and at 40°C for another 4 h and then was poured into hydrochloric acid (37.5 wt %, 60 mL) containing crushed ice. The light yellow powder was obtained from steam distillation followed by filtration. This crude product could be purified by recrystallization from dilute acetic acid to afford yellow needle-like crystals (43.85 g, 75%), mp 221-222°C by differential scanning calorimetry at a scan rate of 10°C/min. IR (KBr, cm^{-1}): 3337 (s, OH), 1696 (m, COOH), 1640 (m, C=O), 1569, 1516, 1485. ^1H NMR (DMSO-d_6, ppm): δ 12.93 (s, 1H, COOH), 11.18 (s, 1H, O-H), 9.09 (d, 1H, 8'-H), 8.24 (d, 1H, 1-H), 7.92 (d, 1H, 5'-H), 7.69 (m, 2H, 3-, 7'-H), 7.62 (t, 1H, 6'-H), 7.56 (t, 1H, 2-H), 7.47 (d, 1H, 4-H), 7.29 (d, 1H, 2'-H), 6.79 (d, 1H, 3'-H). ^{13}C NMR (DMSO-d_6, ppm): 196.85, 167.23, 158.05, 143.23, 135.06, 132.60, 131.74, 130.29, 129.50, 129.31, 128.40, 127.94, 126.01, 125.25, 124.79, 124.62, 122.15, 106.33. Anal. Calcd for $C_{18}H_{12}O_4$: C, 73.97%; H, 4.14%. Found: C, 73.94%; H, 4.16%.

*4-(4-hydroxynaphthalenyl)phthalazin-1-one (**2**)*

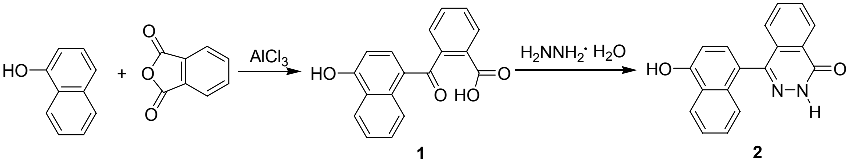
2-(1-hydroxy-1-naphthoyl)benzoic acid (29.23 g, 0.10 mol) and 1-butanol (80 mL) were added into a 250 mL, three-necked flask equipped with a dropping funnel, and the mixture was heated to reflux. Then, excessive hydrazine monohydrate (15 mL, 85 wt %) was carefully dropped into the mixture when the solution was heated to reflux. The reaction was accomplished within 3 h, and the white, powder product was obtained by filtration. The product was then purified by recrystallization for two times in DMAc and washed with anhydrous methanol, then dried under vacuum at 120°C for 24 h. About 19.6 g refined 4-(4-hydroxynaphthalenyl)phthalazin-1-one were obtained (yield 68%), mp 302-303°C by differential scanning calorimetry at a scan rate of 10°C/min. IR (KBr, cm^{-1}): 3385 (s, N-H), 3234 (s, O-H), 1640 (m, C=O), 1583, 1514, 1459, 1344. ^{1}H NMR (DMSO-d_6, ppm): δ 12.85 (s, 1H, N-H), 10.55 (s, 1H, O-H), 8.37 (d, 1H, 8-H), 8.26 (d, 1H, 5'-H), 7.84 (t, 1H, 7-H), 7.75 (t, 1H, 6-H), 7.49 (m, 1H, 6'-H), 7.41 (m, 3H, 2'-, 7'-, 8'-H), 7.15 (d, 1H, 5-H), 7.02 (d, 1H, 3'-H). ^{13}C NMR (DMSO-d_6, ppm): 159.41, 154.11, 146.03, 133.35, 133.06, 131.43, 130.74, 128.68, 127.70, 126.71, 125.77, 124.95, 124.73, 124.41, 122.82, 122.38, 107.42. Anal. Calcd for $C_{18}H_{12}O_2N_2$: C, 74.98%; H, 4.19%; N, 9.72%. Found: C, 74.70%; H, 4.16%, N, 9.89%.

Polymers synthesis

The synthesis of these poly(arylene ether)s was conducted in a similar method, and the typical procedure was described as following. A 100 ml three-necked flask equipped with a nitrogen inlet, mechanical stirrer, and Dean-Stark trap fitted with a condenser was charged with 1.4415 g (5 mmol) of **2** and 1.0915 g (5 mmol) of DFBP. The monomers were carefully washed into flask with 12 ml of DMAc. An excess of K_2CO_3 (1.04 g, 7.5 mmol) and approximate 10 ml toluene were added. The mixture was heated to 150°C for more than 5 h to ensure complete dehydration. After toluene was removed by Dean-Stark trap, the temperature was increased to 170°C and maintained at this temperature for 15 to 20 h until it became very viscous. The mixture was precipitated into 400 ml of methanol-water (1:1) solution containing 4ml acetic acid with vigorous stirring followed by filtration. The resultant white solid polymer **3a** was washed thoroughly with hot water and then dissolved with chloroform and precipitated in methanol. After filtration, it was dried at 150°C under vacuum overnight. The yields were essentially quantitative for all the polymerization (>95% yield).

Results and discussion

Monomer synthesis



Scheme 1. Synthesis of bisphenol-like monomer **2**

The phthalazinone-containing, bisphenol-like monomer **2** was synthesized from 1-naphthol and phthalic anhydride based on the method as the synthesis of monomer DHPZ according to the literature [21] (Scheme 1). The traditional synthesizing route for DHPZ is a multi-step reaction [22-24], while the method used in this paper is only a two-step reaction and easily carried out with a high yield. In the first step, anhydrous $AlCl_3$ was used as the catalyst in the Friedel–Crafts acylation reaction of 1-naphthol and phthalic anhydride to obtain 2-(1-hydroxy-1-naphthoyl)benzoic acid. The attention should be paid to obtain the desired compound **1**, that is because hydroxyl in

1-naphthol is electron-donating group, it may exist ortho or para position selectivity in this step. So it was essential to choose a low reaction temperature, and $AlCl_3$ and phthalic anhydride should also add slowly in several portions. Actually, after the fusion with hydrazine monohydrate in the second step and recrystallization for two times in DMAc, pure product **2** could be obtained. That only a single sharp peak found from the melting curve of product **2** by DSC also expressed its high purity, and suggested that the Friedel-Crafts acylation reaction of 1-naphthol and phthalic anhydride had a good selectivity in our procedure under the chosen reaction conditions mentioned above.

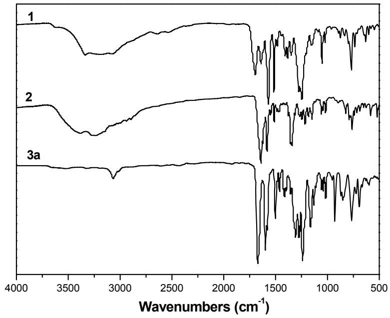


Figure 1. FTIR spectra of compound **1**, monomer **2** and polymer **3a**

The structures of intermediate compound **1** and monomer **2** were characterized by FTIR spectroscopy (Figure 1). In the FTIR spectrum of **1**, the presume peak of COOH is found at 1696 cm^{-1}, and absorption peaks of aromatic rings appear at 1569, 1516 and 1485 cm^{-1}. In the FTIR spectrum of **2**, the absorption peak at 1640 cm^{-1} is due to the stretching vibration of C=O, and the peaks at 1583, 1514 and 1459 cm^{-1} are attributed to the vibration of aromatic rings, the broad strong absorption peak at 3100-3400 cm^{-1} result from the stretching vibration of N-H and O-H. The chemical composition of compound **1** and monomer **2** were also confirmed by high-resolution NMR spectroscopy including ^{1}H NMR, ^{13}C NMR, ^{1}H-^{1}H COSY, and HMBC spectra. The ^{1}H NMR, ^{1}H-^{1}H COSY and HMBC spectra of monomer **2** are illustrated in Figures 2 and 3 respectively. The assignments of each proton are given in the figures, and these spectra are in good agreement with the proposed molecular structure of monomer **2**. Furthermore, the elemental analysis results also generally agree with the calculated values for the proposed chemical compositions.

In comparison with monomer DHPZ, the 1,4-naphthyl unit has a bigger space volume than 1,4-phenyl unit, so the molecular conformation of monomer **2** might be altered. To attain the minimum energy of conformation, it is no doubtful that there will be a bigger dihedral angle between the phthalazinone and naphthyl rings. As shown in Figure 4, the molecular model of the adjacent, monomer **2**, simulated by semi-empirical computations of Cambridge Software, exhibited remarkable twisting and noncoplanar between the phthalazinone and naphthalene rings. The dihedral angle was 76.2°, which is bigger than that of DHPZ (61.3°) [29]. In fact, it is fairly probable that the alternation of the properties of this class of polymers, such as the solubility and thermal properties, might originate from this type of basic variation of the inner molecular conformation.

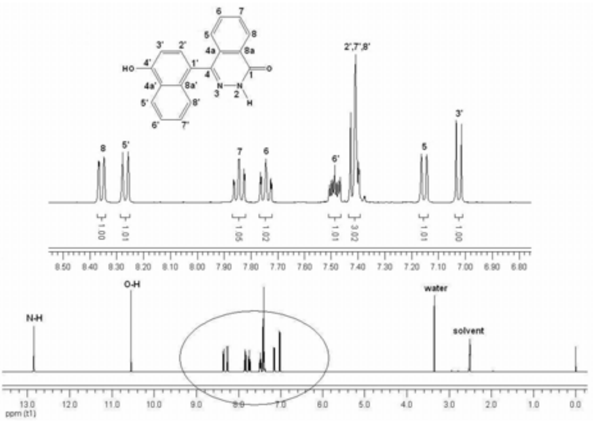


Figure 2. ^{1}H NMR spectra of monomer **2** in DMSO-d_6

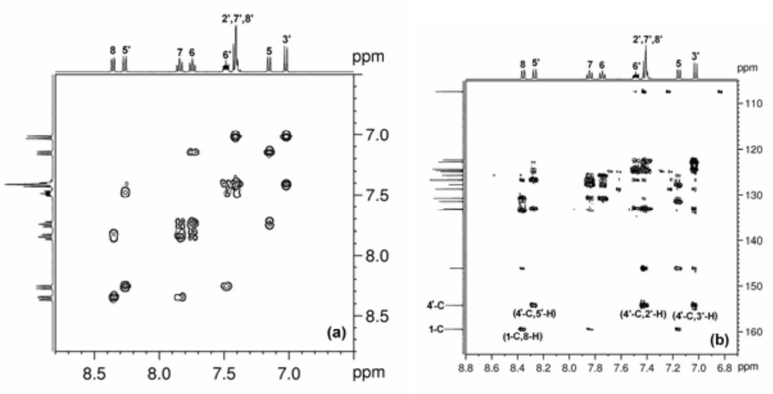


Figure 3. ^{1}H-^{1}H COSY (**a**) and HMBC (**b**) spectra of monomer **2** in DMSO-d_6

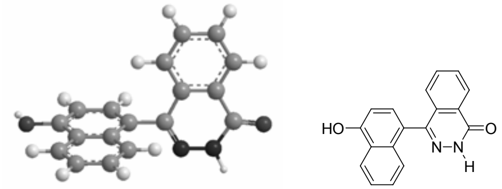
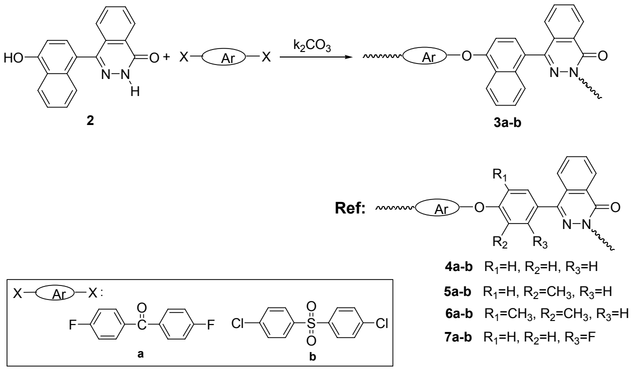


Figure 4. Molecular model of monomer **2**

Polymer synthesis

As depicted in Scheme 2, two new kinds of poly(phthalazinone ether)s, including poly(arylene ketone) (**3a)** and poly(arylene sulfone) (**3b),** were prepared by the reaction of phthalazinone containing bisphenol-like monomers **2** and two activated dihalide in a dipolar aprotic medium of DMAc. During the polymerization, the phthalazinone N-H group behaves like phenolic O-H groups, thereby the polymers could be formed from a novel N-C coupling reaction through step growth polycondensation. However, the 1,4-naphthyl unit in monomer **2** has a bigger volume compared with 1,4-phenyl unit in monomer DHPZ and there is a steric congestion for O-H group during the polycondensation. Actually, after a relative long reactive time (about 15 to 20 hours), strip-like or granule-like polymers could also be easily prepared with quantitative yields, and the inherent viscosities of the polymers were 0.46 and 0.34, respectively.



Scheme 2. Synthesis of polymer **3a** and **3b**

The formation of the polymers was confirmed by FTIR and ^{1}H NMR spectra. The FTIR spectrum of polymer **3a** clearly exhibits the characteristic absorption at about 1672 (C=O), 1601, 1580, 1501 (aromatic rings), 1325, 1310 (C-N) and 1240 cm^{-1} (C-O-C), respectively (Figure 1). There was no absorption in the range of 3100–3400 cm^{-1} which was the characteristic absorption for N-H and O-H suggesting all monomer **2** taken part in the polymerization. ^{1}H NMR spectrum of polymer **3a** was shown in Figure 5. Two sharp peaks at 12.85 and 10.55 ppm corresponding to phthalazinone N-H and phenolic O-H protons in Figure 3 were absent here, and the integral values of all protons given in the spectrum completely accorded with the proposed molecular structure of polymer **3a**. These results demonstrate that the polymers possess high relative molecular weight and the bisphenol-like monomer **2** also has a good polymerization activity for forming poly(aryl ether)s despite the presence of a steric congestion for O-H group.

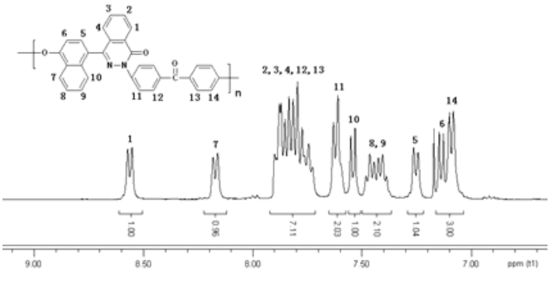


Figure 5. ^{1}H NMR spectra of polymer **3a** in $CDCl_3$

Solubility and crystallinity

The solubility of the polymer **3a** and **3b** in some common solvents was examined and summarized in Table 1. The analogous poly(arylene ether)s **5a-7a** containing methyl or fluoro-substituted groups reported [25,26,28] are also given in Table 1 as comparison. Because of the twist and noncoplanar molecular structures and large free volume caused by bulky phthalazinone and naphthalene configuration in the polymers (**3a** and **3b**), they exhibited better excellent solubility in some polar aprotic solvents compared with the analogous polymer **5a-7a.** This could be attributed to the presence of larger free volume in polymer **3a** and **3b** than polymer **5a-7a** due to present 1,4-naphthyl unit, so that the polar solvent molecules could more easily diffuse into the macromolecular chains of polymer **3a** and **3b**.

Table 1. The solubility of of polymers[a]

polymer	NMP	DMAc	DMF	DMSO	Py	$CHCl_3$	THF	Acetone
3a	++	++	++	+	+	++	+	–
3b	++	++	++	+	+	++	+	–
5a[b]	++	+	–	–	+	++	–	
6a	++	+	–	–	+	++	–	
7a	++	+	+	+–	+	++	+–	

[a] ++ soluble; + soluble when heated; +– partially soluble; – insoluble; NMP= *N*-methyl-2-pyrrolidone, DMAc= *N,N*-dimethylacetamide, DMF= *N,N*-dimethylformamide, DMSO= Dimethyl sulfoxide, Py=Pyridine

[b] Polymers **5a-7a** results cited from Ref. 25, 26, 28

The crystallinity of the polymers was evaluated by wide-angle X-ray diffraction. They all exhibited completely amorphous. This is also attributed to the twist and non-coplanar molecular structures which could interrupt the regular packing of chains. Generally, these polymers with similar structure containing phthalazinone are amorphous [22,25].

Thermal properties

TGA and DSC were used to investigate the thermal properties of the polymer **3a** and **3b** as shown in Figure 6, and the characteristic data are summarized in Table 2. The analogous poly(arylene ether)s **4a-7a** and **4b-7b** based on phthalazinone derivatives

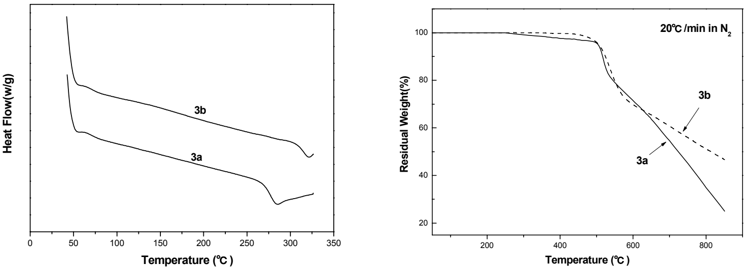


Figure 6. DSC and TGA curves of polymer **3a** and **3b**

monomers reported [24-26,28] are also given in Table 2 as comparison. The T_g values of **3a** and **3b** are 277 and 316°C respectively. Because the sulfone group in polymer **3b** is more rigid than carbonyl in polymer **3a, 3b** exhibits a relative higher T_g than **3a**. The TGA results indicate that both **3a** and **3b** are stable up to around 500°C in nitrogen. The 5% decomposition temperatures (T_{d5}) of these two polymers are 504 and 507°C, which indicate they could meet high temperature resistant requirements as some special materials in modern aerospace, military and microelectronics industries. As anticipated, either polymer **3a** or **3b** shows a improved thermal properties compared with the analogous polymers **4a-7a** or **4b-7b**. This might be attributed to the large rigid naphthyl rings in the backbone of **3a** and **3b.**

Table 2. The thermal properties of polymers

polymer	T_g^a (°C)	T_{d5}^b (°C)	T_{d10}^b (°C)	Residue^c (wt %)
3a	277	504	517	35
4a^d	253	490	–^{e}	–^{e}
5a	252	420	–^{e}	–^{e}
6a	295	416	–^{e}	–^{e}
7a	253	534	–^{e}	–^{e}
3b	316	507	525	51
4b	295	505	–^{e}	–^{e}
5b	290	420	–^{e}	–^{e}
6b	320	419	–^{e}	–^{e}
7b	283	505	–^{e}	–^{e}

[a] Measured by second scan of DSC with a heating rate of 20°C/min from room temperature to 330°C in nitrogen

[b] 5 or 10% weight-loss temperature measured by TGA with a heating rate of 20°C/min in nitrogen

[c] Weight residue measured by TGA with a heating rate of 20°C/min at 800°C in nitrogen

[d] Polymers **4a-7a** and **4b-7b** results cited from Ref. 24-26, 28.

[e] Not available

Optical properties of polymers in solution

The polymers **3a** and **3b** can be easily prepared into thin films by solution casting. All the films and dilute solution of the polymers are transparent in visible spectra region, and have a strong UV absorption. The UV-vis absorption curves of polymer **3a** and **3b** in the dilute chloroform solution are shown in Figure 7 (a). These two polymers exhibited a similar absorption behavior, and the absorption peak appeared at about 250 and 305 nm. Surprisingly, upon irradiating with a UV lamp, the chloroform solution of **3a** and **3b** were violet-blue fluorescence, and this luminescence phenomenon have not been reported in the analogous poly(arylene ther)s **4a-7a** and **4b-7b**. It might be attributed the fluorescent groups, phthalazinone and naphthyl unit. While excited at 305 nm, the polymer solution of **3a** and **3b** displayed maxima emission at about 440 and 410 nm, and the emission spectra were illustrated in Figure 7 (b). It is obvious that the photoluminescence of polymer **3a** or **3b** has a red shift about 50~100nm compared with that of compound 1-naphthol or naphthalene. This shift is probably due to the phthalazinone and naphthyl unit in the macromolecular chain of polymer **3a** or **3b** having a large conjugation, which makes the energy band gap for π→π* smaller and the photoluminescence of corresponding polymers in violet-blue region.

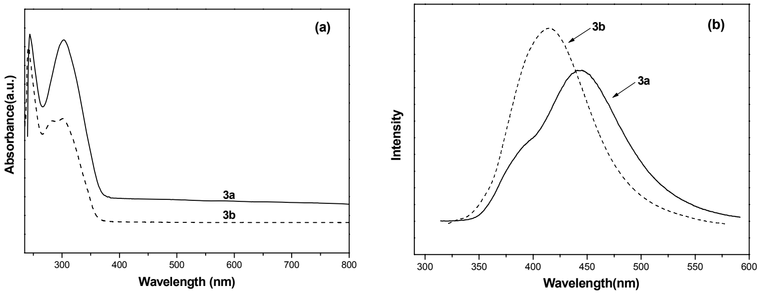


Figure 7. UV-vis absorption spectra (a) and emission spectra (b) for polymer **3a** and **3b** in chloroform solution

Conclusions

A new bisphenol-like monomer, 4-(4-hydroxynaphthalenyl)phthalazin-1-one, was successfully synthesized in a two-step procedure. Two phthalazinone-containing poly(arylene ether)s were prepared from the bisphenol-like monomer **2** via the nucleophilic aromatic substitution polycondensation with two dihalide compounds. The resulting two polymers were completely amorphous and exhibited excellent solubility in various organic solvents. These two polymers were found to have high glass transition temperatures and high thermal stability. They also showed strong optical absorption in the UV-region, and exhibited photoluminescence in dilute solution. These polymers may be considered as good potential candidates for high-temperature resistant materials and fluorescent materials.

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