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BLUE LIGHT EMITTING COPOLY(ARYLENE ETHER)S CONTAINING A 3,6-DIALKOXYPHTHALIMIDE GROUP

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ABSTRACT

Four new bisphenols containing 3,6-dialkoxyphthalimide moieties were prepared in high yields. They were copolymerized with BPA or 2,5-bis(4-hydroxyphenyl)oxadiazole by reaction with decafluorobiphenyl and with NaH as base to give novel copoly(arylene ether)s. The resulting copolymers have glass-transition temperatures (Tg) ranging from 153 to 235°C and very good thermal stabilities with 5% wt loss temperatures (TGA) ranging from 378 to 499°C. These polymers show strong blue light emission in wavelengths ranging from 420 to 430 nm, while their UV absorption peaks vary from 368 to 383 nm. They have excellent solubility in common organic solvents.

Key Words: 3,6-Dialkoxyphthalimide; Copoly(arylene ether); Fluorescence

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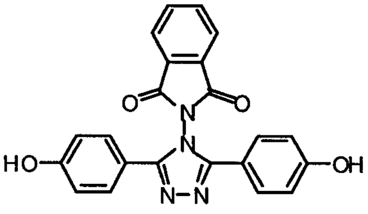
INTRODUCTION

Since the discovery of electroluminescent (EL) light-emitting devices (LEDs),[1] many polymers with EL properties have also been synthesized in the past few years.[2] Polymers possess several advantages over inorganic materials, such as low cost, easy processability, flexibility[3] and ease of coating over large areas.[4] Polymer LEDs therefore exhibit considerable promise in display applications.

Poly(arylene ether)s are recognized as an important class of high-performance engineering thermoplastics with very good physical, chemical, and mechanical properties.[5] The excellent thermo-oxidative stability, high glass-transition temperatures (Tg), and good mechanical strength make modified poly(arylene ether)s potentially useful in organic electroluminescent (EL) devices.[6,7]

Polymer light emitting diodes (PLEDs) require not only good physical properties, but also efficient luminescent properties in devices. The luminescent wavelength of OLEDs can be changed by molecular design. Efficient highly bright and blue light emitting materials are still under intensive research because blue light is necessary for full color EL display applications. In recent years, both low molecular weight oxidiazole derivatives and oxadiazole containing polymers, have shown improved quantum efficiency and also act as the electron transport layer.[3,8–10]

In this paper we describe the synthesis of strong blue light-emitting poly(arylene ether)s **9**–**12** that contain the 3,6-dialkoxyphthalimide moiety. Although phthalimides are not fluorescent it has been shown that 3,6-dimethoxyphthalimide and derivatives are very fluorescent with fluorescence maxima around 450 nm and quantum yields around 0.5.[11] Four novel bisphenols **5a**, **5b**, **5c** and **8** were prepared in high yields from 2,3-dicyanohydroquinone.[12,13] We have recently synthesized the monomer 2-[3,5-bis-(4-hydroxy-phenyl)-[1,2,4]triazol-4-yl]-isoindole-1,3-dione **A**[14] and prepared poly(aryl ether)s by reaction with difluoro-aromatic compounds such as bis(4-fluorophenyl)sulfone at 170°C.



A

The new imide-containing bisphenols **5a**, **5b**, **5c** and **8** were not stable under conventional polycondensation conditions,[15–17] and therefore cannot be polymerized with dihalogeno-aromatic compounds such as bis(4-fluorophenyl)sulfone. We successfully copolymerized these new monomers with BPA (bisphenol A) or 2,5-bis(4-hydroxyphenyl)oxadiazole by reaction with decafluorobiphenyl using sodium hydride as a base at 0°C. The resulting polymers **9–12** were high molecular weight and exhibited excellent solubility in common organic solvents. They also had good thermal stability with 5% wt loss temperatures ranging from 378 to 499°C under nitrogen, as determined by thermogravimetric (TGA) analysis. The polymers showed UV absorption maxima at 368–383 nm and strong emission maxima at 420–430 nm.

EXPERIMENTAL

Materials

The commercial reagents 2,3-dicyanohydroquinone, potassium carbonate, dimethyl sulfate, iodoethane, 1-iodopropane, zinc acetate, hydrazine and sodium hydride, and decafluorobiphenyl were purchased from Aldrich Chemical Co. and used as received. Pure BPA was supplied by the General Electric Company. Compounds **4**, **6** and **7** were prepared as reported in the literature.[18,19] The solvents used for the monomer synthesis and polymerization were distilled before use.

Measurements

^{1}H-NMR spectra were recorded on a Varian Mercury 300 or 400 spectrometer. DMSO-d_6 or $CDCl_3$ was used as the solvent, and tetramethylsilane was used as the internal standard. Mass spectra were measured on a Kratos MS25RFA instrument at the ionization energy of 70 eV. Melting points were taken on a Fisher-Johns melting point apparatus. Molecular weights of copolymers were determined relative to polystyrene standards by gel permeation chromatography (GPC) in THF as the eluent on a Waters 510 system equipped with a UV detector set at 254 nm. The inherent viscosity of the copolymers was measured in chloroform ($CHCl_3$) solution at a concentration of 0.5 g/dL at 25°C using an Ubbelohde viscometer. DSC measurements were carried out on a Seiko DSC220 thermal analyzer at a heating rate of 20°C min^{-1} under nitrogen at a flow rate of 150 mL min^{-1}. Thermogravimetric (TG) and differential thermal analyses (DTA) were performed simultaneously on a Seiko TG/DTA220 at a heating rate of 20°C min^{-1} under nitrogen at a flow rate of 200 mL min^{-1}. UV-vis absorption spectra were recorded on a CARY 50 spectrophotometer. Photoluminescence (PL) measurements were performed on a SPEX FL-3 fluorescence spectrometer.

Synthesis of Monomers

Synthesis of 3,6-Dialkoxyphthalonitrile **1**

General Procedure: A 500 mL three-necked flask was charged with 10 g (62.5 mmol) of 2,3-dicyanohydroquinone, 22 g of potassium carbonate, 150 mL of DMAc and 60 mL of toluene. The mixture was heated to 145°C for 2 h to dehydrate the reaction mixture. After cooling to 60°C, a mixture of 250 mmol of dimethylsulfate (or iodoethane or 1-iodopropane) in 50 mL DMAc was added dropwise. The resulting mixture was stirring for 16 h. At room temperature, 100 mL of water and 100 mL of methanol was added with stirring. After filtering, washing with water and methanol, and then drying, a light brown solid was obtained.

3,6-Dimethoxyphthalonitrile **1a**

Yield: 98%. m.p. 275°C. ^{1}H-NMR (300 MHz, DMSO-d_6): 3.95 (s, 6H), 7.60 (s, 2H). MS (m/z, %): 188 (M^+, 100).

3,6-Diethoxyphthalonitrile **1b**

Yield: 98.7%. m.p. 214°C. ^{1}H-NMR (300 MHz, DMSO-d_6): 1.31 (t, $J = 7.20$ Hz, 6H), 4.18 (q, $J = 6.90$ Hz, 4H), 7.57 (s, 2H). MS (m/z, %): 216 (M^+, 5.4).

3,6-Dipropoxyphthalonitrile **1c**

Yield: 99%. m.p. 198°C. ^{1}H-NMR (300 MHz, DMSO-d_6): 0.97 (t, $J = 7.60$ Hz, 6H), 1.73 (m, 4H), 4.09 (t, $J = 6.40$ Hz, 4H), 7.58 (s, 2H). MS (m/z, %): 244 (M^+, 34).

Synthesis of 3,6-Dialkoxyphthalic Acids **2**

General Procedure: To a suspension of 1.08 g (5 mmol) of compound **1** and 50 mL of ethanol was added aq. sodium hydroxide (4 g in 10 mL H_2O) and the reaction mixture was refluxed for 50 h. After removing the ethanol, the mixture was diluted with 150 mL of water, neutralized with conc. HCl and then treated with saturated $NaHCO_3$. After filtering, the filtrate was neutralized with conc. HCl and extracted with ethyl acetate. Removal of the solvent from ethyl acetate extract gave a yellowish solid.

3,6-Dimethoxyphthalic Acid **2a**

Yield: 99%. MS (m/z, %): 226 (M^+, 38). ^{1}H-NMR (300 MHz, DMSO-d_6): 3.72 (s, 6H), 7.10 (s, 2H).

3,6-Diethoxyphthalic Acid **2b**

Yield: 99%. m.p. 169°C. ^{1}H-NMR (DMSO): 1.24 (t, J = 7.2 Hz, 6H), 3.98 (q, J = 6.9 Hz, 4H), 7.07 (s, 2H), 12.9 (s, 2H). MS (m/z, %): 254 (M^+, 26).

3,6-Dipropoxyphthalic Acid **2c**

Yield: 94%. m.p. 145°C. ^{1}H-NMR (300 MHz, DMSO-d_6): 0.95 (t, J = 7.60 Hz, 6H), 1.72 (m, 4H), 3.87 (t, J = 6.4 Hz, 4H), 7.06 (s, 2H), 12.9 (s, 2H). MS (m/z, %): 282 (M^+, 68).

Synthesis of 3,6-Dialkoxyphthalic Anhydride **3**

General Procedure: To a 50 mL flask there was added 0.7 g (3 mmol) of **2** and 10 mL of Ac_2O. The resulting mixture was refluxed for 3 h. After cooling down, yellowish crystals precipitated out. After filtering and drying, yellowish crystals were obtained.

3,6-Dimethoxyphthalic Anhydride **3a**

Yield: 89%. m.p. 260°C. MS (m/z, %): 208 (M^+, 100). ^{1}H-NMR (300 MHz, DMSO-d_6): 3.97 (s, 6H), 7.60 (s, 2H).

3,6-Diethoxyphthalic Anhydride **3b**

Yield: 94.2%. m.p. 201°C. MS (m/z, %): 237 ($M^+ + 1$, 15). ^{1}H-NMR (300 MHz, DMSO-d_6): 1.34 (t, J = 7.20 Hz, 6H), 4.20 (q, J = 6.80 Hz, 4H), 7.55 (s, 2H).

3,6-Dipropoxyphthalic Anhydride **3c**

Yield: 88%. m.p. 227°C. MS (m/z, %): 265 ($M^+ + 1$, 12). ^{1}H-NMR (300 MHz, DMSO-d_6): 0.98 (t, J = 7.60 Hz, 6H), 1.73 (m, 4H), 4.10 (t, J = 6.80 Hz, 4H), 7.55 (s, 2H).

Synthesis of 2-[3,5-bis(4-Hydroxy-phenyl)-[1,2,4]Triazol-4-yl-4,7-dialkoxy-isoindole-1,3-dione 5

General Procedure: A 50 mL three-necked flask was charged with 230 mg of **3** (1.1 mmol), 310 mg (1.15 mmol) of 4-amino-3, 5-bis(4-hydroxyphenyl)-1,2,4-triazole, 30 mg of $Zn(OAc)_2 2H_2O$ and 6 mL of NMP. The resulting mixture was heated to 180°C under nitrogen for 20 h. After cooling down, the mixture was poured into 30 mL of water and a solid was precipitated out. After filtering and drying, a greenish solid was obtained.

2-[3,5-bis(4-Hydroxy-phenyl)-[1,2,4]Triazol-4-yl-4,7-dimethoxy-isoindole-1,3-dione **5a**

Yield: 80%. Td: 309°C. MS (m/z, %): 458 (M^+, 100). ^{1}H-NMR ($CHCl_3$, TMS): 3.95 (s, 6H), 6.80 (d, J = 8.50 Hz, 4H), 7.30 (d, J = 8.50 Hz, 4H), 7.62 (s, 2H), 10.10 (s, 2H).

2-[3,5-bis(4-Hydroxy-phenyl)-[1,2,4]Triazol-4-yl-4,7-diethoxy-isoindole-1,3-dione **5b**

Yield: 95.7%. m.p. 259°C. MS (m/z, %): 487 ($M^+ + 1$, 21). ^{1}H-NMR (300 MHz, DMSO-d_6): 1.31 (t, J = 7.00 Hz, 6H), 4.18 (q, J = 6.80 Hz, 4H), 6.83 (d, J = 8.70 Hz, 4H), 7.32 (d, J = 8.70 Hz, 4H), 7.58 (s, 2H), 10.09 (s, 2H).

2-[3,5-bis-(4-Hydroxy-phenyl)-[1,2,4]Triazol-4-yl-4,7-dipropoxy-isoindole-1,3-dione **5c**

Yield: 91.5%. m.p. 241.8°C. MS (m/z, %): 515 ($M^+ + 1$, 56). ^{1}H-NMR (300 MHz, DMSO-d_6): 0.93 (t, J = 7.60 Hz, 6H), 1.70 (m, 4H), 4.08 (t, J = 6.80 Hz, 4H), 6.82 (d, J = 8.00 Hz, 4H), 7.33 (d, J = 8.00 Hz, 4H), 7.59 (s, 2H), 10.10 (s, 2H).

Synthesis of **8**

The synthetic procedure for compound **8** was the same as that of compound **5**. Yield: 87%. m.p. 374°C. MS (m/z, %): 587 ($M^+ + 1$, 0.88). ^{1}H-NMR (DMSO): 3.95 (s, 6H), 6.95 (d, J = 8.5 Hz, 4H), 7.25 (d, J = 8.5 Hz, 4H), 7.60 (s, 2H), 7.78 (m, 2H), 7.82 (m, 2H), 9.78 (s, 2H).

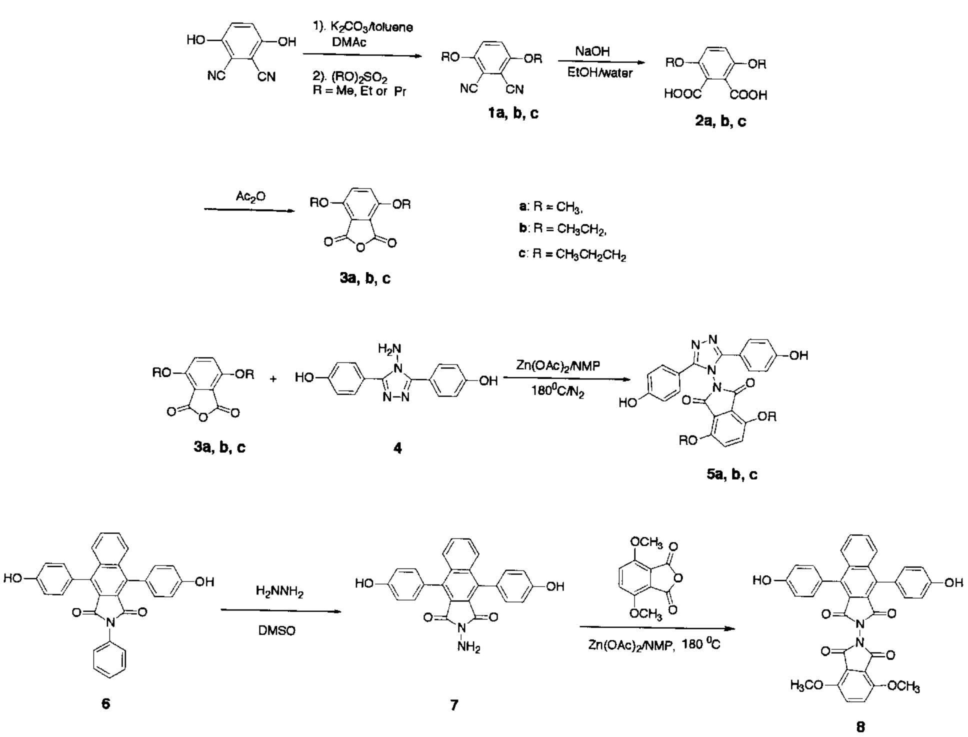
Synthesis of Copolymers

A typical procedure follows. To a solution of **5c** (0.4 mmol), BPA (0.6 mmol) and decafluorobiphenyl (1 mmol) in 2 mL of DMAc was added sodium hydride (3 mmol) at 0°C under nitrogen. The resulting mixture was stirred at 0°C for 1 h, and then allowed to warm to 25°C. over 0.5 h. At room temperature, the mixture was stirred for 30 min. The viscous solution was diluted with additional DMAc (3 mL). The mixture was precipitated into 100 mL of methanol containing a few drops of hydrochloric acid, filtered, and dried in vacuum. The resulting copolymer was dissolved in chloroform (10 mL), filtered through Celite, and reprecipitated in methanol (100 mL). The copolymer was dried at 40°C under vacuum for 24 h to give 548 mg of light yellow fiber, copolymer **9c** (n = 0.4; yield: 86%). ^{1}H-NMR ($CHCl_3$, TMS): 1.08 (t, J = 7.60 Hz, 6H), 1.68 (s, 9H), 1.84 (m, 4H), 4.06 (t, J = 6.80 Hz, 4H), 6.95 (d, J = 8.00 Hz, 6H), 7.08 (d, J = 8.10 Hz, 4H), 7.21 (d, J = 8.00 Hz, 6H), 7.71 (d, J = 8.10 Hz, 4H), 7.95 (s, 2H).

RESULTS AND DISCUSSION

Synthesis of Monomers

The synthetic route to new monomers **5a**, **5b**, **5c** and **8** is shown in Sch. 1. 3,6-Dimethoxyphthalic anhydride **3a** was synthesized from 2,3-dicyanohydroquinone in three steps. In the literature,[19] compound **3a** was prepared from compound **1a**, by treatment with H_2SO_4. We found that under these conditions the reaction was difficult to control and side reactions could not be avoided. We modified the reaction and synthesized compound **3a** in two steps. Hydrolysis of **1a** with sodium hydroxide as a base in a mixture of ethanol and water yielded 2,3-dimethoxyphthalic acid **2a**, and then

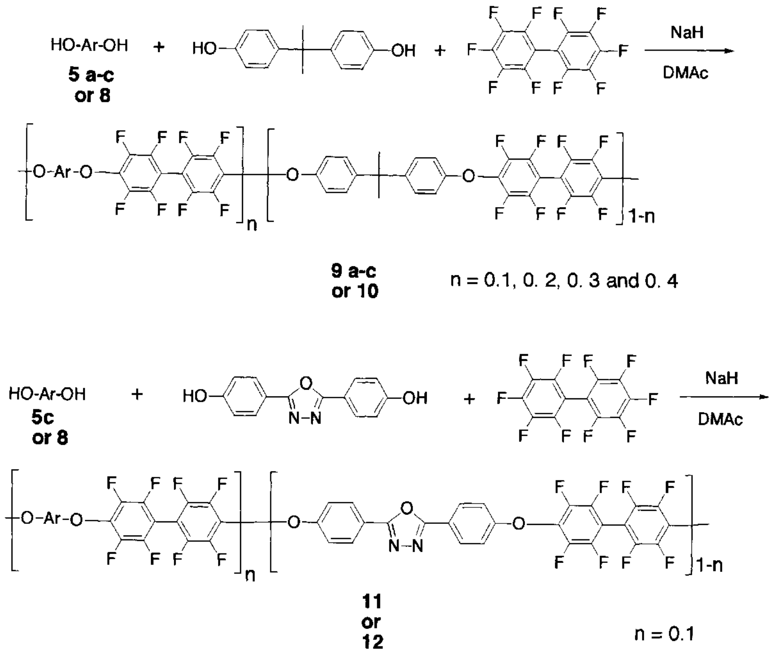


Scheme 1. Synthesis of compounds **5a**, **5b**, **5c**, and **8**.

cyclization of **2a** in acetic anhydride gave the target compound **3a**, in a total yield of 88%. Compounds **3b** and compound **3c** were similarly synthesized. Reactions of **3** with 4-amino-3, 5-bis(4-hydroxyphenyl)-1,2,4-triazole **4** in the presence of $Zn(OAc)_2$ catalyst gave compounds **5** (**a**, **b**, and **c**). Monomer **8** was prepared by the reaction of **3** and **7** in 87% yield.

Synthesis of Copolymers

Bisphenols **5a**, **5b**, **5c** and **8** were not stable at the elevated temperatures generally used for the synthesis of poly(aryl ether)s because of hydrolysis of the imide group. Copolymers with other bisphenols were successfully prepared from the very active decafluorobiphenyl at 0°C using sodium hydride as a base. Gels were formed when high percentages (more than 40%) of the new monomers were introduced into the copolymers. (Sch. 2) The polymers **9a**, **9b**, **9c** and **10** were highly fluorescent with excellent solubility in organic solvents. The 1,3,4-oxadiazole electron transporting moiety was introduced into the polymers **11** and **12** polymer.



Scheme 2. Synthesis of copolymers **9**–**12**.

Properties of Copoly(Arylene Ether)s 9–12

The molecular weights, thermal analyses and viscosities of the resulting polymers are listed in Table 1. Polymers **9**–**12** have high molecular weights with number-average molecular weights ranging from 16, 900 to 66, 900 and with very narrow molecular weight distribution (Mw/Mn). Inherent viscosities in chloroform varied from 0.18 to 0.45. Tg's of **9a** ($R=CH_3$) and **9b** ($R=CH_2CH_3$) tended to increase when the percentages of the new monomers increased, while Tg's of **9c** ($R=CH_2CH_2CH_3$) decreased slightly. The TGA data indicated that polymers **9**–**12** are thermally stable. The 5% wt loss temperatures under nitrogen ranged from 378 to 500°C. For the same series of polymers (**9a**, **9b** and **9c**), the wt losses decrease with increasing percentages of the new moieties.

The UV absorption and photoluminescent (PL) spectra of monomers **5a**–**c** and **8** and polymers **9**–**12** in dilute THF solution (80 µg/mL) were measured. The relevant maxima are listed in Table 2. The absorption spectra of polymers **9**–**12** are similar in shape with maxima around 368–383 nm. All the polymers showed strong fluorescence under UV irradiation with maxima around 420–430 nm.

Table 1. Properties of Copoly(Arylene Ether)s **9**–**12**

Entry	R	n	Yield (%)	Mn[a] (× 10^3)	Mw[a] (C10^3)	MW D	Tg (°C)	TGA[b] (°C)	η_{inh}[c] (dL/g)
9a-1	CH_3	0.1	88	35.5	56.0	1.57	162	496	0.27
9a-2	CH_3	0.2	86	58.7	89.7	1.53	177	483	0.43
9a-3	CH_3	0.3	85	49.9	81.1	1.63	182	441	0.33
9a-4	CH_3	0.4	85	21.2	32.8	1.55	183	432	0.22
9b-1	C_2H_5	0.1	89	39.7	58.2	1.47	154	480	0.30
9b-2	C_2H_5	0.2	85	38.1	58.8	1.54	166	426	0.29
9b-3	C_2H_5	0.3	84	20.5	32.0	1.56	170	389	0.21
9b-4	C_2H_5	0.4	84	22.3	32.8	1.47	165	378	0.23
9c-1	n-C_3H_7	0.1	88	66.9	96.3	1.44	174	496	0.45
9c-2	n-C_3H_7	0.2	87	54.5	88.1	1.62	172	419	0.41
9c-3	n-C_3H_7	0.3	86	34.9	57.7	1.65	171	395	0.26
9c-4	n-C_3H_7	0.4	86	16.9	26.9	1.59	171	379	0.18
10	CH_3	0.1	91	52.0	85.9	1.65	180	500	0.37
11	n-C_3H_7	0.1	87	35.0	53.0	1.51	210	462	0.28
12	CH_3	0.1	87	56.9	86.9	1.53	236	454	0.42

[a] Molecular weights determined by GPC in THF with polystyrene standards.

[b] Five-percent wt loss temperature under nitrogen.

[c] Inherent viscosities measured in $CHCl_3$ (0.5 g/dL) at 25.0 ± 0.1°C with an Ubbelohde viscometer.

Table 2. The Absorption and Emission Maxima of Polymers **9**–**12** and Monomers (**5a**–**c** and **8**)

Entry	n	λ^{d}_{max} (nm)	λ^{e}_{em} (nm)
9a-1	0.1	375	427
9a-2	0.2	374	428
9a-3	0.3	376	429
9a-4	0.4	378	429
9b-1	0.1	380	428
9b-2	0.2	378	430
9b-3	0.3	378	429
9b-4	0.4	381	429
9c-1	0.1	380	426
9c-2	0.2	381	428
9c-3	0.3	381	428
9c-4	0.4	383	430
10	0.1	368	420
11	0.1	368	428
12	0.1	368	421
5a	–	383	426
5b	–	382	427
5c	–	382	427
8	–	370	440

[d] Maximum UV absorption wavelength in THF solution.

[e] Maximum emission wavelength excited at the absorption maximum in THF solution.

Even though the monomers have strong UV absorption, they have very weak fluorescence emission. The resulting polymers containing these new monomers show very strong emission. The intensities of emission of the polymers depend on the percentages of new monomers. Polymer **9c** is taken as an example. Figures 1 and 2 show the UV-Vis absorption and emission spectra of monomer **5** and polymer **9c**, respectively. We can see that polymer **9c** (n = 0.4) that contains the highest percentage of the new moiety, shows the strongest absorption and emission, while polymer **9c**, as expected, (n = 0.1) exhibited the weakest.

These copolymers exhibited excellent solubility in common organic solvents, which makes them potentially very useful in organic electroluminent devices. Polymers **9a**, **9b**, **9c**, and **10** that were copolymerized with BPA were soluble at room temperature in common organic solvents, such as toluene, acetone, THF, $CHCl_3$, DMF, DMAc, DMSO. Polymers **11** and **12**, were copolymerized with 2,5-bis(4-hydroxyphenyl)oxadiazole were soluble in acetone and toluene upon heating, and readily soluble in THF, $CHCl_3$, DMF, DMAc and DMSO.

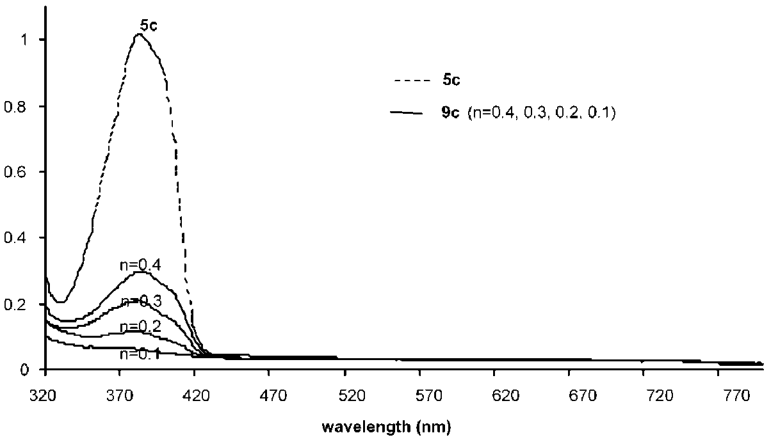


Figure 1. UV/Vis absorption spectra of monomer **5c** and polymer **9c** (n = 0.1–0.4).

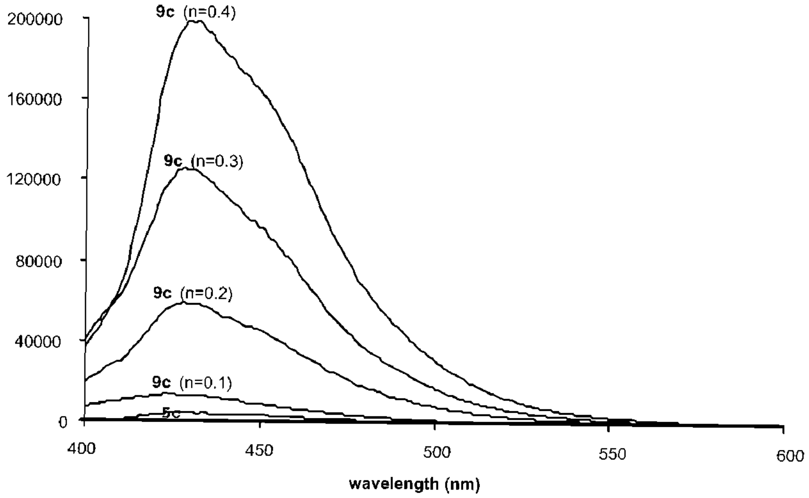


Figure 2. The emission spectra of monomer **5c** and polymer **9c** (n = 0.1–0.4).

CONCLUSION

Four new bisphenols, **5a–c** and **8**, containing 3,6-dialkoxyphthalimide moieties were successfully synthesized in high yield from 2,3-dicyanohydroquinone. The new bisphenols were not stable at temperatures used in conventional poly(aryl ether) polymerization reactions. They were successfully copolymerized with BPA or 2,5-bis(4-hydroxyphenyl)oxadiazole and decafluorobiphenyl using NaH as a base at 0°C. The resulting copolymers had high molecular weights and narrow molecular weight distributions. The polymers are amorphous and have excellent solubility in organic solvents, such as toluene, acetone, DMF, THF, DMSO, etc. The copolymers have glass transition temperatures between 153 and 235°C as measured by DSC. The 5% wt loss temperatures ranged from 378 to 499°C. They emit strong blue light at 420–430 nm in solution.

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