

Synthesis and characterization of new poly(aryl ether)s containing alternate emitting and electron transporting chromophores

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Abstract

Three poly(aryl ether)s consisting of alternate isolated blue chromophore (substituted *p*-aryl vinylene) and electron transport structure (1,3,4-oxadiazole) have been synthesized and characterized. The poly(aryl ether)s ($\eta_{\text{red}} > 0.5$ dl/g) were obtained by nucleophilic displacement reaction of oxadiazole-activated bis(fluoride) monomers with bis(phenol) monomers. Introduction of ether linkages results in enhanced solubility in organic solvent such as *N,N*-dimethylacetamide (DMAc), *N*-methyl-2-pyrrolidone (NMP) and Pyridine. The polymers have the high glass transition temperature ($T_g = 181$ – 223°C) and exhibit good thermal stability with the onset decomposition temperature at around 470°C in nitrogen. The photoluminescence spectra shift to longer wavelength with additional phenyl ring in aryl vinylene chromophores. On the other part, the electronic affinity of these polymers has also been confirmed by cyclic voltammograms in film state. © 2000 Elsevier Science Ltd. All rights reserved.

Keywords: Poly(aryl ether)s; Photoluminescence; 1,3,4-oxadiazole

1. Introduction

In 1990, Holmes et al. first reported the electroluminescence of conjugated poly(*p*-phenylenevinylene) (PPV) sandwiched between an anode and a cathode of appropriate work functions [1]. Up to now, the syntheses of electroluminescent polymers for organic light emitting diodes (OLED) have been investigated enthusiastically [2]. The conjugated polymers have excellent advantages for low cost, large-area flat-panel displays because of their unique processability, band gap tunability, and mechanical flexibility. Many conjugated polymers, such as PPV, poly(*p*-phenylene)s (PPPs) [3,4], polyfluorenes (PFs) [5], and polythiophenes (PTs) [6,7], have been synthesized for tentative application as emissive layers in organic light emitting diodes. However, some problems such as luminescent efficiency and long-term stability remain to be resolved.

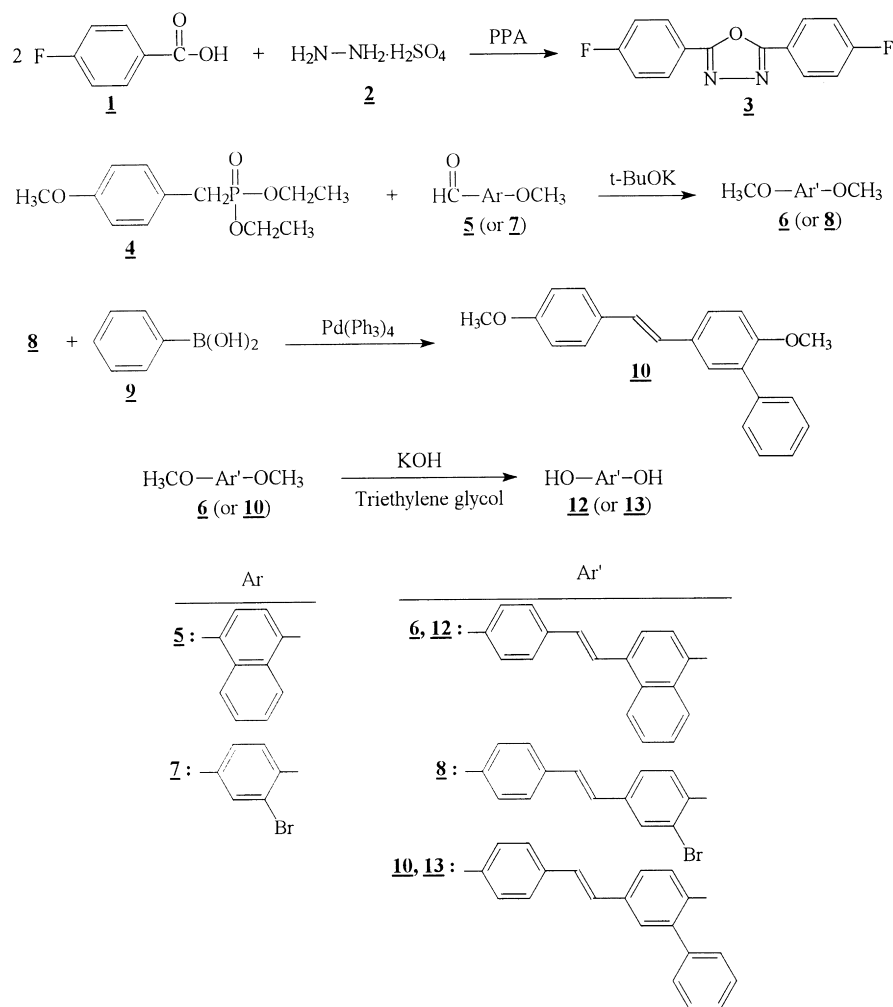
The emissive light of OLEDs arises from the recombination of electrons and holes, which are injected from the two opposite electrodes, to become excitons in an emissive polymer layer. To achieve high electroluminescence efficiency, it is necessary to balance the rates of injection of electrons and holes from the opposite contacts into the polymer layer [8]. In general, the conjugated polymers, like PPV, PPPs,

PFs, and PTs, have excess π electrons to own better holes affinity than electrons. This causes the imbalance of injection between electrons and holes. To overcome this problem, one is to use metals with low work function (such as Ca and Mg) as cathode materials [9]. Such metals, however, are so highly susceptible to atmospheric degradation and are difficult to encapsulate that the resulting devices suffer from poor stability. The other approach is to insert an additional electron-transporting layer between the emissive layer and the cathode [10]. Although this has been proven to be an effective way of achieving improved efficiency, the fabrication of multilayer devices is tedious and requires careful selection of each layer so that the solution of a subsequent layer will not attack the previous one. How to fabricate a single layer device with high efficiency and various colors has been attempted recently.

Many conjugated polymers containing electron-transporting chromophores have been synthesized to overcome these problems about work function and balance of injection [11–13]. However, excess conjugated length usually results in red-shifting the wavelength of photoluminescence or electroluminescence [12]. It has been demonstrated that the incorporation of flexible linkages in the backbone improves both the solubility and the processability [14].

In this work, new poly(aryl ether)s (**P1**, **P2**, **P3**) consisting of alternate isolated emitting chromophore and electron transporting 1,3,4-oxadiazole have been synthesized and

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Scheme 1.

characterized. The optical and electrochemical properties of these poly(aryl ether)s have been investigated in detail.

2. Experimental

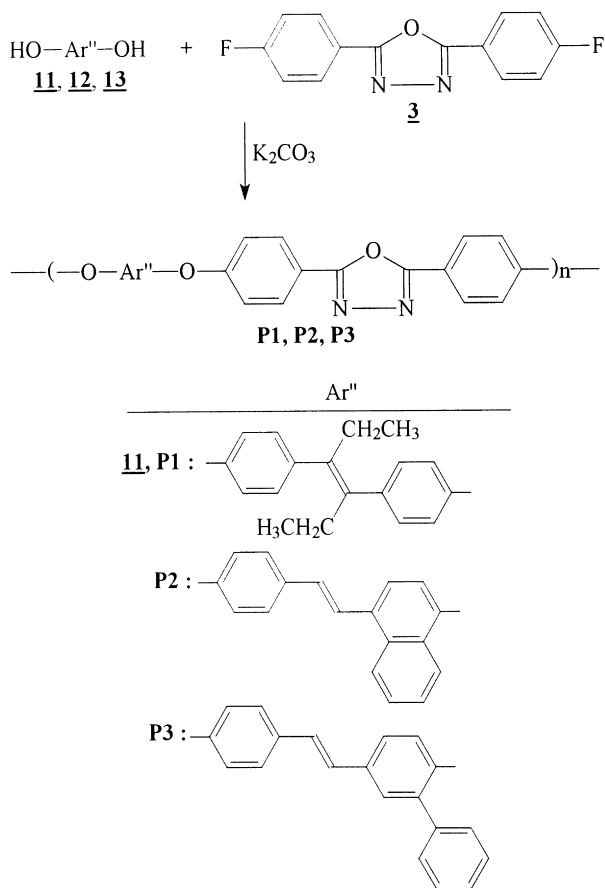
2.1. General methods

Reagent chemicals were purchased from Aldrich, Lancaster, TCI, and Acros Chemical Co. All new compounds were characterized by ^1H NMR, FT-IR, and elemental analyzer (EA). ^1H NMR spectra were recorded on Bruker 200 MHz FT-NMR. All chemical shifts were reported in δ ppm using tetramethylsilane (TMS) as internal standard. Infrared spectra were measured as KBr pellets on a fourier transform infrared spectrometer, model Valor III from Jasco. Elemental analyses were carried out on a Heraeus CHN-Rapid elemental analyzer. Thermal properties of the polymers were measured using a differential scanning calorimetry (DSC), model DSC-50 from Shimadzu, under nitrogen atmosphere at a heating rate of $10^\circ\text{C}/\text{min}$. The

thermogravimetric analysis (TGA) of the polymers were measured under nitrogen atmosphere at a heating rate of $20^\circ\text{C}/\text{min}$ using a Perkin–Elmer TGA-7 thermal analyzer. The UV/visible spectra were measured using a Jasco V-550 spectrophotometer. The photoluminescence (PL) spectra were obtained using a HITACHI F-4500 fluorescence Spectrophotometer. The cyclic voltammetry diagrammatic curves were measured at room temperature under nitrogen atmosphere using platinum disc electrode as working electrode, Ag/Ag^+ (0.01 M AgNO_3 , 0.1 M $(n\text{-Bu})_4\text{NClO}_4/\text{Acetonitrile}$) electrode as reference electrode and platinum wire electrode as auxiliary electrode supporting in $(n\text{-Bu})_4\text{NClO}_4/\text{Acetonitrile}$ solvent.

2.2. Monomers syntheses (Scheme 1)

The bis(fluoride) monomer (**3**) containing 1,3,4-Oxadiazole chromophore was synthesized from 4-fluorobenzoic acid and hydrazine sulfate. The emitting bis(phenol) monomers **12** and **13** were synthesized by the Horner–Wadsworth–Emmons



Scheme 2.

Olefination [15] and the Suzuki biaryl coupling reaction [16], respectively.

2.2.1. 2,5-Bis(4-fluorophenyl)-1,3,4-oxadiazole (3)

A round-bottom flask equipped with a condenser was charged with 4-fluorobenzoic acid (**1**) (1.405 g, 10 mmol), hydrazine sulphate (**2**) (0.6574 g, 5 mmol), and poly(phosphoric acid) (20 g). The reaction mixture was heated to 150°C and allowed to react for 6 h and then raised to 200°C for 2 h to effect the ring closure. The resulting product was precipitated in water, and then dried in a vacuum oven. It was recrystallized in 95% ethanol to afford **3** as white crystals. The yield was 74% (m.p.: 203–204°C). IR (KBr, cm^{-1}): 3058, 1607 ($-\text{C}=\text{N}-$), 1493, 1226, 1160, 1093, 1070, 845, 745. ^1H NMR (DMSO- d_6 , ppm): δ 7.44–7.53 (m, 4H), 8.16–8.23 (m, 4H). Anal. Calcd. (%) for $\text{C}_{14}\text{H}_8\text{F}_2\text{N}_2\text{O}$: N, 10.85; C, 65.12; H, 3.12. Found: N, 10.84; C, 65.18; H, 3.23.

2.2.2. 1-(1'-Methoxy-4'-naphthylvinyl)-4-methoxybenzene (6)

A mixture of diethyl-4-methoxybenzylphosphonate (**4**) (1.5495 g, 6 mmol) and *t*-BuOK (0.8753 g, 7.8 mmol) was dissolved in 12 ml *N,N*-dimethylformamide (DMF). The mixture was stirred at 5°C for 1 h in a nitrogen atmosphere.

It was added dropwise with DMF solution to 4-methoxy-1-naphthaldehyde (**5**) (1.1173 g, 6 mmol), and then allowed to react at room temperature for 24 h. The reaction mixture was poured into cold water to precipitate the products. The precipitates were isolated by filtration and dried in a vacuum oven. Recrystallisation in 95% ethanol results in white crystals of **6**. The yield was 61% (m.p.: 114–115°C). IR (KBr, cm^{-1}): 3009, 2960, 1580, 1507, 1463, 1386, 1301, 1264 (C–O–C), 1246, 1173, 1093, 1029 (C–O–C), 960 (*trans* C=C), 805. ^1H NMR (acetone- d_6 , ppm): δ 3.80–4.03 (s, 6H), 6.94–7.14 and 7.50–7.87 and 8.26–8.33 (m, 12H). Anal. Calcd. (%) for $\text{C}_{20}\text{H}_{18}\text{O}_2$: C, 82.73; H, 6.25. Found: C 82.70; H, 6.20.

2.2.3. 3-Bromo-4,4'-dimethoxystilbene (8)

A mixture of diethyl-4-methoxybenzylphosphonate (**4**) (2.3247 g, 9 mmol) and *t*-BuOK (1.212 g, 10.8 mmol) was dissolved in 18 ml *N,N*-dimethylacetamide (DMAc). The mixture was stirred at 5°C in a nitrogen atmosphere for 1 h, and then added with 3-bromo-4-methoxybenzaldehyde (**7**) (9 mmol) dissolved in 9 ml of DMF. It was allowed to react to 120°C for 24 h. The reaction mixture was poured into a large excess of cold water. The precipitates were isolated by filtration and dried in a vacuum oven. Recrystallisation in 95% ethanol resulted in white crystals of **8**. The yield was 42.4% (m.p.: 154–155°C). IR (KBr, cm^{-1}): 3010, 2939, 2840, 1603, 1509, 1493, 1455, 1263 (C–O–C), 1243, 1178, 1049, 1015 (C–O–C), 966 (*trans* C=C), 825. ^1H NMR (acetone- d_6 , ppm): δ 8.44 and 9.11 (s, 2H), 6.84–7.07 and 7.44–7.81 and 8.26–8.31 (m, 12H). Anal. Calcd. (%) for $\text{C}_{16}\text{H}_{15}\text{BrO}_2$: C, 60.21; H, 4.74. Found: C 60.26; H, 4.78.

2.2.4. 3-Phenyl-4,4'-dimethoxystilbene (10)

A round-bottom flask equipped with a condenser was charged with **8** (0.6384 g, 2 mmol), $\text{Pd}(\text{Ph}_3)_4$ (0.0694 g, 0.06 mmol), toluene (4 ml), Na_2CO_3 (2 ml of 2 M aqueous solution) under nitrogen atmosphere. The mixture was then added with phenylboric acid (**9**) (0.2926 g, 2.4 mmol) in 99% ethanol (1 ml) after completely mixing. It was allowed to react at 80°C for 12 h, followed with the addition of 10 ml benzene and then washed with distilled water after cooling to room temperature. The crude products obtained by concentration were recrystallized in 95% ethanol to result in white crystals of **10**. The yield was 82.2% (m.p.: 120–121°C). IR (KBr, cm^{-1}): 3001, 2962, 2837, 1604, 1511, 1441, 1268 (C–O–C), 1250, 1178, 1145, 1028 (C–O–C), 967 (*trans* C=C), 824. ^1H NMR (acetone- d_6 , ppm): δ 3.80 and 3.82 (s, 6H), 6.9–6.94 and 7.03–7.11 and 7.35–7.57 (m, 14H). Anal. Calcd. (%) for $\text{C}_{22}\text{H}_{20}\text{O}_2$: C, 83.51; H, 6.37. Found: C 83.42; H, 6.39.

2.2.5. 1-(1'-Hydroxy-4'-naphthylvinyl)-4-hydroxybenzene (12)

A mixture of **6** (1.1616 g, 4 mmol) and KOH (5.81 g, quintuple of weight of compound **6**) was dissolved in

Table 1
Polymerisation conditions and results of polymers

No.	Reaction temperature (°C)	Reaction solvent	Reaction time (h)	Yield (%)	η_{red} (dl/g) ^a
P1	170	NMP/CHP	8	88	0.58
P2	180	NMP/CHP	20	95	0.86
P2^b	180	Diphenyl sulfone	20	64	0.21
P3	170	NMP/CHP	20	87	1.34

^a 0.3 g/dl in NMP, 30°C.

^b 1 mmol **3** and **11**, 5 g diphenyl sulfone, 10 ml toluene.

16 ml triethylene glycol. The reaction mixture was stirred at 200°C for 12 h in a nitrogen atmosphere. The mixture was added with 50 ml of distilled water, and then washed with chloroform after cooling to ambient temperature. The aqueous layer was neutralized by adding 6 N HCl_(aq), and then extracted with ethyl acetate. The organic layer was dried with magnesium sulfate, filtered, and concentrated to give crude products of **12**. The crude products were purified by column chromatography on silica gel using mixture of ethyl acetate/hexane (*v/v* = 1/1) as eluent. Evaporation of the eluent and then recrystallization in chlorobenzene afforded pink powders of **12**. The yield was 46% (m.p.: 214–215°C). IR (KBr, cm⁻¹): 3287 (–OH), 1593, 1509, 1445, 1376, 1229, 1169, 960 (*trans* C=C), 828. ¹H NMR (acetone-*d*₆, ppm): δ 8.44 and 9.11 (s, 2H), 6.84–7.07 and 7.44–7.81 and 8.26–8.31 (m, 12H). Anal. Calcd. (%) for C₁₈H₁₄O₂: C, 82.42; H, 5.38. Found: C 82.35; H, 5.40.

2.2.6. -Phenyl-4,4'-dihydroxystilbene (**13**)

The synthetic procedures are the same as those of **12**. The yield was 81.4% (m.p.: 139–141°C). IR (KBr, cm⁻¹): 3375 (–OH), 3015, 1606, 1512, 1442, 1406, 1372, 1240, 1173, 963 (*trans* C=C), 825. ¹H NMR (acetone-*d*₆, ppm): δ 6.80–6.85 and 6.96–7.03 and 7.31–7.49 and 7.60–7.65 (m, 14H), 8.39 and 8.42 (s, 2H). Anal. Calcd. (%) for C₂₀H₁₆O₂: C, 83.31; H, 5.59. Found: C 83.34; H, 5.55.

2.3. Polymers syntheses (Scheme 2)

The syntheses of poly(aryl ether)s were obtained from the nucleophilic displacement reaction of oxadiazole-activated bis(fluoride) monomers (**3**) with bis(phenol) monomers (**11**–**13**). A typical synthesis of these polymers was conducted in a three-neck flask equipped with a nitrogen inlet, stirrer, Dean–Stark trap, and condenser.

For example, the flask was charged with **3** 0.2582 g (1 mmol), **11** 0.2684 g (1 mmol), and toluene (10 ml), 5 ml solvent mixture of NMP/*N*-cyclohexyl-pyrrolidone (CHP) (*v/v* = 1/1) and an excess of K₂CO₃ (0.2903 g, 2.1 mmol). The reaction mixture was then heated to 150°C for 2 h until the toluene was all condensed in the Dean–Stark trap. Upon dehydration, the polymerization was heat to a proper temperature of reaction as depicted in Table 1. The viscous mixture was diluted with 5 ml of NMP, and then dropped into 300 ml of methanol/acetone (*v/v* = 1/1)

solvent mixture. The precipitates were collected by filtration and then extracted with boiled water to remove trapped salts. The polymer (**P1**) was further purified by Soxhlet extractor using methanol as solvent for 24 h.

P1, IR (KBr, cm⁻¹): 3035, 2967 (–CH₂–), 2871 (–CH₂–), 1615, 1599, 1488, 1244 (C–O–C), 1167, 1099, 1064, 1013 (C–O–C), 960, 871. ¹H NMR (CDCl₃, ppm): δ 0.73–0.95 and 1–1.07 (t, 6H), 2.16–2.27 and 2.54–2.62 (m, 4H), 7.12–7.23 and 8.03–8.14 (m, 16H). Anal. Calcd. for C₃₂H₂₆N₂O₃. Found: C_{32.3}H₂₆N_{1.9}O₃.

P2, (using NMP/CHP as solvent): IR (KBr, cm⁻¹): 3057, 1606 (–C=N–), 1597, 1488, 1237 (C–O–C), 1236, 1166, 1097, 1051 (C–O–C), 1010, 960, 840. ¹H NMR (pyridine-*d*₅, ppm): δ 7.32–8.53 ppm (m, 20H). Anal. Calcd. for C₃₂H₂₀N₂O₃. Found: C_{32.2}H_{21.8}N₂O₃.

P3, IR (KBr, cm⁻¹): 3031, 1613, 1600, 1489, 1417, 1306, 1243, 1167, 1069, 1012, 962, 876. Anal. Calcd. for C₃₄H₂₂N₂O₃. Found: C_{34.6}H_{23.2}N₂O₃.

3. Results and discussion

3.1. Syntheses and characterization of poly(aryl ether)s

Nucleophilic displacement reaction of aryl halide with phenol usually needs catalyst to enhance reactivity and yield [17]. However, aryl halide become more susceptible toward nucleophilic aromatic substitution when it was activated by a substituent that can also accept the negative charge developed through the formation of a stabilized transition state (Meisenheimer complex) [18]. 1,3,4-Oxadiazole derivatives, which are commonly used as electron transporting layer in OLED, have the electron-withdrawing ability so that it can activate nucleophilic displacement reaction. The NMP/CHP solvent mixture was used as the medium of polymerisation since CHP was immiscible with water at elevated temperature and water that deactivated phenoxide was effectively removed as the toluene azeotrope via the Dean–Stark trap. Alternatively, diphenyl sulfone, a high temperature solvent used to prepare poly(aryl ether ether ketones), was also investigated as a solvent medium for the polymerisation. However, the reduced viscosity (η_{red}) of **P2** using diphenyl sulfone as solvent was lower than with NMP/CHP mixture as solvent (Table 1). The ¹NMR

Table 2
Solubility of polymers **P1–P3** (++, soluble at room temperature; +, soluble by heating; --, insoluble)

No.	η_{red}	DMSO	DMAc	NMP	Pyridine	CHCl ₃	THF	Toluene
P1	0.58	--	++	++	++	++	++	++
P2^a	0.86	--	++	++	++	--	--	--
P2^b	0.21	++	++	++	++	--	--	--
P3	1.34	--	++	++	+	--	--	--

^a NMP/CHP is the solvent of polymerization.

^b Diphenyl sulfone is the solvent of polymerization.

spectrum also showed that polymerisation in diphenyl sulfone led to unknown byproducts. Therefore, the NMP/CHP solvent mixture was a more suitable solvent for this polymeric system. All of the polymers have η_{red} greater than 0.58 dl/g as shown in Table 1. Another advantage of poly(aryl ether)s compared to fully conjugated PPV is the improvement of solubility. The **P1**, **P2** and **P3** were miscible with common organic solvents (Table 2). **P1** exhibits the best solubility since it can be dissolved in common chloroform (CHCl₃), tetrahydrofuran (THF), and toluene. In addition, ethyl side chain also tends to increase the solubility of **P1** by the same reason. The ¹H NMR spectrum (Fig. 1) shows that **P1** is a mixture of *cis*- and *trans*-form. The ratio of *cis* and *trans*-form is about 1/4 estimated from the area of triplets at 1.04 and 0.84 ppm, respectively. The *cis*-section of **P1** increases the solubility because of its larger free volume than *trans* one. However, monomer

11 bought from TCI Chemical Co. is 100% *trans*-form. The isomerization maybe occurred during polymerization.

Thermal transition properties of the poly(aryl ether)s were investigated using a DSC. All of polymers exhibit glass transition temperature (T_g) greater than 181°C as shown in Fig. 2. Their decomposition temperatures in nitrogen were estimated by a TGA and are above 470°C as shown in Fig. 3. The excellent thermal properties promise that these polymers could endure the high temperature in the process of fabrication and operation.

3.2. Optical characterization

An expected gradual bathochromic shift of photoluminescent spectra (Fig. 4) is exhibited with increasing the conjugated aryl ring in the polymers. The model polymer, **P1**, has a maximum emissive wavelength at 382 nm and a shoulder at 396 nm. The shoulder seems due to the emission of 2,5-bisphenyl-1,3,4-oxadiazole chromophore. In order to increase the conjugation in aryl ring, naphthalene and biphenylene was introduced into **P2** and **P3**, respectively. The photoluminescent maxima are 430 nm (blue) for **P2** and 395 nm (purple) for **P3**. It is worth noticing that the bathochromic shift is 48 nm for **P2**, but only 13 nm for **P3**. As it is known that the distortion from the planarity in unsubstituted oligophenyls is because of the *ortho*-hydrogen repulsion [19], it is expected that the presence of the substituent will lead to a nonplanar conformation. The

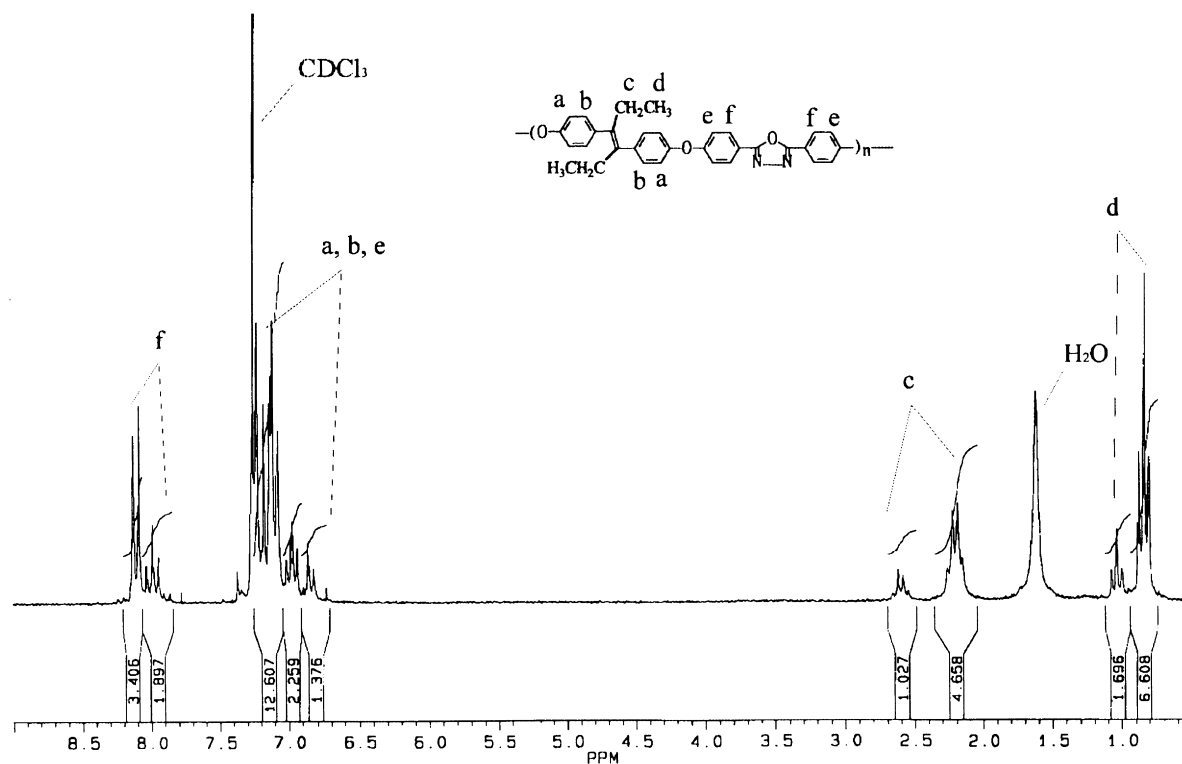


Fig. 1. ¹H NMR spectrum of **P1**.

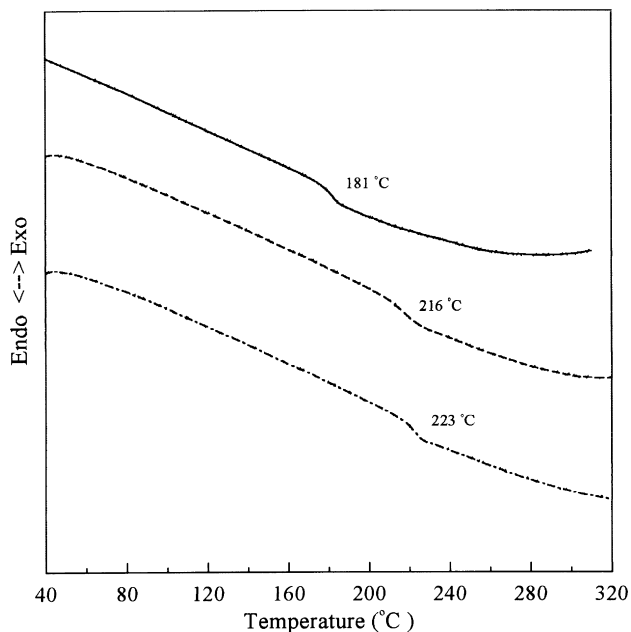


Fig. 2. Differential scanning calorimetric curves of: **P1** (—); **P2** (---); **P3** (- · -) obtained from the second scan with heating rate of 10 $^{\circ}\text{C}/\text{min}$.

nonplanar conformation decreases the effective conjugated length so that the bathochromic shift in **P3** is smaller.

Photoluminescence spectra of **P2** in film state and in toluene, THF, NMP, and DMAc were shown in Fig. 5. There was a conspicuous hypsochromic shift in low polar solvent. For example, the luminescence maximum shifts from 382 nm in film state to about 360 nm in toluene.

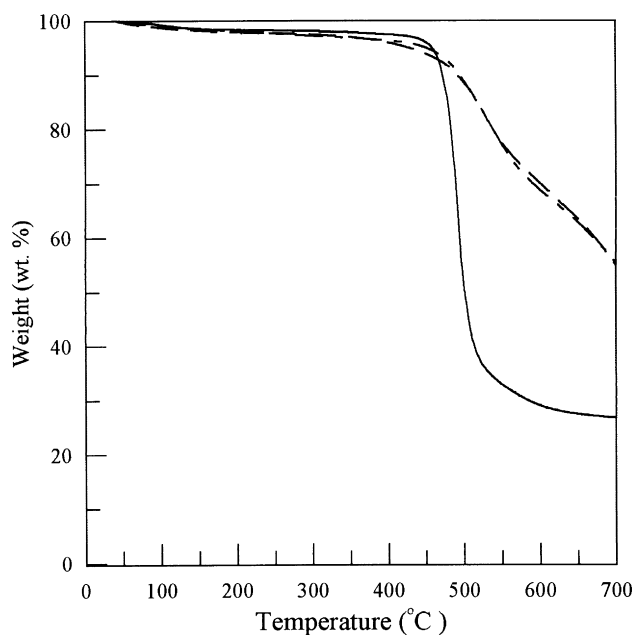


Fig. 3. Thermogravimetric curve of: **P1**(—); **P2**(---); **P3**(- · -) at a heating rate of 20 $^{\circ}\text{C}/\text{min}$ in nitrogen.

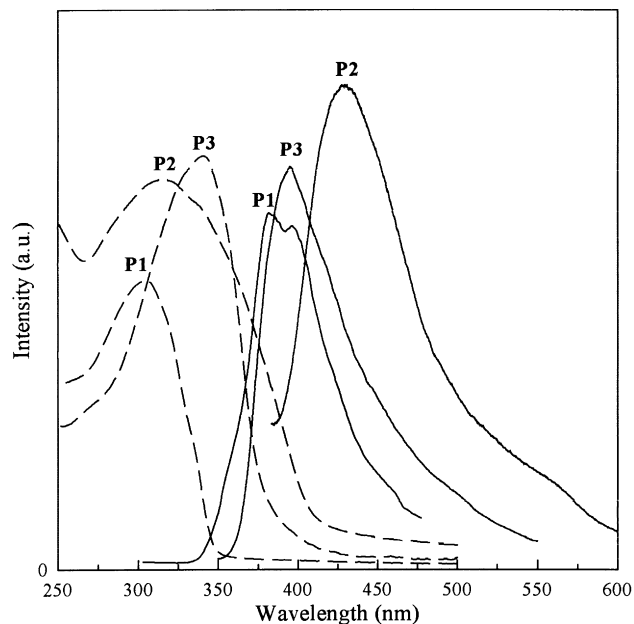


Fig. 4. Photoluminescence (—) and UV/vis absorption (---) spectra of **P1**, **P2**, **P3** in film state at room temperature.

However, only little shift could be observed in other higher polar solvents such as THF, DMAc, and NMP. The reason is maybe that the interaction between polymer chains and polar solvent is analogous with the interaction between polymer chains in film state. As shown in Fig. 4, the absorption maxima of polymer films are 305 nm for **P1**, 314 nm for **P2**, and 341 nm for **P3**. The wavelength of luminescence maxima are longer than that of absorption and the Stokes

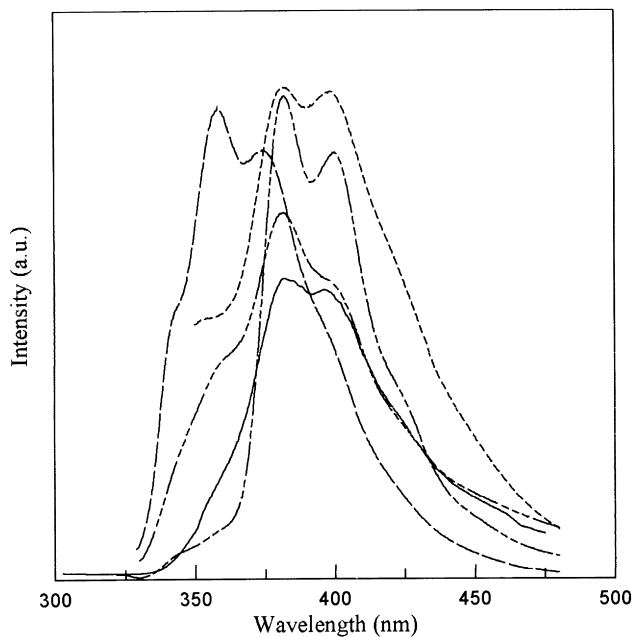


Fig. 5. Photoluminescence spectra of **P1** in film state (—) and in Toluene (---); THF (- · -); NMP (· · ·); and DMAc (— · —).

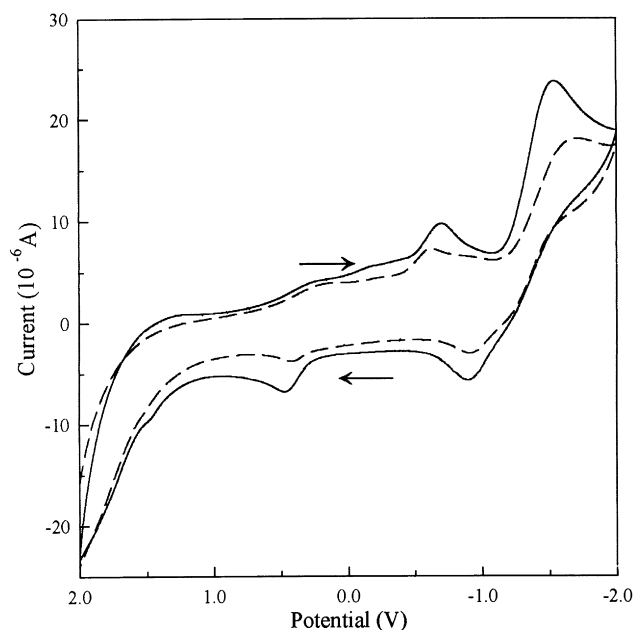


Fig. 6. Cyclic voltammograms of **P2** (—) and **P3** (---) in film on Pt electrode in CH_3CN solution of Bu_4NClO_4 (0.1 M) at a scan rate of 100 mV/s.

shift can be determined to be 77 nm for **P1**, 116 nm for **P2**, 54 nm for **P3**.

Clearly, in our system the tuning factor of the emissive wavelength is not only the number of conjugated ring but also the steric conformation. Accordingly, aryl vinylene chromophores could be molecularly designed by suitable substitution to regulate its emitting colors. In the future, three kinds of chromophores (blue, green and red) could be designed and further copolymerized each other to obtain a copolymer which emits white light.

3.3. Electrochemical properties

The cyclic voltammograms (CV) between 2.0 and -2.0 V of **P2**, **P3** are shown in Fig. 6. Both polymers exhibit reversible redox processes in film state. For **P2**, the reduction potentials are located at -0.699 and -1.501 V, while reoxidation potentials are at 0.466 and -0.915 V, respectively. **P3** shows similar redox trend to **P2**, i.e. reduction potential at -0.608 and -1.653 V with reoxidation ones at 0.405 and -0.929 V. Unlike the conjugated polymers, emissive chromophores and electron-transporting units in **P2** and **P3** retain their own electrochemical properties. For example in **P2**, -0.699 and 0.466 V are the redox peaks of emissive aryl vinylene chromophores; -1.501 and -0.915 V are the peaks of electron-transporting 1,3,4-oxadiazole units. It means that the intramolecular charge transfer along the backbones of the polymers is not allowed because of the ether-spacers. However, the electron-transporting units can be the gateway at which electrons inject into the polymers and then jump to emissive chromophores by intermolecular charge transfer.

4. Conclusions

Three new poly(aryl ether)s consisting of alternate emissive aryl vinylene and electron transporting 1,3,4-oxadiazole chromophores have been successfully synthesized and characterized. All of the polymers have η_{red} greater than 0.58 dl/g and excellent thermal properties. Their decomposition temperatures are above 470°C in nitrogen atmosphere. Unlike fully conjugated polymers, these polymers are miscible with organic solvent such as NMP, DMAc and pyridine. The photoluminescent maxima of **P1**, **P2** and **P3** are, respectively, 382, 430 and 395 nm, which are controlled by the conjugated length and the steric conformation of emissive chromophores. In the observation of cyclic voltammograms, electron-withdrawing 1,3,4-oxadiazole exhibits a reduction potential at -1.501 V for **P2** and -1.653 V for **P3**. It indeed offers a section on which electrons can be accepted by the polymers.

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