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White Electroluminescence from a Star-like Polymer with an Orange Emissive Core and Four Blue Emissive Arms**

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White light-emitting molecules and polymers have received great attention because of their potential utilization in full-color displays with color filters, backlights, and lighting applications.^[1–4] A general approach to obtain white emission from a single molecule or polymer is to use blue emission from the molecule or polymer itself and orange emission from the aggregate/excimer/electroplex. Several white-light emitting molecules have been developed, such as 1,3,5-tris(2-(9-ethyl-carbazyl)ethylene)benzene,^[2a] 3,6-di-*tert*-butyl-9-(4-[4-(3,6-Di-*tert*-butyl-carbazol-9-yl)-phenyl]-*E*but-1-en-3-ynyl)-phenyl),^[2b] and 3,5-dimethyl-2,6-bis(dimesitylboryl)-dithieno[3,2-*b*:2',3'-*d*]thiophene.^[2c] A few white-light emitting polymers, for example, polyaniline,^[3a] an anthracene-containing polymer,^[3b] an oxadiazole-containing poly(phenylene vinylene) (PPV) with a nonconjugated spacer,^[3c] and platinum-functionalized random copolymers^[3d] have also been reported. However, white emission of this kind suffers from low electroluminescence (EL) efficiency because the formation of the aggregate/excimer/electroplex decreases the radiative decay rate of excitons.

Recently, our group proposed several approaches to generate two- and three-color white EL from a single-polymer system with multiple emission components based on the control of energy transfer and charge trapping among the emissive species.^[5,6] For example, one approach for two-color white emission is to incorporate an orange chromophore to the main chain or the side chain of a blue light emitting polymer host.^[5] Another approach for three-color emission is to covalently attach both a green chromophore and a red chromophore to a blue emissive polymer host with different attachments,^[6a,6b] or to use blue, green, and red dopant units as individual emissive species and polyfluorene as individual polymer host.^[6c] White EL from these single polymers has promising color stability

and excellent EL performance. We have also enhanced the EL performance of the white electroluminescent polymers by improving the photoluminescence (PL) quantum efficiency of the orange species,^[5c] by covalently attaching the orange chromophore to the side chain of the blue polymer to form an intramolecular dopant/host system,^[5d] and by enhancing the efficiency and red-shifting the spectrum of the blue species.^[5e] Very recently, several research groups, such as Cao et al.,^[7] Shu et al.,^[8] Shim et al.,^[9] Hsu et al.,^[10] and Chen et al.^[11] have also succeeded in developing white electroluminescent single polymers with both singlet and triplet emission and/or with three emission species. However, all of the white electroluminescent single polymers reported so far are linear polymers. Therefore, from a synthetic chemistry perspective, it is still a great challenge to develop white electroluminescent single polymers with novel architecture. With respect to their topology and molecular design as well as emission color tuning, two-color white electroluminescent polymers with two emissive species can be linear (attachment of an orange emissive species on the main chain or side chain of a blue light-emitting polymer host), end-capped (attachment of an orange dye to the chain termini of a blue emissive polymer host), star-shaped or branched (attachment to an orange emissive species as the central core with blue light-emitting polymer host as the outer arms or branches).

Star-shaped molecules and polymers have recently attracted considerable attention because of their unusual molecular structures and opto-electronic properties.^[12] Highly branched and globular features can be expected to reduce or eliminate intermolecular interaction. In this Communication, we report our effort to design an efficient star-like white electroluminescent polymer with an orange emissive core and four blue light-emitting arms (see Fig. 1). By adjusting the content of the orange core, partial energy transfer and charge trapping from the blue arms to the orange core is realized and leads to emission from both the core and the arms. As a result, white EL with simultaneous blue emission ($\lambda_{\max} = 420$ nm/440 nm) and orange emission ($\lambda_{\max} = 562$ nm) is obtained. A single-layer device of this polymer emits white light with Commission Internationale de l'Éclairage (CIE) coordinates of (0.35, 0.39) and a luminous efficiency of 7.06 cd A⁻¹.

The chemical structures of the designed polymers are shown in Figure 1. Fluorene segments were selected as the arms because polyfluorene (PF), which can be regarded as the model compound for the fluorene segments, has a large bandgap, blue

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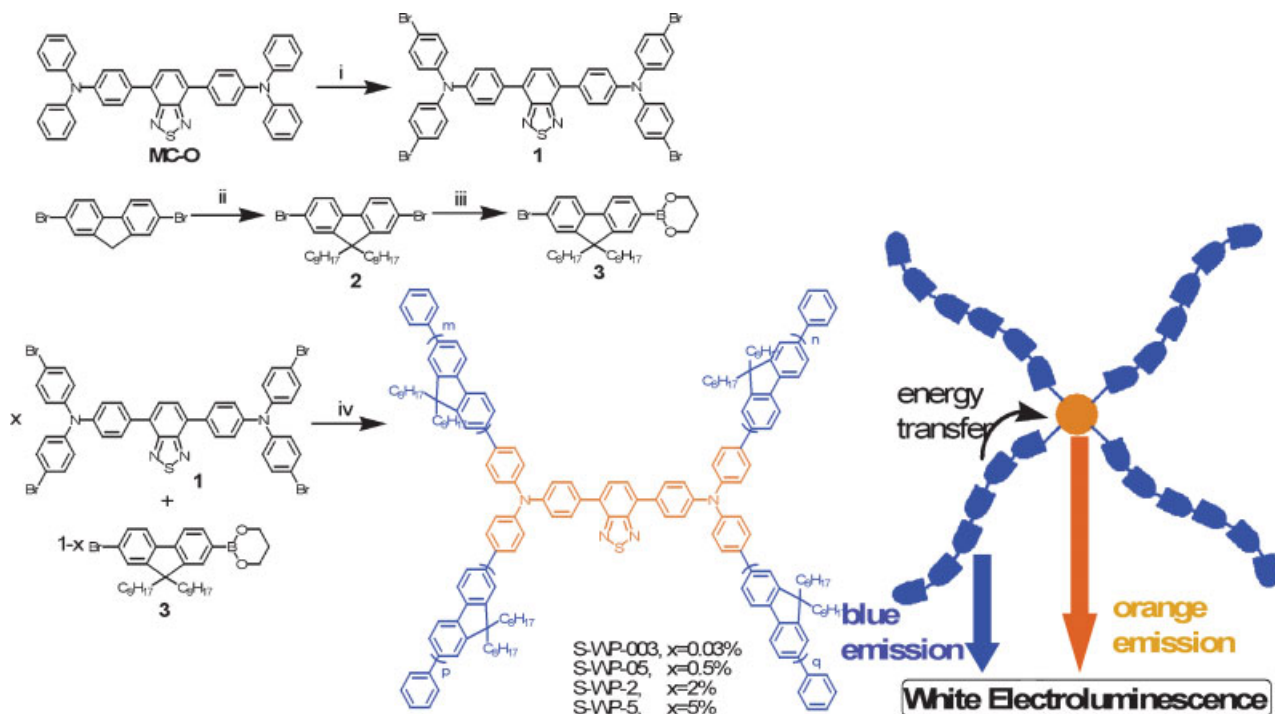


Figure 1. Schematic illustration, chemical structures, and synthetic routes of the polymers. Reagent and conditions: (i) $(n\text{-C}_4\text{H}_9)_4\text{NBr}_3$, CH_2Cl_2 ; (ii) $n\text{-C}_8\text{H}_{17}\text{Br}$, NaOH (aq. 50 wt%), $(n\text{-C}_4\text{H}_9)_4\text{NBr}$, toluene, 70°C ; (iii) a) BuLi , THF, -78°C , b) $\text{B}(\text{OCH}_3)_3$, c) HCl , d) $\text{HO}(\text{CH}_2)_3\text{OH}$, toluene, reflux; (iv) $\text{Pd}(\text{PPh}_3)_4$, toluene, K_2CO_3 (aq., 2 M), Aliquat 336, 90°C .

emission, high PL and EL efficiencies, and good charge-carrier transport properties.^[13] A 4,7-(4-(diphenylamino)phenyl)-2,1,3-benzothiadiazole (TPABT) unit (model compound: MC-O) was chosen as the core because of its orange emission with high PL quantum efficiency.^[14] The contents of the orange core in these polymers are adjusted from 0.03 mol% to 5 mol% to tune the relative intensities of the blue emission and orange emission.

Figure 1 shows the synthetic routes towards the monomers and the polymers. Bromination of the model compound with $(n\text{-C}_4\text{H}_9)_4\text{NBr}_3$ afforded the key orange monomer 1. The star-shaped polymers were synthesized by a one-pot Suzuki polycondensation with an A_4 type monomer (3) and an AB type monomer (1), according to a procedure described in the literature.^[12] After polymerization, these polymers were end-capped with phenyl groups. Because the polymer was prepared by polycondensation, the length of the arms in the star polymers should not be uniform. During the polymerization, because of the very low feed ratio of the A_4 type monomer, linear polyfluorene would inevitably form and intermix with the star polymers. The proportion of the linear component is expected to be reduced by using iodo-substituted A_4 monomer instead of bromo-substituted A_4 monomer to carry out the “ A_4+AB ” polymerization in the future.^[12a] All of the polymers are soluble in common organic solvents, for example, toluene, chloroform, and THF. Their number-average molecular weights (M_n), as determined by gel permeation chromatography (GPC) with

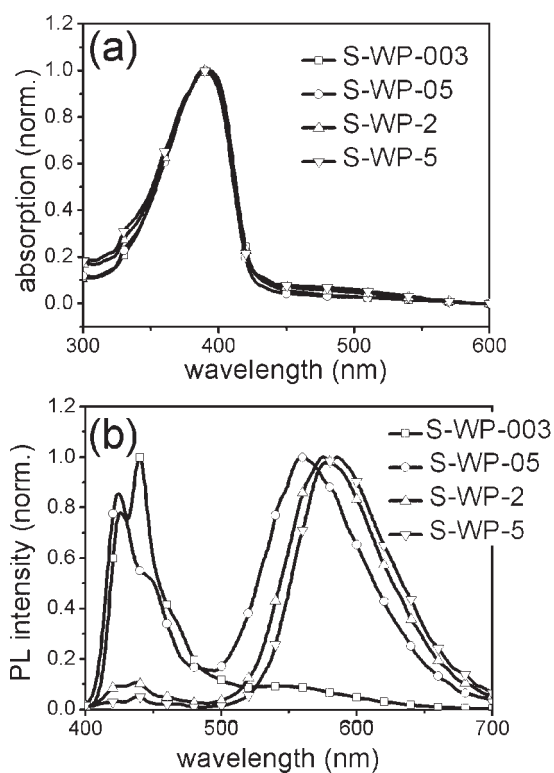


Figure 2. a) Absorption, and b) PL spectra of the polymers in solid films.

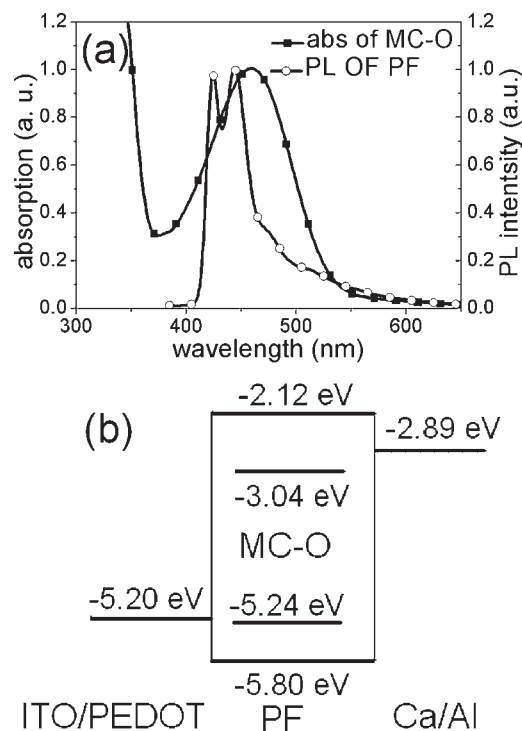


Figure 3. a) Absorption spectrum of MC-O and emission spectrum of PF. b) Lowest unoccupied and highest occupied molecular orbital (LUMO and HOMO) energy levels of MC-O and PF.

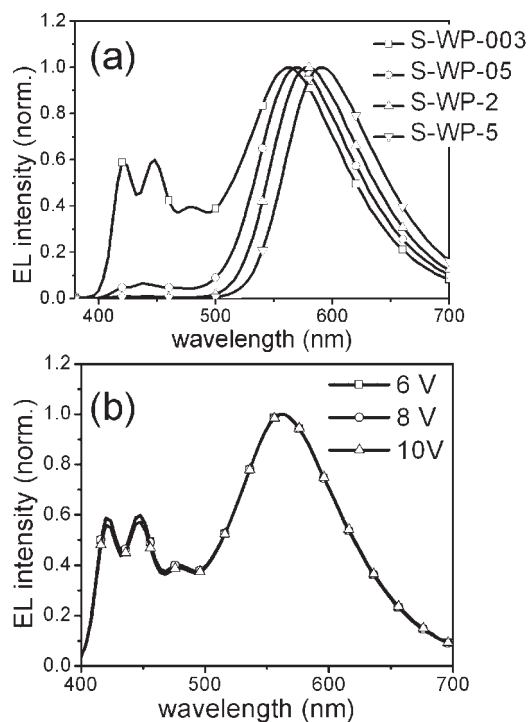


Figure 4. a) EL spectra of the devices of the polymers. b) EL spectra of the device based on S-WP-003 under different bias voltages.

polystyrene as standards, ranged from 14000 to 27900, with the polydispersity index (PDI) ranging from 2.17 to 2.53.

The absorption spectra of the polymers in solid film are shown in Figure 2a. S-WP-2 and S-WP-5 (star-like white polymer S-WP-*x*, where *x* denotes the content of the orange TPABT unit in mol%) both exhibit a very weak absorption band at ca. 460 nm and a strong absorption band at 390 nm. The former is assigned to the TPABT unit^[14] and the latter is attributed to the fluorene segments^[13] of both the linear polyfluorene and the arms of the star polymer. In the absorption spectra of S-WP-003 and S-WP-05, the absorption band at 460 nm disappears because of the low content of TPABT core unit. Figure 2b shows the PL spectra of the polymers in solid films with an excitation wavelength at 370 nm. The PL spectrum of S-WP-003 exhibits dominant blue emission at 420 nm/440 nm from the fluorene segments (of both the linear polyfluorene and the arms of the star polymer) and weak orange emission at 560 nm from the TPABT core unit. The orange emission band

comes from the Förster energy transfer from the fluorene segments to the TPABT core unit because of the overlap of the emission spectrum of polyfluorene and the absorption spectrum of the model compound (see Fig. 3a). When the content of TPABT core unit increases to 0.5 mol%, the PL spectrum of S-WP-05 exhibits simultaneous blue emission and orange emission with comparable intensity. The CIE coordinates calculated from the PL spectra of S-WP-05 are (0.35, 0.38), which are very close to the values of standard white emission, (0.33, 0.33), indicating that the PL of S-WP-05 in solid film is white. When the TPABT content further increases to 2 mol% and 5 mol%, the PL spectra of S-WP-2 and S-WP-5 exhibit dominant orange emission from the TPABT unit and negligible blue emission from the fluorene segments. With the increase of TPABT content, the intensity of the orange emission increases and the intensity of the blue emission decreases in the PL spectra owing to the more and more complete energy transfer from the fluorene segments to the TPABT unit.

Table 1. EL performance of the polymer-based devices.

Polymer	Turn-on voltage	Luminous efficiency	Power efficiency	Maximum brightness	λ_{\max}	CIE coordinates (<i>x</i> , <i>y</i>)
	[V]	[cd A ⁻¹]	[lm W ⁻¹]	[cd m ⁻²]	[nm]	
S-WP-003	3.5	7.06	4.43	17442	420/440/562	(0.35, 0.39)
S-WP-05	4.5	4.62	2.42	13200	568	(0.46, 0.49)
S-WP-2	7.0	1.56	0.53	3400	580	(0.51, 0.47)
S-WP-5	9.0	1.23	0.31	2670	588	(0.55, 0.43)

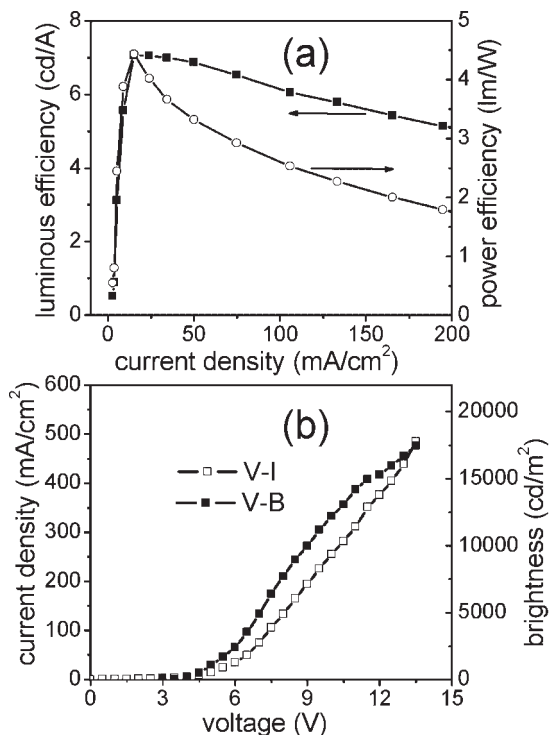


Figure 5. a) Dependence of the luminous efficiency and power efficiency on the current density of the device based on S-WP-003. b) Voltage–current density–brightness curves of the device based on S-WP-003.

To investigate the EL properties of these polymers, single-layer devices were fabricated with the configuration of indium tin oxide (ITO)/poly(ethylenedioxythiophene):poly(styrene sulfonic acid) (PEDOT:PSS) (40 nm)/polymer (90 nm)/Ca (10 nm)/Al (100 nm). Their EL spectra are shown in Figure 4a, and the corresponding CIE coordinates are listed in Table 1. The EL spectrum of S-WP-003 exhibits simultaneous blue emission at 420 nm/440 nm from the fluorene segments and orange emission at 562 nm from the TPABT core unit. Comparison of the EL spectra and PL spectra of S-WP-003 indicates that the orange emission band is much stronger in the EL spectra compare to in the PL spectra. This is attributed to the charge-trapping effect of the TPABT unit in the EL process because the lowest unoccupied molecular orbital (LUMO) and highest occupied molecular orbital (HOMO) energy levels of MC-O lie between those of PF (see Fig. 3b).^[15,16] In the EL spectra of S-WP-003, the intensities of the blue emission band and the orange emission band are comparable, leading to white emission with CIE coordinates of (0.35, 0.39) and a color rendering index of 65. The CIE coordinates are close to standard white emission, (0.33, 0.33). Moreover, its EL spectral shape keeps unchanged when the bias increases from 6 to 10 V. When the TPABT content increases, on the one hand, the blue emission band in the EL spectra becomes very weak in the case of S-WP-05 and becomes negligible in the cases of S-WP-2 and S-WP-5 because of the more and more complete energy transfer and charge trapping from the blue emissive fluorene segments to the orange emissive TPABT unit. On the other hand, the maxi-

imum of the orange emission band red-shifts from 568 nm with S-WP-05 to 580 nm with S-WP-2 and to 588 nm with S-WP-5. This red-shift is due to the interaction of the orange core unit.^[17]

The EL performance of the devices is listed in Table 1. The device based on S-WP-003 emits white light with CIE coordinates of (0.35, 0.39), has a turn-on voltage of 3.0 V, luminous efficiency of 7.06 cd A⁻¹, power efficiency of 4.43 lm W⁻¹, and a maximum brightness of 17442 cd m⁻². This performance is comparable to that of previously reported white electroluminescent linear polymers in literature.^[5–11] Figure 5 shows the dependence of the luminous efficiency and power efficiency on the current density as well as the voltage–current density–brightness curve of the device based on S-WP-003. With the increase of the orange core unit content, the turn-on voltages of the resulting devices increase but the luminous efficiencies and power efficiencies decrease. The increased turn-on voltage is attributed to the charge-trapping effect of the orange TPABT core unit.^[16] Because the HOMO of the TPABT core is higher than that of the fluorene segments and the LUMO of the TPABT core is lower than that of the fluorene segments, the TPABT core can trap both electrons and holes. At low driving voltage, a small amount of electrons and holes injected from two electrodes should be trapped by the TPABT units near the two electrodes. They are confined in these regions and cannot migrate to recombine with opposite charge carriers. Hence, higher voltage is required for the recombination of electrons and holes and for light emission. The decreasing EL efficiencies with increasing TPABT content are probably due to concentration quenching of the orange TPABT unit.

In summary, by using a one-pot “A₄ + AB” type Suzuki polycondensation, we have succeeded in developing star-like white electroluminescent polymers with a TPABT unit as the orange emissive core and fluorene segments as the blue emissive arms. The resulting polymer with an orange core unit content of 0.5 mol% exhibits white PL. For the polymer with an orange core unit content of 0.03 mol%, white EL is observed with simultaneous blue emission ($\lambda_{\text{max}} = 420 \text{ nm}/440 \text{ nm}$) from the fluorene segments and orange emission ($\lambda_{\text{max}} = 562 \text{ nm}$) from the TPABT unit. Its single-layer device (ITO/PEDOT/polymer/Ca/Al) emits white light with CIE coordinates of (0.35, 0.39), a turn-on voltage of 3.5 V, luminous efficiency of 7.06 cd A⁻¹, and a power efficiency of 4.43 lm W⁻¹.

Experimental

Synthesis of 4,7-bis(4-(di(4-bromophenyl)amino)phenyl)-2,1,3-benzothiadiazole (1): (n-C₄H₉)₄NBr₃ (2.41 g, 5.0 mmol) was added to a solution of 4,7-bis(4-(diphenylamino)phenyl)-2,1,3-benzothiadiazole (MC-O; 0.63 g, 1.0 mmol) in CH₂Cl₂ (30 mL). The resulting mixture was kept stirred at room temperature for 3 h. After workup, the mixture was poured into aqueous NaHSO₃. The organic layer was washed with water and then dried with anhydrous Na₂SO₄. After filtration and evaporation of the solvent, the residue was purified by silica column chromatography to give the title compound as an orange-yellow solid. Yield: 0.86 g (92%). ¹H NMR (CDCl₃, 300 MHz) δ (ppm): 7.94 (d, 4H), 7.79 (s, 2H), 7.43 (d, 8H), 7.23 (d, 4H), 7.08 (d, 8H). Anal. calcd. for C₄₂H₂₆Br₄N₄S: C, 53.76; H, 2.79; N, 5.97. Found: C, 53.98; H, 2.62; N, 5.82

Synthesis of 2-bromo-7-(trimethyleneborate)-9,9-dioctylfluorene (3): To a solution of 2,7-dibromo-9,9-dioctylfluorene (2) (15.54 g, 28.3 mmol) in dry THF (250 mL) at -78°C was added ($n\text{-C}_4\text{H}_9$)Li (17.7 mL, 1.6 M solution in hexane, 28.3 mmol). After stirring for 1 h, trimethylborate (4.2 mL, 37.1 mmol) was added and the mixture was kept stirred for another 24 h, followed by the addition of 2 M hydrochloric acid (100 mL). The mixture was extracted with ether and the combined extracts were evaporated to give a white solid, 7-bromo-9,9-dioctylfluorene-2-yl boric acid. The boric acid was mixed with 1,3-propanediol (2.5 mL, 34.5 mmol) and toluene (150 mL), and the resulting mixture was refluxed overnight. Evaporation of solvent under reduced pressure gave the crude product, which was recrystallized in ethanol two times to afford the title compound as a white solid. Yield: 5.81 g (37%). $^1\text{H NMR}$ (CDCl_3 , 300 MHz) δ (ppm): 7.79 (d, 1H), 7.74 (s, 1H), 7.67 (d, 1H), 7.59 (d, 1H), 7.46 (m, 2H), 4.24 (t, 4H), 2.13 (m, 2H), 1.96 (m, 4H), 1.25–1.06 (m, 20H), 0.83 (t, 6H), 0.59 (br, 4H). Anal. calcd. for $\text{C}_{32}\text{H}_{46}\text{BBrO}_2$: C, 69.45; H, 8.38. Found: C, 69.34; H, 8.00.

General Procedure of Suzuki Polymerization: To a mixture of 1, 3, Aliquat 336 (0.10 g, 0.25 mmol) and $\text{Pd}(\text{PPh}_3)_4$ (0.011 g, 0.01 mmol) under an argon atmosphere was added 2 M aqueous K_2CO_3 (2.5 mL) and toluene (6 mL). The mixture was stirred at 90°C for 24 h. Then, the polymer was end-capped by adding 9,9-dioctyl-2,7-bis(trimethyleneborate)fluorene (3 mL 0.03 M solution in toluene, 0.09 mmol) followed by stirring for 6 h, and by adding bromobenzene (1.0 mL 1.0 M solution in toluene, 1.0 mmol) followed by stirring for 8 h. After workup, the mixture was poured into methanol. The precipitate was collected by filtration, dried, and then dissolved in dichloromethane. The solution was washed with water and dried over anhydrous Na_2SO_4 . After most of the solvent had been removed, the residue was poured into stirred methanol to give polymer fiber. The polymer was further purified by extracting with acetone for 24 h. The reprecipitation procedure in dichloromethane/methanol was then repeated several times. The final product was obtained after drying in vacuum with a yield of 45–60%.

S-WP-003: light-orange fiber. 1 (0.6 mL 5×10^{-4} M solution in toluene, 3×10^{-4} mmol) and 3 (0.5534 g, 1.0000 mmol) were used in the polymerization. $^1\text{H NMR}$ (300 MHz, CDCl_3) δ (ppm): 7.88 (d, 2H), 7.73 (br, 4H), 2.10 (br, 4H), 1.14 (br, 24H), 0.81 (t, 6H). Element Anal. Found: C, 89.15, H, 9.66. GPC: $M_n = 27900$, $\text{PDI} = 2.17$.

S-WP-05: light-orange fiber. 1 (0.0047 g, 0.005 mmol) and 3 (0.5501 g, 0.995 mmol) were used in the polymerization. $^1\text{H NMR}$ (300 MHz, CDCl_3) δ (ppm): 7.88 (d, 2H), 7.73 (br, 4H), 2.10 (br, 4H), 1.14 (br, 24H), 0.81 (t, 6H). Element Anal. Found: C, 89.11, H, 9.21. GPC: $M_n = 23200$, $\text{PDI} = 2.34$.

S-WP-2: orange fiber. 1 (0.0188 g, 0.02 mmol) and 3 (0.5427 g, 0.98 mmol) were used in the polymerization. $^1\text{H NMR}$ (300 MHz, CDCl_3) δ (ppm): 7.88 (d, 2H), 7.73 (br, 4.48H), 7.53 (br, 0.30 H), 2.15 (br, 4H), 1.18 (br, 20H), 0.85 (t, 10H). Element Anal. Found: C, 88.12, H, 9.35, N, 0.18. GPC: $M_n = 16900$, $\text{PDI} = 2.53$.

S-WP-5: orange-red fiber. 1 (0.0466 g, 0.05 mmol) and 3 (0.5257 g, 0.95 mmol) were used in the polymerization. $^1\text{H NMR}$ (300 MHz, CDCl_3) δ (ppm): 8.01 (br, 0.28H), 7.88 (d, 2.04H), 7.73 (br, 4.65H), 7.53 (br, 0.35 H), 7.40 (br, 0.57 H), 2.15 (br, 4H), 1.17 (br, 20H), 0.84 (br, 10H). Element Anal. Found: C, 86.56, H, 9.06, N, 0.54. GPC: $M_n = 14000$, $\text{PDI} = 2.51$. The actual content of the core calculated from the elemental analysis result is 5.95 mol%.

Device Fabrication and Characterization: An ITO glass substrate was precleaned in an ultrasonic bath. After drying, a 40 nm thick layer of PEDOT:PSS was spin-coated onto the ITO glass substrate, followed by baking at 120°C for 15 min. The polymer layer (90 nm) was then spin-coated onto the PEDOT:PSS/ITO-coated glass substrate from a fresh toluene solution (15 mg mL^{-1}). Finally, a thin layer of Ca (10 nm) with Al (100 nm) capping layer was deposited through a shadow mask in a thermal evaporator with a pressure of 3×10^{-3} – 5×10^{-3} Pa. The active area of the device was 10 mm^2 . The EL spectra, CIE coordinates, current–voltage and brightness–voltage characteristics of the devices were measured with a Spectrascan PR650 spectrophotometer at the forward direction and a computer-controlled Keithley 2400 instrument.

All the fabrication and characterization of the device were carried out at ambient atmosphere.

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