Electro-Active Oligomers Containing Pendent Carbazolyl or Indolyl Groups as Host Materials for OLEDs

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ABSTRACT: Oligoethers containing electroactive carbazolyl, indolyl or 2-phenylindolyl fragments were synthesized and characterized by NMR spectroscopy, elemental analysis and gel permeation chromatography. The oligomers represent amorphous materials of high thermal stability with glass transition temperatures of 107–161°C. The electron photoemission spectra of layers of the synthesized oligomers showed ionization potentials of 5.9–5.95 eV. Some of the derivatives were tested as host materials in phosphorescent OLEDs with iridium(III) [bis(4,6-difluorophenyl)-pyridinato-N,C2′]picolinate as the guest. The device based on oligomer containing carbazolyl fragments exhibited the best overall performance with a turn-on voltage of 3.5 V, maximum power efficiency of 4.1 lm/W and maximum brightness of 937 cd/m². © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 122: 908–913, 2011

Key words: oxetane; electroactive material; polyether; ionization potential; host; light emitting diode

INTRODUCTION

The efficiencies of organic light-emitting devices (OLEDs) have advanced rapidly in recent years because of the development of efficient phosphorescent guest molecules containing transition metals.[1–3] In the phosphorescent devices, to reduce quenching associated with relatively long excited-state lifetimes of triplet emitters and triplet–triplet annihilation etc., triplet emitters of the metal complexes are normally used as emitting guests in a host material, and thus suitable host materials are of equal importance for the blue phosphorescent OLEDs. For electrophosphorescence from triplet guests, it is essential that the triplet level of the host be larger than that of the triplet emitter to prevent reverse energy transfer from the guest back to the host and to effectively confine triplet excitons on guest molecules.[4–6] It was reported earlier that carbazole- and indole-based derivatives demonstrate rather large triplet energies and are potential host materials for blue electrophosphorescent devices.[7–10] Here, we report new electroactive oligomers containing pendent carbazolyl, indolyl or 2-phenylindolyl fragments. The oligomeric hosts can be suitable for preparation of large area devices.

EXPERIMENTAL

Instrumentation

^{1}H NMR spectra were recorded using a Varian Unity Inova (300 MHz) apparatus. Mass spectra were obtained on a Waters ZQ 2000 spectrometer. FTIR spectra were recorded using a Perkin Elmer FTIR System. UV spectra were measured with a Spectronic Genesys™ 8 spectrometer. Fluorescence (FL) spectra were recorded with a MPF-4 spectrometer.

The molecular weights of polymers were determined by a gel permeation chromatography (GPC) system including GMH$_{HR}$-M columns and Bischoff LAMBDA 1000 detector. Polystyrene standards were used for calibration of the columns and chloroform was chosen as an eluent.

Differential scanning calorimetry (DSC) measurements were carried out using a Bruker Reflex II thermosystem. Thermogravimetric analysis (TGA) was performed on a Netzsch STA 409. The TGA and DSC curves were recorded in a nitrogen atmosphere at a heating rate of 10°C/min.

The ionization potentials of the layers of the compounds synthesized were measured by the electron photoemission method in air.[11,12] The samples for the ionization potential measurement were prepared as we described earlier.[13,14]

The devices were fabricated on glass substrates and consisted of multiple organic layers sandwiched between the bottom indium tin oxide (ITO) anode and the top metal cathode (Al). The device structure used was ITO/PEDOT:PSS (ca. 300 Å)/**7** or **9** doped with 16 wt % of **FIrpic** (250 Å)/LiF (5 Å)/Al (1,500 Å), where the conducting polymer poly(3,4-ethylenedioxythiophene) : poly(styrenesulfonate) (PEDOT : PSS) was used as the hole-injection layer, **7** or **9** doped with the blue phosphorescent iridium(III) [bis(4,6-difluorophenyl)-pyridinato-*N*,*C2′*]picolinate (**FIrpic**) was used as the emitting layer and LiF was used as the electron-injection layer.

The luminance and CIE chromatic coordinates of the resulting OLEDs were measured using a Minolta CS-100A luminance meter. A Keithley 2400 electrometer was used to measure the current-voltage (I-V) characteristics. All the devices were characterized without encapsulation and all the measurements were carried out under ambient condition. The emission area of all the devices was 25 mm^2 and only the luminance in the forward direction was measured.[15]

Materials

9H-Carbazole (**1**), 2-pheny-1H-indole (**2**), 1H-indole (**3**), potassium hydroxide, potassium carbonate, tetrabutylammonium hydrogen sulphate (TBAS) and boron trifluoride diethyl etherate (BF$_3$·O(C$_2$H$_5$)$_2$) were purchased from Aldrich and used as received.

3,3′-Bis(carbazol-9-ylmethyl)oxetane (**4**) was prepared by the reaction of 3,3′-bis(chloromethyl)oxetane with an excess of 9H-carbazole (**1**) under basic conditions in the presence of a phase transfer catalyst -TBAS. Ten grams (60 mmol) of 9H-carbazole and 3.1 g (20 mmol) of 3,3′-bis(chloromethyl)oxetane were heated to reflux in 100 mL of ethyl methyl ketone. Then 8.25 g (60 mmol) of powdered potassium carbonate, 10 g (179 mmol) of potassium hydroxide and a catalytic amount of TBAS were added to the mixture, and it was refluxed for 24 h. Then the cooled mixture was decanted and the new portion of inorganic materials was added to the solution, and the mixture was again refluxed for 24 h. After thin layer chromatography (TLC) control the mixture was filtered, the solvent was evaporated and the product was purified by column chromatography with silica gel using hexane/ethyl acetate (vol. ratio 5 : 1) as an eluent. After crystallization from the eluent yield of the product was 65% (5.34 g). Mp: 192 −194°C. MS (APCI$^+$, 20 V), *m/z*: 439.5 ([M+Na]$^+$, 60%), 417.5 ([M+H]$^+$, 45%).

^{1}H NMR (300 MHz, CDCl$_3$, δ, ppm): 8.12 (d, 4H, *J* = 7.5 Hz, Ar), 7.41 (t, 4H, *J* = 7.5 Hz, Ar), 7.3–7.22 (m, 8H, Ar), 4.67 (s, 4H, —N-CH$_2$—), 4.63 (s, 4H, —CH$_2$-O—). IR (KBr, cm^{-1}): 3052 (C—H, Ar); 2956, 2859 (C—H); 1625, 1594 (C—C, Ar); 1334, 1325 (C—N); 1229, 1154, 1058 (C—O—C); 746, 717 (CH=[CH). 3,3′-Bis(2-phenyindol-9-ylmethyl)oxetane (**5**) was prepared from 10 g (52 mmol) of 2-pheny-1H-indole (**2**) and 2.67 g (17 mmol) of 3,3′-bis(chloromethyl)oxetane in the presence potassium carbonate (7.14 g, 52 mmol), potassium hydroxide (8.7 g, 155 mmol) and a catalytic amount of phase transfer catalyst in the same way as monomer **4**. The compound was purified by column chromatography with silica gel using hexane/ethyl acetate (vol. ratio 5 : 1) as an eluent. Yield of the product was 45% (3.62 g). MS (APCI$^+$, 20 V), *m/z*: 469.5 ([M+H]$^+$, 80%), 208.4 (100%). ^{1}H NMR (300 MHz, CDCl$_3$, δ, ppm): 7.62 (d, 2H, *J* = 7.5 Hz, Ar), 7.41–7.32 (m, 6H, Ar), 7.29–7.03 (m, 10H, Ar), 6.55 (s, 2H, Ar), 4.2 (s, 4H, —N-CH$_2$—), 3.75 (s, 4H, —CH$_2$-O—).

IR (KBr, cm^{-1}): 3056 (C—H, Ar); 2965, 2935, 2876 (C—H); 1706, 1662, 1605, 1548 (C=C, Ar); 1342, 1315 (C—N); 1240, 1164, 1074 (C—O—C); 794, 769, 750, 701, 668 (CH=CH). 3,3′-Bis(indol-9-ylmethyl)oxetane (**6**) was prepared from 10 g (85 mmol) of 1H-indole (**3**) and 4.41 g (28 mmol) of 3,3′-bis(chloromethyl)oxetane in the presence potassium carbonate (11.8 g, 85 mmol), potassium hydroxide (14.3 g, 256 mmol) and a catalytic amount of phase transfer catalyst in the same way as monomer **4**. The compound was purified by column chromatography with silica gel using hexane/ethyl acetate (vol. ratio 5 : 1) as an eluent. After crystallization from methanol yield of the product was 53% (4.8 g). Mp: 120–122°C. MS (APCI$^+$, 20 V), *m/z*: 439.5 ([M+Na]$^+$, 60%%), 417.5 ([M+H]$^+$, 45%).

^{1}H NMR (300 MHz, CDCl$_3$, δ, ppm): 7.66–7.61 (m, 2H, Ar), 7.19–7.07 (m, 6H, Ar), 6.95 (d, 2H, *J* = 3.3 Hz, Ar), 6.57 (d, 2H, *J* = 3.3 Hz Ar), 4.59 (s, 4H, —N-CH$_2$—), 4.33 (s, 4H, —CH$_2$—O—). IR (KBr, cm^{-1}): 3119, 3101, 3058 (C—H, Ar); 2957, 2919, 2875 (C—H); 1609, 1574 (C=C, Ar); 1308 (C—N); 1270, 1242, 1090 (C—O—C); 766, 739, 724 (CH=CH).

Poly[3,3′-bis(carbazol-9-ylmethyl)oxetane] (**7**). 1 g (2.4 mmol) of 3,3′-bis(carbazol-9-ylmethyl)oxetane (**4**) was polymerized in 4.8 mL of 1,2-dichlorethane using 0.072 mmol of BF$_3$·O(C$_2$H$_5$)$_2$ as initiator. The polymerization mixture was stirred at 60°C under nitrogen for 24 h. After precipitation into methanol, the low molecular weight fractions of the polymer were removed by Soxhlet extraction of the crude product with methanol. Yield: 0.56 g (56%) of white amorphous powder. M_w = 2820, M_n = 1320.

^{1}H NMR (300 MHz, CDCl$_3$, δ, ppm): 8.22 (d, 4H, *J* = 7.5 Hz, Ar), 7.85–7.72 (m, 4H, Ar), 7.5 (t, 4H, *J* = 7.5 Hz, Ar), 7.26 (t, 4H, *J* = 7.5 Hz Ar), 4.81 (bs, 4H,
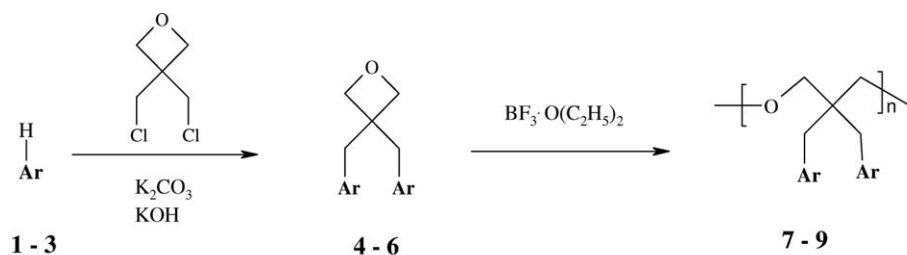
Scheme 1

—N—CH$_2$—), 4.53 (bs, 4H, —CH$_2$—O—). IR (KBr, cm^{-1}): 3571 (O—H); 3049 (C—H, Ar); 2874 (C—H); 1626, 1597 (C═C, Ar); 1326 (C—N); 1231, 1154, 1062 (C—O—C); 748, 722 (CH═[CH).

Poly[3,3′-bis(2-phenyindol-9-ylmethyl)oxetane] (**8**). 1 g (2.13 mmol) of 3,3′-bis(2-phenyindol-9-ylmethyl)-oxetane (**5**) was polymerized in 4.3 mL of 1,2-dichlorethane using 0.065 mmol of BF$_3$·O(C$_2$H$_5$)$_2$ as initiator. The polymerization mixture was stirred at 60°C under nitrogen for 24 h. After precipitation into methanol, the low molecular weight fractions of the polymer were removed by Soxhlet extraction of the crude product with methanol. Yield: 0.49 g (49%) of white amorphous powder. $M_w = 3410$, $M_n = 2210$.

^{1}H NMR (300 MHz, CDCl$_3$, δ, ppm): 7.6–6.4 (m, 20H, Ar), 4.2 (s, 4H, —N—CH$_2$—), 3.75 (s, 4H, -CH$_2$-O-). IR (KBr, cm^{-1}): 3452 (O—H); 3056, 3029 (C—H, Ar); 2960, 2926, 2878 (C—H); 1666, 1604, 1546 (C═C, Ar); 1343, 1315 (C—N); 1240, 1165, 1074 (C—O—C); 792, 750, 701, 666 (CH═[CH).

Poly[3,3′-bis(indol-9-ylmethyl)oxetane] (**9**). 1 g (3.16 mmol) of 3,3′-bis(indol-9-ylmethyl)oxetane (**6**) was polymerized in 4.3 mL of 1,2-dichlorethane using 0.097 mmol of BF$_3$·O(C$_2$H$_5$)$_2$ as initiator. The polymerization mixture was stirred at 60°C under nitrogen for 24 h. After precipitation into methanol, the low molecular weight fractions of the polymer were removed by Soxhlet extraction of the crude product with methanol. Yield: 0.27 g (27%) of white amorphous powder. $M_w = 2380$, $M_n = 8730$.

^{1}H NMR (300 MHz, CDCl$_3$, δ, ppm): 7.66–7.61 (m, 2H, Ar), 7.19–7.07 (m, 6H, Ar), 6.96 (d, 2H, $J = 3.3$ Hz, Ar), 6.58 (d, 2H, $J = 3.3$ Hz Ar), 4.6 (s, 4H, —N—CH$_2$—), 4.35 (s, 4H, —CH$_2$—O—). IR (KBr, cm^{-1}): 3378 (O—H); 3050 (C—H, Ar); 2933, 2872

(C—H); 1610 (C═C, Ar); 1310 (C—N); 1268, 1204, 1086 (C—O—C); 766, 740 (CH═[CH).

RESULTS AND DISCUSSION

The synthesis of derivatives containing carbazol-9-yl or indol-1-yl fragments (**4 - 9**) was carried out by a multi-step synthetic route as shown in Scheme 1. Monomers **4–6** were prepared by nucleophilic substitution reaction of 3,3′-bis(chloromethyl)oxetane with an excess of a heterocyclic compound (9H-carbazole, 2-pheny-1H-indole or 1H-indole) under basic conditions using a phase transfer catalyst- TBAS. Polymers **7-9** were obtained by cationic polymerization of the corresponding monomers (**4–6**) in 1,2-dichloretane solutions using BF$_3$·O(C$_2$H$_5$)$_2$ as an initiator. Low-molar-mass fractions of the products of polymerizations were removed by Soxhlet extraction of the raw polymers with methanol.

Cationic ring-opening polymerization of the oxetane-based monomers **4–6** was well confirmed by IR spectroscopy. The oxetane ring shows a characteristic IR band at ca 980 cm^{-1} (asymmetric stretch vibration). During polymerization, the oxetane band disappears while the spectra of polymers demonstrate broad signals in the area between 1170 and 1020 cm^{-1} due to the formation of linear polyether. An additional broad band in the area between 3300 and 3500 cm^{-1} was detected after the polymerization, which is attributed to hydroxy end groups of the polymers.

The newly synthesized derivatives were identified by mass spectrometry (for monomers), IR- and ^{1}H NMR spectroscopy. The data were found to be in good agreement with the proposed structures. All

TABLE I
Molecular Weights and PDI of the Polymers Synthesized

Polymer	M_n	M_w	PDI
7	1940	3690	2.1
8	2260	3620	1.6
9	2410	8440	3.5

the synthesized materials except polymer **7** were good soluble in common organic solvents, such as acetone, chloroform, and tetrahydofuran (THF) at room temperature. Transparent thin films of these materials could be prepared by spin coating from solutions. Thin films of the polymer **7** could be prepared from its dichlorobenzene or DMSO solutions.

Molecular weights and polydispersities of the synthesized derivatives were estimated by GPC. The number-average molecular weights (M_n), weight-average molecular weights (M_w) as well as the polydispersity indexes (PDI) are summarized in Table I. It was established that cationic polymerization of the monomers yielded products with rather low molecular weights, however the materials demonstrate good film forming properties and are promising derivatives for preparation of electro-active layers. Earlier, we have studied in detail syntheses and properties of other carbazole containing polyoxiranes and polyoxetanes.[16,17] We have also observed that oligomers having 4–6 monomeric units demonstrate better film forming properties than that of their polymers. Electro-active films of the oligomers could be applied in optoelectronic devices.

The molecular weights of the oligomers **7–9** depend slightly on the nature of electrophores attached to the polyether chain. The monomer **6** containing indol-1-yl fragments yielded oligomers with higher molecular weight than monomers **4** and **5**. The different polymerization degree of these oligomers could probably be explained by the different steric hindrance of the substituents attached to oxetane ring.

The behavior under heating of the polymers **7-9** was studied by DSC and TGA. All these materials demonstrate rather high thermal stability. The tem-

TABLE II
UV Absorption and FL Maxima of the Synthesized Derivatives[a]

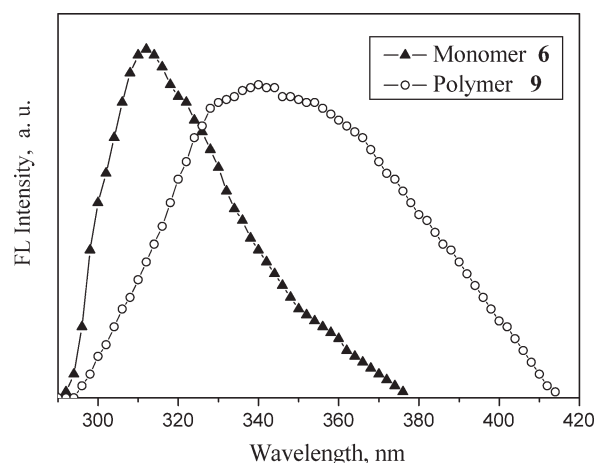| Compound	UV: λ_{max} (nm)	FL: λ_{max} (nm)
4	262, 292, 328, 342	362
5	296	368
6	274	314
7	260, 294, 342	364
8	296	372
9	276	341

[a]Excitation wavelength 280 nm.

Figure 1 Comparison of FL spectrum of dilute THF solutions of oligomer **9** with that of its monomer **6**.

peratures at which initial loss of mass ($T_{5\%}$) was observed are 350°C for **7,** 345°C for **8,** and 290°C for **9**. The TGA measurements were also done for mixtures of the oligomers **7–9** containing 16 wt % of **FIrpic**. It was observed that this amount of quest material has not an evident influence on thermal degradation of the hosts. The values of $T_{5\%}$ of the mixtures were very close to those of pure oligomers.

DSC measurements confirmed that the oligomers are amorphous materials. When samples of the materials were subjected to DSC heating scans the glass-transitions were observed in the range of 107–161°C and no peaks due to crystallisation and melting appeared on further cooling and heating cycles between −30°C and 250°C.

UV absorption and FL spectra of dilute solutions of the synthesized oligomers were recorded. The values λ_{max} are presented in Table II. For comparison, the corresponding spectral data of the monomers **4–6** are presented in the Table. The electronic absorption energies of the oligomers **7–9** are comparable,
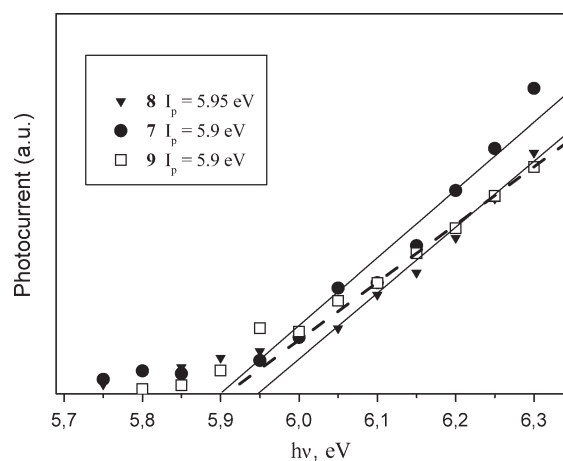

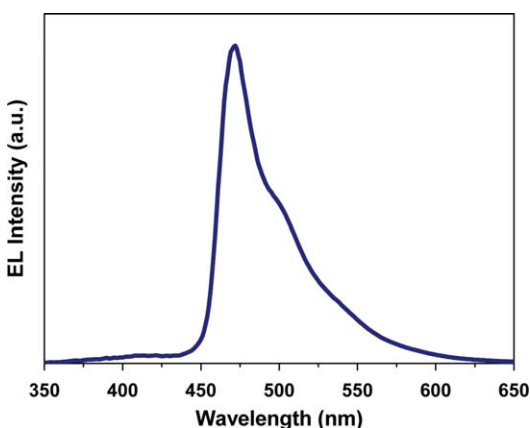**Figure 2** Electron photoemission spectra of layers of polymers **7–9**.

Figure 3 Electroluminescent spectrum of the oligomer 7-based device. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

and the λ_{max} values are in the range of 230–348 nm. FL maxima of the polymers appear in the region of wavelengths from 341 to 372 nm.

The comparison of the UV absorption and FL spectra of the oligomers with those of the corresponding monomers did not reveal in most of the cases any considerable bathochromic shift of the spectra of oligomers with respect to those of the monomers. Only the FL spectrum of oligomer 9 containing indolyl fragments is considerably red shifted in respect of the spectra of its monomer 6. The FL spectra of the latter derivatives are presented in Figure 1. This observation demonstrates that considerable intramolecular interaction is observed in solutions of the oligomer 9.

Ionization potentials (I_p) of thin amorphous layers of the oligomers were determined from electron photoemission spectra of the layers. The spectra as well as the values of I_p are presented in Figure 2. It was observed that Ip of these oligomers are rather close and range from 5.9 to 5.95 eV. These values are very similar to that of other derivatives containing unsubstituted carbazolyl or indolyl fragments.[17,18] The Ip level suggests that an additional hole inject-

ing layer would be necessary for application of the oligomers 7–9 as host materials for phosphorescent OLEDs.

To evaluate the performance of the oligomers 7–9 as hosts, phosphorescent OLEDs were fabricated by using blue emitter- **FIrpic** as the guest. However the oligomer 8 had a bad compatibility with 12–22 wt % of the quest in solid state (thin films). The films were not homogeneous and were not suitable for preparation of OLEDs.

We have used 12, 14, 16, 18, 20, and 22 wt % of **FIrpic** in the host materials and tested the mixtures for preparation of the OLEDs. The structure of the multilayer devices is described in Experimental part. It was observed that the OLEDs containing 16 wt % of **FIrpic** in the host materials demonstrated the best performance and the results will be presented. EL spectrum of the oligomer 7-based device is shown in Figure 3 as an Example. It is evident that the device shows emission from **FIrpic**, indicating the effective confinement of triplet excitons on the triplet dopants and the sufficient injection of both holes and electrons into the emitting layer. CIE chromaticity coordinates of the OLEDs were (0.19, 0.31) for material 7-based device and (0.19, 0.34) for 9-based device at 8 V.

Figure 4 shows the current density–voltage–luminance characteristics and curves of power efficiency versus voltage for the devices. These devices exhibit turn-on voltages of 3.5−7 V and power efficiencies of 1.9−4.1 lm/W. The device using carbazole-based polymer 7 as the host exhibits better overall performance with low turn-on voltage of 3.5 V, maximum brightness of 937 cd/m^2 and maximal power efficiency of 4.1 lm/W. For the technically important brightness of 100 cd/m^2, the efficiency of the device containing 7 was above 3.5 lm/W. These findings look rather promising when compared to the similar devices containing poly(9-vinylcarbazole) as a host.[19] It should be mentioned that these characteristics were obtained for nonoptimized test devices under ordinary laboratory conditions.

In conclusion, oligoethers containing pendent electroactive carbazolyl, 2-phenylindolyl or indolyl
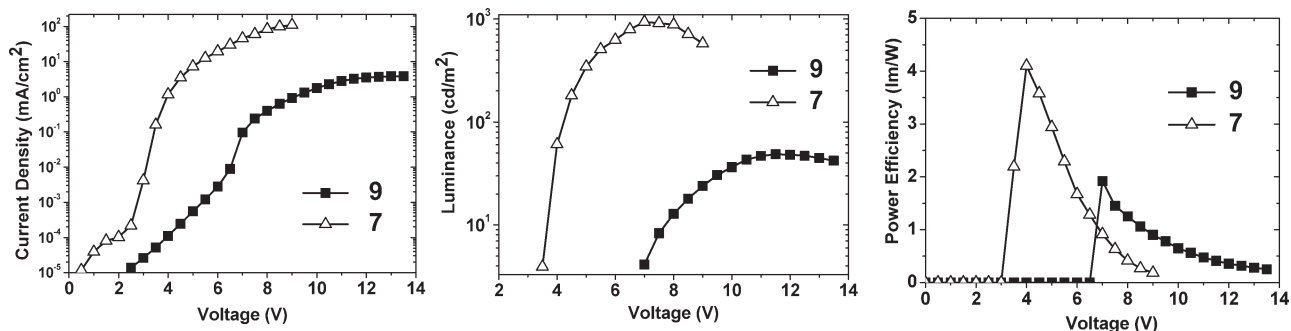

Figure 4 OLED characteristics of devices using 7 or 9 as the host: a) Current–voltage characteristics; b) Luminance–voltage characteristics and c) Power efficiencies versus voltage.

moieties were synthesized from the corresponding oxetane monomers by cationic polymerization in solution. The amorphous oligomers show high thermal stability with glass transition temperatures of 107–161°C. Some of the materials were tested as hosts in phosphorescent OLEDs with iridium(III) [bis(4,6-difluorophenyl)-pyridinato-*N*,*C2'*]picolinate as the guest. The device with the host oligomer containing pendent carbazolyl fragments exhibited the best overall performance (turn-on voltage: 3.5 V; maximum power efficiency: 4.1 lm/W; maximum brightness: 937 cd/m^2).

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