

Synthesis and optical properties of an alternating copolymer composed of MEHPV and 1, 3, 4-oxadiazole

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Abstract: A poly (*p*-phenylenevinylene) (PPV) alternating copolymer, poly [(2, 5-diphenylene-1, 3, 4-oxadiazole)-4,4'-vinylene-alt-2-methoxy-5-(2-ethylhexyloxy)-1, 4-phenylenevinylene] (oxa-MEHPV), is synthesized by Heck coupling reaction and characterized with UV-vis, Fourier transform infrared (FT-IR), ¹H-NMR and photoluminescence (PL) spectroscopy. oxa-MEHPV possesses an outstanding thermal stability and shows excellent solubility in common organic solvents such as dichloromethane, chloroform, toluene, and tetrahydrofuran (THF). The introduction of the electron-deficient 1, 3, 4-oxadiazole units into the MEH-PPV backbone also increases the electron affinities of the conjugated segment, which leads to the blue-shift of the maximum absorption wavelength and makes the polymer have a high optical band-gap energy, good electron-transporting stability and high PL quantum yield.

Key words: MEH-PPV; 1, 3, 4-oxadiazoles units; electron injection; photoluminescence; polymer light-emitting diode

Since the first poly (*p*-phenylenevinylene) (PPV) was reported by the Cambridge group in 1990^[1], the synthesis and applications for polymer light-emitting diodes of PPV derivatives (PPVs) have attracted much attention because of their easily adjustable structures, excellent properties of light-electric interconversion, and good mechanical properties^[2–3]. Great effects have been made to design novel PPVs containing different kinds of side-chains or main-chains for various applications such as absorption spectrum, quantum yield, and optical band gap energy E_g ^[4]. Among these, poly-[2-methoxy-5-(2-ethylhexyloxy)-1, 4-phenylenevinylene] (MEH-PPV) is the most active material due to the presence of long branched ethylhexyloxy substituent on an aromatic ring, which makes the MEH-PPV possess remarkable solubility and processability^[5]. Lee et al.^[6] showed that PLEDs with high red, green, and blue color purity could be fabricated using MEH-PPV and polyalkylfluorenes as a single light emitting layer. Chuangchote et al.^[7] reported color change of electrospun polystyrene/MEH-PPV fibers from orange to yellow through partial decomposition of MEH side groups. Winder et al.^[8] also measured the degenerate two-photon absorption spectrum for MEH-PPV in a different spectral range (700 to 900 nm), using two-photon excited fluorescence measurements. Thus, MEH-PPV may potentially be

used in polymer light-emitting diodes and will attract great attention.

It is well known that MEH-PPV is a hole-conducting material with high levels of lowest unoccupied molecular orbital (LUMO), which makes electron injection more difficult than hole injection, and it results in the imbalance of electron or hole injections at cathodes or anodes^[9]. Previous studies have demonstrated that the balance of electrons and holes injected from opposite contacts was crucial in order to gain high electroluminescent efficiency^[10–11]. There are several strategies to improve the efficiency of organic light-emitting diodes (OLEDs) with PPVs as the emitting layer. For example, using a metal with a low work function can reduce the charge injection barrier at the cathode and improve the luminance efficiency by balancing injected electrons and holes^[12]. Multilayer devices with the electron transport layer (ETL) between a polymer layer and negative electrodes have been found to be more efficient than single-layer devices^[13]. Moreover the introduction of electron transport moieties in the polymer side chain or polymer main chains have been reported by many research groups^[14].

In this paper, we use 1, 3, 4-oxadiazole units as the electron transport moieties to synthesize oxa-MEHPV by a palladium catalyzed Heck coupling reaction. The 1, 3, 4-oxadiazole unit is a chromophore, which has good electron-transportation, thermal stability and antioxidant ability. After being introduced to the MEH-PPV molecule, the polymer exhibits higher optical band-gap energy and high PL quantum yield, which can hopefully be used as a highly efficient polymer light-emitting diode (PLED).

1 Experimental

1.1 Materials

Dimethyl sulfoxide (DMSO) and chloroform were used after distillation over potassium carbonate. 2-ethylhexyl bromide (98.5%), triphenylphosphine (99%), sodium methoxide (98.5%), PbCl₂ and other chemicals were purchased from Aldrich Chemical Co., Inc (NJ) and used as received unless otherwise described.

1.2 Characterization methods

¹H-NMR spectra of the compounds and polymers were recorded on a 300 MHz Bruker NMR spectrophotometer in CDCl₃ at room temperature, containing a small amount of TMS as an internal standard. Infrared spectra of the samples were recorded with a Nicolet 700 Fourier transform infrared (FT-IR) spectrophotometer in the solid state. High-performance liquid chromatography (HPLC) was performed on a Perkin-Elmer instrument using Spheri-5 RP-18 column (4.6 mm × 250 mm i. d., 5 μm coating), and the compounds were eluted with methanol and detected by a UV spectrophotode-

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tector at 254 nm. Elemental analyses were performed with Elementar Vario MICRO. The molecular weights of the polymers were determined by gel permeation chromatography (GPC) using a Perkin-Elmer series 200 apparatus in THF with polystyrene as a standard. The flow rate of THF was maintained as 1.0 mL/min at 40 °C. The thermal stability of the polymers was determined using a DuPont 9900 analyzer at a heating rate of 10 °C/min in nitrogen atmosphere. The absorption and emission studies were done using a UV-2201 UV-vis spectrophotometer and a Spex (NJ) FL-2T2 spectrofluorometer. The solution spectra were recorded in chloroform, while the solid state spectra were obtained from polymer thin films prepared by spin-casting the chloroform solutions on glass substrates.

1.3 Synthesis of oxa-MEHPV

● **Methoxy-4-(2-ethylhexyloxy) benzene (1)** The synthesis was performed as in Ref. [15]. Briefly, 5.4 g (0.1 mol) sodium methoxide, 50 mL methanol, and 12.4 g (0.1 mol) 4-methoxyphenol were added to a dry 250-mL, three-necked, round-bottom flask under an N₂ atmosphere. After being solvated in an ultrasonic bath, 20.0 mL (0.1 mol) of 2-ethylhexyl bromide was then added dropwise over a period of 0.5 h. The reaction was kept in the supersonic waves (33 ± 2) kHz for 7 h. The liquid mixture was concentrated under reduced pressure and was followed by the extraction of the product with chloroform and water. By the current method, a 19.5 g amount of the target product was achieved, which reflected an 83% yield and a purity of 99.2% by HPLC. The ¹H-NMR spectrum was consistent with that previously reported^[13], ¹H-NMR (ppm): 6.87 (s, 4H), 3.79 (s, 5H), 1.69 to 0.92 (m, 15H).

● **α, α'-dibromo-2-methoxy-5-(2-ethylhexyloxy) xylene (2)** The synthesis of compound (2) was performed according to a method recorded in Ref. [16]. Yield: 76%; purity: 97.5% (HPLC); ¹H-NMR (ppm): 6.84 (s, 2H), 4.66 (s, 4H), 3.89 (s, 2H), 3.77 (s, 3H), 1.69 (t, 1H), 1.68 to 1.30 (m, 8H), 0.92 to 1.00 (t, 6H).

● **Polymer MEH-PPV** Compound MEH-PPV was prepared by a modified method^[17]. To a round-bottom flask, 0.64 g (1.5 mmol) compound (2) and 15 mL THF were added, and an N₂ atmosphere was established. A solution of potassium tert-butoxide in THF 30 mL (0.25 mol/L) was then added at a rate of 20 mL/h via a syringe pump under stirring. After the complete addition of the base, the reaction mixture was stirred for an additional 8 h. The reaction mixture was poured into a volume of methanol under vigorous stirring. Then the polymer was collected by suction filtration. The polymer was then dissolved in THF, precipitated with methanol, then collected and dried under reduced pressure to yield 0.2 g (52%) of MEH-PPV.

● **1-methoxy-4-(2-ethylhexyloxy)-2,5-yield divinylbenzene (4)** The Stille coupling reaction was used to prepare compound (4)^[18]. In a dry 100-mL conical flask, 2.11 g (5 mmol) compound (2), 3 g (11 mmol) triphenylphosphine and 15 mL toluene were added. The reaction was then heated to reflux for 4 h. After cooling to ambient temperature, 3.5 g (yield: 80%) of α, α'-dibromo (triphenylphosphine)-2-methoxy-5-(2-ethylhexyloxy) xylene (3) solid with purity of 97.3% (HPLC) was collected by filtration and dried in vacuum. In a dry 100-mL, three-necked, round-bottom flask,

1.33 g (1.5 mmol) compound (3) was dissolved in 25 mL CH₂Cl₂ and 6 mL formalin and 20 mL 1.0 mol/L NaOH solution in distilled water was then added dropwise over a period of 1 h. After being stirred at room temperature for 12 h, the resulting mixture was partitioned between CH₂Cl₂ and water. The aqueous phase was removed and extracted twice with CH₂Cl₂. The combined organic phase was washed twice with saturated sodium chloride and dried over anhydrous Na₂SO₄. The crude material was recrystallized from ethanol/water (1:1) to give 0.24 g (yield: 53%) of compound (4) (purity: 96.9%, HPLC). ¹H-NMR (ppm): 7.33 (d, 2H), 7.08 (s, 2H), 5.92 (d, 2H), 5.43 (d, 2H), 3.77 to 3.81 (s, 2H), 3.74 to 3.77 (s, 3H), 1.67 to 1.69 (t, 1H), 1.29 to 1.67 (m, 8H), 0.89 to 1.02 (t, 6H).

● **2,5-bis(4-bromophenyl)-1,3,4-oxadiazole (5)** To a round-bottom flask, 2.7 mL phosphoric acid and 6.0 g (42.1 mmol) P₂O₅ were added. The mixture was stirred and heated at about 120 °C for approximately 0.5 h until the white solid dissolved. Then 1.3 g (10 mmol) hydrazine sulfate and 4 g (19.9 mmol) 4-Bromobenzene carboxylic acid were added. The mixture was subsequently heated at about 140 °C for 8 h and then poured into water. The whitish solid was filtered and washed with water five times. The crude material was recrystallized from ethanol to afford 3.4 g (yield: 45%) of compound (5) (purity: 98.8%, HPLC). IR (KBr, cm⁻¹): 1601.43, 1478.62, 1071.38, 1009.28, 836.43, 742.10. ¹H-NMR (ppm): 7.71 (d, 4H), 8.02 (d, 4H).

● **Polymer oxa-MEHPV** Polymer oxa-MEHPV was prepared by a Heck coupling reaction^[19-20]. 0.30 g (1 mmol) compound (4), 0.4 g (1 mmol) compound (5), 0.007 g (0.04 mmol) PdCl₂, 0.0656 g (0.25 mmol) triphenylphosphine, 7 mL DMF and 0.7 mL triethylamine were added to a flask purged with N₂. The mixture was stirred at 110 °C for 12 h under N₂. The reaction mixture was filtered and the filtrate was poured into methanol. The yellow-brown precipitate was filtered, washed with methanol three times, and dried to afford 0.42 g oxa-MEHPV (yield: 69%).

2 Results and Discussion

2.1 Synthesis and characterization of polymers

Fig. 1 shows the synthetic route of the monomers and polymers. In particular, using sodium methoxide and methanol as catalysts instead of KOH and water, the increased yield and purity of compound (1) are obtained in an ultrasonic bath instead of by way of heat as described in previous literature^[18], but the reaction time is decreased to 7 h.

The chemical structure of MEH-PPV and oxa-MEHPV are characterized by FT-IR spectroscopic measurements. Compared with MEH-PPV, oxa-MEHPV displays some new peaks at 1207, 1070, 1010, 1482, 1674, 1548, 1528 cm⁻¹, respectively. The peaks at 1207, 1070 and 1010 cm⁻¹ are attributable to the deformation C—O—C stretching of ether bond and oxadiazole in a 1,3,4-oxadiazole ring. A strong absorbance band at 1482 cm⁻¹ is assigned to the asymmetric C=N stretching in the 1,3,4-oxadiazole ring, while the peaks at 1674, 1548 and 1528 cm⁻¹ correspond to specific absorbance bands of the 1,3,4-oxadiazole ring. This suggests that the binding of the 1,3,4-oxadiazole ring to MEH-

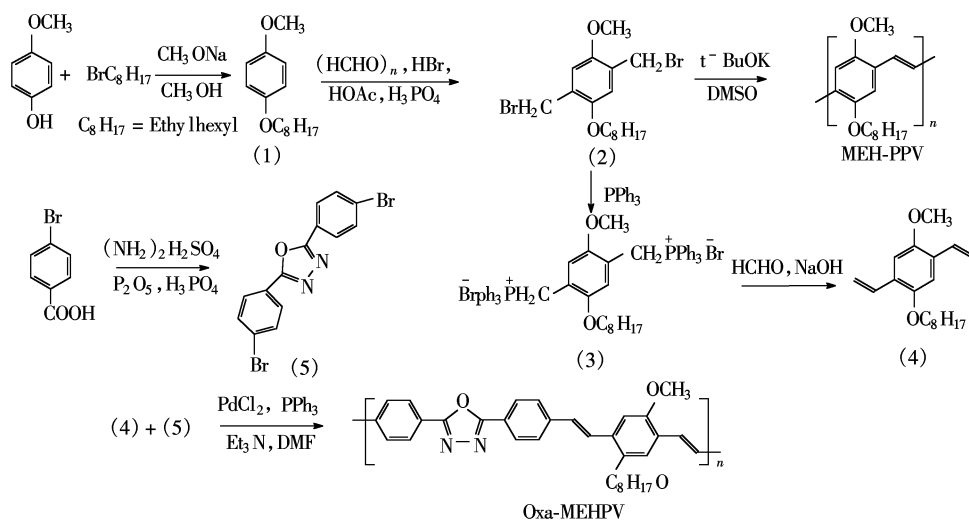


Fig. 1 Synthetic routes for the monomer and polymers

PPV and the oxa-MEHPV are successfully polymerized. This can be further supported by our $^1\text{H-NMR}$ measurements. The $^1\text{H-NMR}$ spectrum of oxa-MEHPV in CDCl_3 solution shows peaks at 7.19 to 8.27 (m, 10H, aromatic, and 4H, $\text{HC}=\text{CH}$); 3.73 to 4.02 (m, 5H, OCH_2); 0.96 to 1.69 (m, 15H, other aliphatic). All these results from FTIR and $^1\text{H-NMR}$ spectra of the polymers are well consistent with their chemical structures.

2.2 Physical properties of polymers

Oxa-MEHPV containing phenylene, oxadiazole rings and vinylene bonds along the main chain per repeat unit leads to a fully conjugated backbone. It shows excellent solubility in common organic solvents like THF, chloroform, dichloromethane, chlorobenzene; thus, it is easy to obtain a transparent and homogeneous thin film on a glass slice by spin-casting it from these solvents. Fig. 2 shows the TGA curves of MEH-PPV and oxa-MEHPV. It is found that both of the polymers lose less than 5% of their mass after being heated up to 300 $^\circ\text{C}$, indicating a good thermal stability of two polymers. However, oxa-MEHPV is more stable than MEH-PPV from 250 to 420 $^\circ\text{C}$, which can be ascribed to the increased chain rigidity caused by a combination of the 1,4-phenylene and 1,3,4-oxadiazole rings along the backbone of polymers. However, oxa-MEHPV loses a little more of its mass than that of MEH-PPV after being heated up to 420 $^\circ\text{C}$. This can be ascribed to the thermal decomposition of 1,3,4-oxadiazole rings.

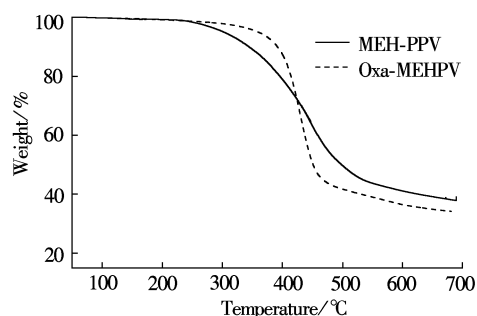


Fig. 2 TGA curves of oxa-MEHPV and MEH-PPV

2.3 Optical properties of polymers

Fig. 3 shows the UV-vis absorption spectra of the two polymers in CHCl_3 . The spectrum of MEH-PPV displays two absorbance peaks at 325 and 482 nm, respectively, while the absorption peak of oxa-MEHPV is blue-shifted and shows a maximum absorbance at about 319 nm and a weak shoulder peak at about 420 nm. The longer wavelength peak is attributed to the electronic $\pi-\pi^*$ transition along the conjugated backbone, whereas the shorter wavelength peak is originated from the electronic transition of the 1,3,4-oxadiazole ring. Therefore, the existence of 1,3,4-oxadiazole units reduces the conjugation length of the polymer backbone.

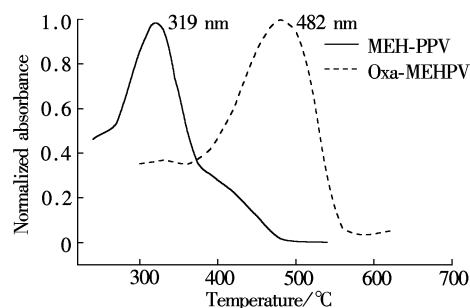


Fig. 3 UV-vis spectra of MEH-PPV and oxa-MEHPV in CHCl_3

E_g is calculated according to the following formula:

$$\alpha h\nu = B(h\nu - E_g)^n \quad (1)$$

where α is the absorbance coefficient; h is Planck's constant; ν is the light frequency; B is the constant with the given material, and n is the transition type. The value of n is 0.5 for a direct band transition, and amounts to the absorbance for the given sample, so the $(h\nu A)^2 - h\nu$ curves of the polymers can be drawn as shown in Fig. 4. The linear edge of absorbance is extended and intercrossed with the energy axis, and E_g values of MEH-PPV and oxa-MEHPV are 2.30 and 3.31 eV, respectively. It is obvious that the introduction of the 1,3,4-oxadiazole unit in oxa-MEHPV can increase the band gap energy.

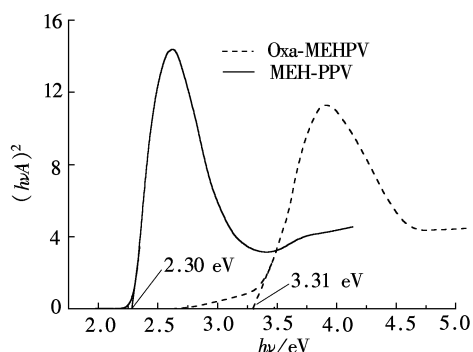


Fig. 4 $(h\nu A)^2$ - $h\nu$ curves of MEH-PPV and oxa-MEHPV in CHCl_3

Fig. 5 shows the excitation and emission curves of the two polymers in thin films. With the same thickness of the films, the PL spectra are obtained by excitation at absorption maxima of about 485 nm for MEH-PPV and 352 nm for oxa-MEHPV. The UV-vis absorption maxima of the films are shifted to longer wavelengths in comparison to those in solution. This phenomenon can be accounted for by the fact that PPVs in solution can be in the form of a coil with a small effective conjugation length, and the solid state can be a more extended structure with more extensive electronic delocalization. The polymers emitted red light with PL maxima at 593 nm for MEH-PPV and yellowish-green light at 525 nm for oxa-MEHPV. The emission spectra of oxa-MEHPV is blue shifted by 68 nm. Such a blue shift for the polymer can be attributed to an intramolecular energy transfer from the higher composition of the electron-donating alkyloxy groups to the 1,3,4-oxadiazole unit of the polymer. Therefore, the increase in oxadiazole ring composition in the polymer backbone results in the increase of the electron density along the π -conjugated polymer backbone by extension of the effective chromophore. The PL quantum yields of the poly-

mers in THF solution are determined using quinine sulfate as a standard^[21]. The oxa-MEHPV has an obviously higher PL quantum yield of 0.41, compared to that of MEH-PPV of 0.21.

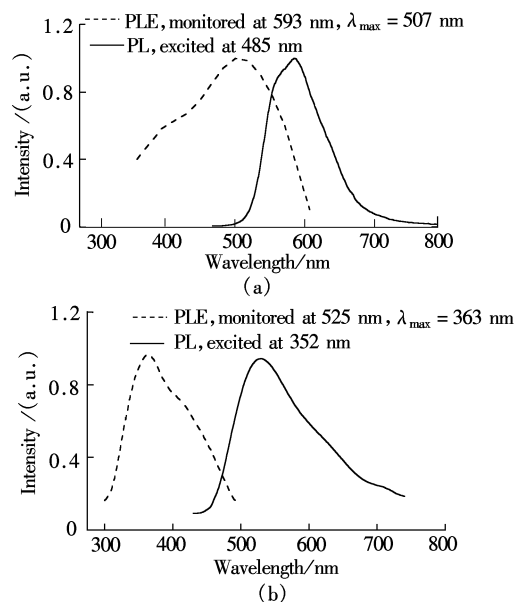


Fig. 5 Normalized PLE and PL spectra of MEH-PPV and oxa-MEHPV in thin films. (a) MEH-PPV; (b) Oxa-MEHPV

The PLE spectra of MEH-PPV and oxa-MEHPV in thin films are overlapped with the absorption spectra, with maxima at 507 and 363 nm, respectively. The red shift of the PLE spectra from the absorption spectra indicates that the PLE spectra are much more affected by the aggregated states and the chromophores with long conjugation length than by the absorption spectra^[22]. The properties of the polymers are summarized in Tab. 1.

Tab. 1 Properties of the polymers

Polymer	M_n	M_w	PDI	$T_d/^\circ\text{C}$	Solution(CHCl_3)			Film/nm		
					Absorption $\lambda_{\text{max}}/\text{nm}$	E_g/eV	Φ_f	λ_{ex}	PL λ_{max}	PLE λ_{max}
MEH-PPV	24 000	30 960	1.29	300	482	2.30	0.21	485	593	507
oxa-MEHPV	8 029	13 650	1.7	350	319	3.31	0.41	352	525	363

Note: M_w is the weight-average molecular weight; M_n is the number-average molecular weight; T_d is the temperature resulting in 5% weight loss based on initial weight; E_g is the optical energy gap calculated from the UV-vis spectra; and Φ_f is the PL quantum yield in THF solution which were determined by comparison to quinine sulfate.

3 Conclusion

In this paper, we synthesize an alternating copolymer (oxa-MEHPV) with 1,3,4-oxadiazole units in the backbone via Heck reaction. In comparison with MEH-PPV, oxa-MEHPV has a good solubility in common organic solvent and outstanding thermal stability. Optical results show that the optical band-gap energy of oxa-MEHPV (3.31 eV) is higher than that of MEH-PPV (2.30 eV), and has an obviously higher PL quantum yield of 0.41 as compared with 0.21 for the latter. The UV-vis absorption, PL and PLE results show that oxa-MEHPV has an obvious blue shift due to the introduction of the electron-deficient 1,3,4-oxadiazole units in MEH-PPV backbone. The oxa-MEHPV has good electron-transporting stability and can be potentially used in polymer

light-emitting diodes.

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一种含噁二唑基团 MEHPV 交替共聚物的合成及其荧光性能

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摘要:通过钯催化的 Heck 耦合反应,合成了一种含噁二唑基团与 MEHPV 单元的交替共聚物(oxa-MEHPV),并利用 UV-vis, FT-IR, $^1\text{H-NMR}$ 对其单体和聚合物的结构进行了表征,研究了目标聚合物的荧光性能.由于在 MEH-PPV 的主链骨架上引入噁二唑基团,oxa-MEHPV 分子链的刚性得到增强,表现出更好的热稳定性能,在常规有机溶液如氯仿、二氯甲烷、甲苯等中也有更好的溶解性.同时,由于噁二唑具有很好的电子亲和能力,聚合物紫外吸收波长发生了较大的蓝移,较 MEH-PPV 拥有了更好的电子迁移率和更高的荧光量子效率,因而在聚合物电致发光(PLED)领域有潜在的应用前景.

关键词:MEH-PPV;噁二唑;电子注入;光致发光;聚合物电致发光

中图分类号:O361;TN383.1