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Solution-processable and thermal-stable triphenylamine-based dendrimers with truxene cores as hole-transporting materials for organic light-emitting devices

Zhongfeng Yang, Bin Xu, Jiating He, Lili Xue, Qing Guo, Haijian Xia, Wenjing Tian *

State Key Laboratory of Supramolecular Structure and Materials, Jilin University, Changchun 130012, China

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ABSTRACT

Two solution processable π-conjugated triphenylamine-based dendrimers, Tr-TPA3 and Tr-TPA9 were served as hole-transporting materials (HTMs) for organic light-emitting devices (OLEDs). The two dendrimers exhibit similar absorption and emission behaviors in solutions and thin films, which demonstrate that these dendrimers can form amorphous states in their films. The dendrimers showed excellent solubility, which are soluble in common organic solvents such as chloroform, tetrahydrofuran, and 1,1,2,2-tetrachloroethane, high thermal stability with high glass-transition temperature (T_g) of 115 °C for Tr-TPA3 and 140 °C for Tr-TPA9, high the highest unoccupied molecular orbital (HOMO) energy level (−5.12 eV for Tr-TPA3 and −4.95 eV for Tr-TPA9, respectively) and good film forming property. When we employed these dendrimers as hole transport layer (HTL) in tris-(8-hydroxyquinoline) aluminum (Alq_3)-emitting electroluminescence (EL) devices, the Tr-TPA9-based double-layer device exhibited the turn-on voltage of 2.5 V, the maximum luminance of about 11,058 cd m^{-2} and the maximum current efficiency of 4.01 cd A^{-1}. The comparison of the properties between the EL devices with dendrimers as HTL and the EL device with 1,4-bis(1-naphthylphenylamino)biphenyl (NPB) as HTL indicated that this series of dendrimers can be good candidates for HTM in OLEDs.

1. Introduction

Organic light-emitting devices (OLEDs) have attracted much attention because of their potential applications in full-color flat-panel displays and solid-state lighting [1–4]. OLEDs with high performance can be effectively realized by fabricating multilayer device using transporting and injection layers in order to balance the injection and transport of both holes and electrons [5]. Small organic molecules containing triphenylamine group, such as N,N′-di(1-naphthyl)-N,N′-diphenyl-[1,1′-biphenyl]-4,4′-diamine (NPB) and those of starburst triphenylamine derivatives are widely used as hole-transporting materials (HTMs) due to their high hole mobility, electron donating ability and good optoelectronic properties [5–12]. But because of the poor film forming property from solution of small molecules, these HTM must be used in OLEDs by vacuum evaporation which is not benefit for commercial applications [13,14]. In contrast, solution processing methods, such as spin-coating and ink-jet printing, are relatively inexpensive and can be utilized for the preparation of large-area displays [15]. Hence, solution-processable materials in OLEDs should be an attractive choice when good performance and film forming property can also be simultaneously obtained [16,17]. For example, Promarak et al. [18] reported a solution-processable hole transport material in a double-layer OLED which showed a bright green emission from Alq_3 of 6500 cd m^{-2} at 15 V and a low turn-on voltage of 3.8 V with T_g of 70 °C. Zhou et al. [19] have demonstrated a solution-processable binaph-

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* Corresponding author. Tel.: +86 431 85166368; fax: +86 431 8519 3421.

E-mail address: wjtian@jlu.edu.cn (W. Tian).

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thyl-containing molecule which was used as light emitting material in OLED with a turn-on voltage of 2.2 V, a maximum luminance of 8315 cd m^{-2}, and a maximum luminescence efficiency of 1.95 cd A^{-1}.

One of the significant properties of the electroluminescence (EL) devices is the durability. The degradation mechanisms in the OLEDs are not yet fully understood, but several studies indicate that one reason of the degradation is the morphological changes of the organic layers [20,21], such as dimerization and crystallization which seem to be caused by joule heating during device operation [22]. The organic materials tend to dimerize or crystallize when they are allowed to stand at ambient temperature or heated above their glass-transition temperature (T_g) [23]. Thus organic materials with high T_g is propitious to realize high stability OLEDs [23–27]. In addition, it was found that the lower the energy barrier of hole injection at the interface of the ITO/hole-transporting layer (HTL), the lower joule heat produced at the interface [6,28]. For this reason, the energy barrier between the ITO/HTL interface should be as small as possible in order to implement high stable OLEDs, which means the HTM should have a proper the highest unoccupied molecular orbital (HOMO) energy level for hole injection [6]. Therefore, the ideal HTM should have excellent solubility, good film forming property from solution, high thermal stability, proper HOMO energy level for efficient hole injection and high mobility.

Along this respect, in this paper, we reported two solution-processable and thermal-stable hole-transporting triphenylamine-based dendrimers whose structures are shown in Scheme 1 [29]. As compared to those of reported cases, the dendrimers showed high thermal stability with high T_g of 115 °C for Tr-TPA3 and 140 °C for Tr-TPA9 and the Tr-TPA9-based device exhibited the turn-on voltage of 2.5 V, the maximum luminance of about 11,058 cd m^{-2} and the maximum current efficiency of 4.01 cd A^{-1}. The dendrimers also exhibited excellent solubility, good film forming property, and proper HOMO energy level for hole injection.

2. Experimental

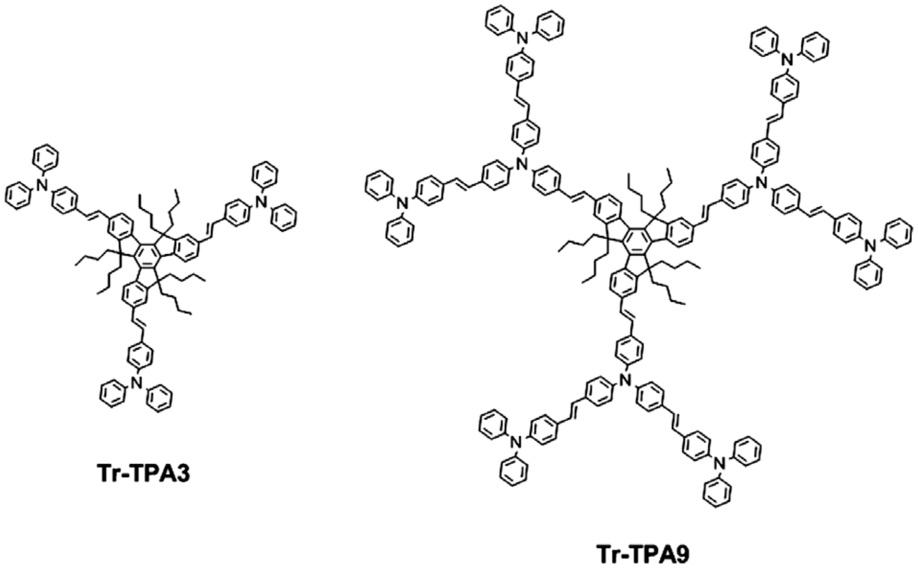
2.1. Materials and measurements

The synthesis of two truxene-centered dendric triphenylamine derivatives Tr-TPA3 and Tr-TPA9 have been described in our previously work [29]. Poly-(3,4-ethylenedioxy-thiophene):poly-(styrenesulphonic acid) PEDOT:PSS was purchased from Bayer AG. Alq_3 and NPB were synthesized in our laboratory and purified by train sublimation.

Differential scanning calorimetry (DSC) measurements and thermogravimetric analysis (TGA) were performed on a NETZSCH (DSC-204) and a Perkine Elmer Pyris 1 analyzer under N_2 atmosphere, respectively. Photoluminescence (PL) and absorption spectra were recorded by a Shimadzu RF-5301PC fluorescence spectrophotometer and a Shimadzu UV-3100 spectrophotometer, respectively. Electrochemical measurements of these derivatives were performed with a Bioanalytical Systems BAS 100 B/W electrochemical workstation.

2.2. OLED fabrication and measurement

A double-layer devices with a configuration of ITO/dendrimers (70 nm)/Alq_3 (60 nm)/LiF (0.6 nm)/Al were fabricated, in which dendrimers were used as HTL and Alq3 was used as the electron-transporting layer (ETL) and emitting layer. The indium-tin oxide ITO-coated (20Ω/□) glass substrates were cleaned by ultrasonic treatment in detergent solutions, rinsed with acetone, boiled in isopropyl alcohol, rinsed with methanol, and then with de-ionized water. The dendrimers was spin-coated on the precleaned ITO glass at the speed of 2000 cycles/min for 30 s from the 10 mg/ml chloroform solution. The thickness of



Scheme 1. Chemical structures of the dendrimers.

the dendrimer layer was ~70 nm, measured by the Ambios Technology XP-2. A 60 nm-thick Alq_3 was deposited onto the hole-transporting layer (HTL) at a rate of 0.1–0.3 nm/s as the ETL and emitting layer through a mask at a pressure of 3×10^{-4} Pa. The devices were completed by evaporating a 0.6 nm LiF layer protected by 100 nm of Al at a base pressure of 5×10^{-4} Pa. A device with similar configuration but using NPB evaporation film as HTL was also prepared under the identical conditions. The hole-only devices with a general structure of ITO/PEDOT:PSS/dendrimers (70 nm)/Au (60 nm) were fabricated in the same way for hole mobility measurement by space-charge-limited current (SCLC) method. The EL spectra and current–voltage–luminescence characteristics were measured with a Spectrascan PR 650 photometer and a computer-controlled direct-current (DC) power supply. All the measurements were performed under ambient atmosphere at room temperature.

3. Results and discussion

3.1. Thermal properties

The thermal stabilities of dendrimers were examined by DSC and TGA analysis. From the DSC thermograms of dendrimers measured at a scanning rate of 10 °C/min in nitrogen, the dendrimers exhibited glass-transition at 115 °C for Tr-TPA3 and 140 °C for Tr-TPA9, respectively. The T_g of the dendrimers enhanced with the increase of the generation because the multibranched molecules with higher generation tend to hinder translational, rotational, and vibrational motions of the molecule. The T_g of dendrimers is higher by 19 °C for Tr-TPA3 and 44 °C for Tr-TPA9, respectively, than that of the widely used HTM NPB (96 °C), suggesting better morphological stability of the dendrimers. In addition, the dendrimers shows excellent decomposition temperature by TGA measurement (T_d, which corresponded to a 5% weight loss) at 409 °C for Tr-TPA3 and 426 °C for Tr-TPA9, respectively. To investigate the dendrimers morphology, we prepared thin films of the dendrimers through spin-coating from chloroform solution onto ITO substrates and imaged them using atomic force microscopy (AFM, Supplementary Fig. S3). The surface topographies clearly revealed that the spin-coated films exhibited fairly smooth morphologies with a root-mean-square (RMS) surface roughness of 0.58 nm for Tr-TPA3 and 1.06 nm for Tr-TPA9. The films had smooth surfaces with no obvious crystallized aggregation or cracks which demonstrate that Tr-TPA3 and Tr-TPA9 are capable of forming amorphous films through solution processing.

3.2. Photophysical properties

The absorption of dendrimers in dilute chloroform solutions and solid films are presented in Fig. 1. For the UV–vis absorption spectra in chloroform solution, Tr-TPA3 displays two absorption peaks at 311 and 392 nm, respectively. The first absorption peak in the short wavelength region at 311 nm was attributed to the triphenylamine moiety. The second absorption peak at 392 nm was ascribed to the TPA-vinylene modified truxene unit [30,31]. A red-shift in the absorption maximum of Tr-TPA3 was observed in comparison with the triphenylbenzene core dendrimer (TPB-TPA3, λ_{max} = 384 nm) [30] and truxene linked with vinylene (TM3, λ_{max} = 311 nm) [31], which was indicative of enhanced effective conjugation length. While Tr-TPA9 displays two absorption peaks at 310 and 413 nm, which exhibit similar behavior to Tr-TPA3. These results also demonstrate that the effective conjugation length improves with the increasing generation of the dendrimers. The absorption behaviors of Tr-TPA3 and Tr-TPA9 in the solid films are quite similar to those in solution. The absorption maxima of Tr-TPA3 and Tr-TPA9 in the films are red-shifted 4 and 1 nm, which demonstrates that the higher of the generation, the smaller the red-shift in the absorption. The absorption of the dendrimers indicates that there is no aggregation in the dendrimers because of the large size of the molecules with six butyl substituents in truxene and lots of noncoplanar triphenylamines dendrons. There is no significant ordering of the dendrimers either from aggregation or crystallization, and hence the dendrimers films are in good amorphous states [32], which are very important for the application of materials in optical and electronic devices such as OLEDs.

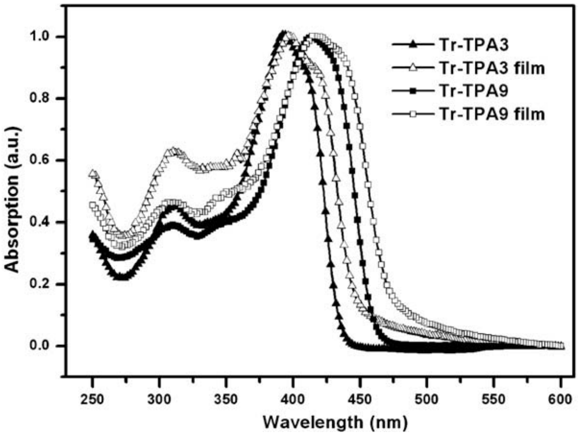


Fig. 1. Normalized absorption spectra of Tr-TPA3 and Tr-TPA9 in dilute chloroform solution (1×10^{-6} M) and solid films.

Fig. 2 illustrates the PL spectra of dendrimers in dilute chloroform solutions and solid films. For the emission spectra in dilute chloroform solutions, Tr-TPA3 and Tr-TPA9 display similar behaviors. The emission peaks of Tr-TPA3 and Tr-TPA9 locate at 445 and 476 nm, respectively. The red-shifted of 31 nm of the emission peak of Tr-TPA9 from that of Tr-TPA3 shows that there is obvious π–π^* delocalization and the significant increase of the effective conjugation length with the increase of the generation of dendrimers [33]. The two peaks in PL of Tr-TPA3 film can be ascribed to the S1–S0 0–0 transition at about 440 nm (about 22,750 cm^{-1}) and the vibronic satellites with an appr. 1200 cm^{-1} vibration, respectively. In solution the vibronic splitting is less obvious because the individual components are broadened. Tr-TPA9 displays similar behaviors to Tr-TPA3. Actually, the principal character of the PL spectra is the same for film and solution.

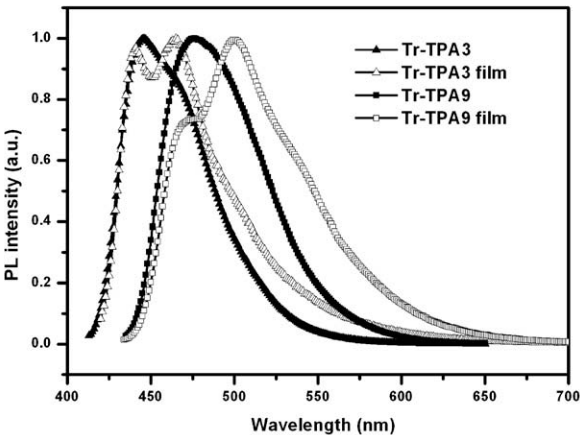


Fig. 2. Normalized fluorescence spectra of Tr-TPA3 and Tr-TPA9 in dilute chloroform solution (1×10^{-6} M) and solid films.

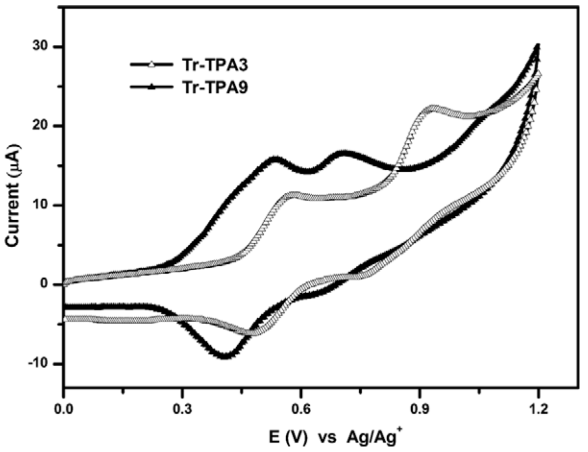


Fig. 3. Cyclic voltammograms of Tr-TPA3 and Tr-TPA9 in 0.1 M Bu_4NPF_6/CH_2Cl_2, scan rate 100 mV s^{-1}.

3.3. Electrochemical properties

The electrochemical behaviors of Tr-TPA3 and Tr-TPA9 were examined by cyclic voltammetry measurement. As shown in Fig. 3, two reversible oxidation potentials for Tr-TPA3 and Tr-TPA9 were observed. The first oxidation potentials of Tr-TPA3 and Tr-TPA9 are 0.57 and 0.53 V, and the second oxidation potentials are 0.94 and 0.71 V, suggesting a successive formation of the cation radical and then dication species [10]. All of the oxidations should be attributed to the removal of electrons from the triphenylamine groups. The lower oxidation potential of Tr-TPA9 is likely to be related to the extended π-conjugation length [10].

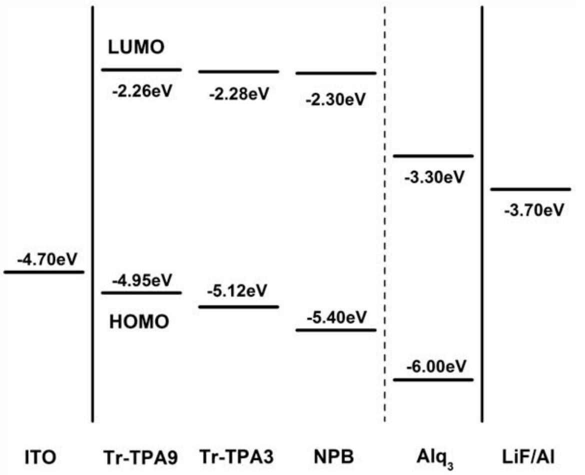


Fig. 4. Energy levels of the dendrimers, NPB and Alq_3.

On the basis of the roughly evaluated onset oxidation potentials (E_{Red}^{onset}), the HOMO energy levels of Tr-TPA3 and Tr-TPA9 are estimated as −5.12 and −4.95 eV, respectively (HOMO = $-e(E_{ox}^{onset}$+ 4.66)) [34–36]. The LUMO energy levels of Tr-TPA3 and Tr-TPA9 are −2.28 and −2.26 eV, respectively, calculated from the HOMO energy level and energy bandgap (E_g) determined from the UV–vis absorption threshold (LUMO = HOMO − E_g). The electrochemical properties as well as the energy level parameters of the dendrimers are listed in Table 1. From Table 1 it can be seen that the electron donating ability of the dendrimers increased with the increase of the number of triphenylamine units and resulted in the enhancement of the HOMO energy level. Fig. 4 shows the energy levels of the dendrimers and that of NPB and Alq_3. It can be seen that the HOMO energy level of Tr-TPA3 and Tr-TPA9 are higher than that of NPB [37], which means that the energy barrier at the interface of ITO and dendrimers is smaller than that of ITO and NPB resulting in the better hole injection from ITO to dendrimers than to NPB and lower joule heat produced at the interface during device operation.

3.4. EL properties

To investigate the hole-transport ability of triphenylamine-based dendrimers, double-layer OLEDs with the

Table 1
Electrochemical properties of the dendrimers.

Compound	E_g[a] (eV)	E_{onset}^{ox}[b] (V)	E_{p1}^{ox}[c] (V)	E_{p2}^{ox}[c] (V)	HOMO/LUMO[d] (eV)
$Tr\text{-}TPA_3$	2.84	0.46	0.57	0.94	–5.12/−2.28
$Tr\text{-}TPA_9$	2.69	0.29	0.53	0.71	–4.95/−2.26

[a] Determined from UV–vis absorption spectra.

[b] E_{onset}^{ox}: onset oxidation potential; potentials versus Ag/Ag+, working electrode glassy carbon, 0.1 M Bu_4NPF_6/CH_2Cl_2, scan rate 100 mV s^{-1}.

[c] E_{p1}^{ox}: potential of first oxidation peak; E_{p2}^{ox}: potential of second oxidation peak.

[d] HOMO (eV) = $-e(E_{ox}^{onset} + 4.66$; LUMO(eV) = HOMO − E_g.

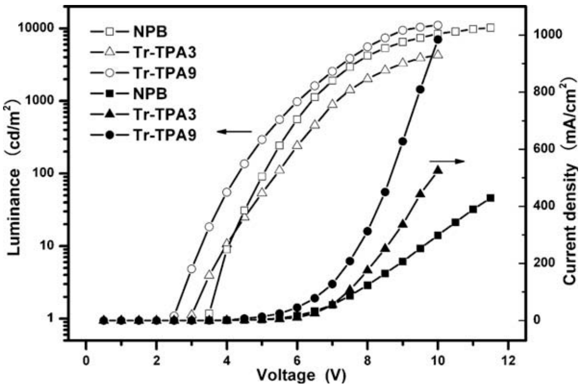


Fig. 5. The current density–luminance–voltage curves of the ITO/Tr-TPA3 or Tr-TPA9/Alq$_3$/LiF/Al device and the ITO/NPB/Alq$_3$/LiF/Al standard device.

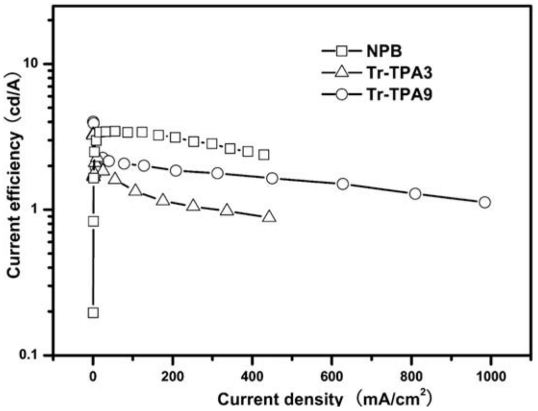


Fig. 6. The current efficiency–current density characteristic of the ITO/Tr-TPA3 or Tr-TPA9/Alq$_3$/LiF/Al device and the ITO/NPB/Alq$_3$/LiF/Al standard device.

configuration of ITO/dendrimers (70 nm)/Alq$_3$ (60 nm)/LiF/Al were fabricated, where dendrimers as HTL and Alq$_3$ as emitting layer and ETL. As comparison, a standard device with the similar structure but using NPB as the HTL was also prepared. The devices based on dendrimers showed green emission from Alq$_3$ with a peak at 528 nm, suggesting that the charge recombination is localized in the Alq$_3$ layer and dendrimers act primarily as HTM without causing exciplex formation at the interface with Alq$_3$. The density–voltage (*J*–*V*) and luminance–voltage (*L*–*V*) characteristics of the devices are shown in Fig. 5. The Tr-TPA3-based device turns on at a voltage of 3 V, and the brightness of the device is 4265 cd m^{-2} at 10 V. The Tr-TPA9-based device turns on at a voltage of 2.5 V, and the brightness of the device reach 11,058 cd m^{-2} at 10 V. It is clear that the brightness of the Tr-TPA9-based device is comparable with the NPB standard device. The low turn-on voltage of the Tr-TPA3 and Tr-TPA9 devices is attribute to the lower hole injection barrier from ITO to dendrimers than that from ITO to NPB.

Fig. 6 shows the current efficiency–current density characteristics of the dendrimers devices. The Tr-TPA9-based device gives a maximum current efficiency of 4.01 cd A^{-1}, which is substantively higher than that (3.24 cd A^{-1}) of the Tr-TPA3-based device. The current efficiency for the Tr-TPA3-based device decreases more rapidly with increasing current density above 25 mA cm^{-2}. While the current efficiency for the Tr-TPA9-based device remains almost constant with current density up to 150 mA cm^{-2} and shows only a small decline even when the current density increases to 620 mA cm^{-2}. The EL properties of the devices are summarized in Table 2. Significantly, even at the present stage when the device has not yet to be optimized, the EL performances of the Tr-TPA9-based device are already comparable to those of the NPB/Alq$_3$ device, which suggests that Tr-TPA9 is at least as good as NPB when used as a HTM in EL devices, apart from its high T_g, good solubility and film forming property from solution.

We also evaluate the hole mobilities of the dendrimers by analyzing space-charge-limited current (SCLC) from the hole-only device with the configuration of ITO/PEDOT:PSS/dendrimers/Au [38–41], where *L* varied between 50 and 100 nm. The hole mobilities of the dendrimers were calculated according to the following equations [42]:

$$J_{\mathrm{SCLC}} = \frac{9}{8}\varepsilon_0\varepsilon_r\mu_0\frac{(V_{\mathrm{appl}} - V_{\mathrm{bi}})^2}{L^3}\exp\left[0.891\gamma\sqrt{\frac{V_{\mathrm{appl}} - V_{\mathrm{bi}}}{L}}\right]$$

$$\ln\left[\frac{JL^3}{(V_{\mathrm{appl}} - V_{\mathrm{bi}})^2}\right] = \ln\frac{9}{8}\varepsilon_0\varepsilon_r + \ln\mu_0 + 0.891\gamma\sqrt{\frac{V_{\mathrm{appl}} - V_{\mathrm{bi}}}{L}}$$

The results are plotted as ln(JL^3/V^2) versus $(V/L)^{0.5}$, as shown in Fig. 7. The hole mobilities of Tr-TPA3 and Tr-TPA9, calculated from the intercepts of the corresponding lines, are about 10^{-5} cm^2/Vs and 10^{-4} cm^2/Vs, respectively. The hole mobilities of dendrimers are comparable to those of well-known triphenylamine derivatives [11]. The spatial geometry of the dendrimers could be beneficial for intra- and intermolecular hole transportation and the excellent hole mobility observed in Tr-TPA9 than Tr-TPA3 is likely to be related to the extended π-conjugation length which may enhance the capability of the effective interchromophore interaction between the neighboring molecules [38,43].

Table 2
Summary of device performances.

HTL	EL (nm)	V_{on} (V)	$L_{\max}$ (cd m^{-2})	Maximum current efficiency (cd A^{-1})	Maximum power efficiency (lm W^{-1})
NPB	528	3.5	10,220	3.45	1.78
Tr-TPA$_3$	528	3	4265	3.24	2.91
Tr-TPA$_9$	528	2.5	11,058	4.01	3.60

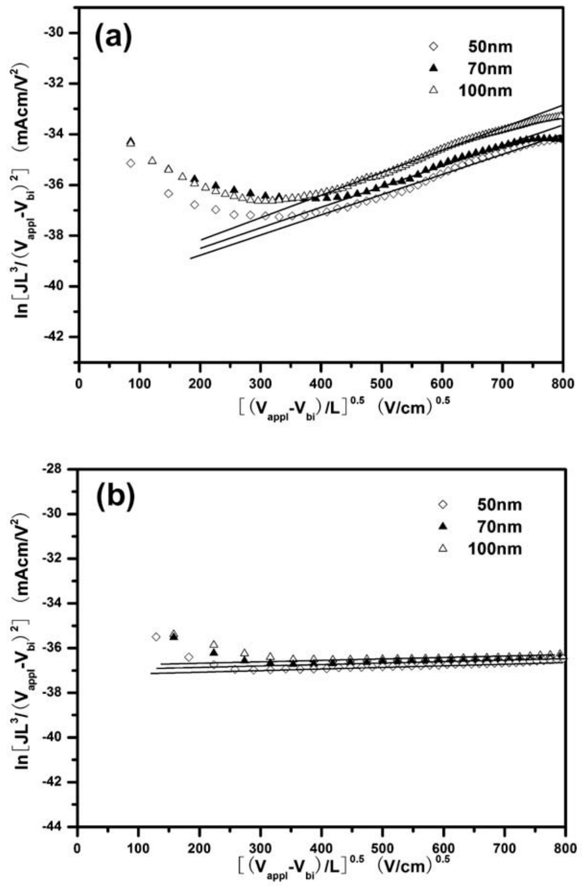


Fig. 7. Current–voltage data from the device of ITO/PEDOT:PSS/Tr-TPA3 or Tr-TPA9/Au, plotted in the format $\ln(JL^3/V^2)$ versus $(V/L)^{0.5}$, where J is the current density, L is the thickness of Tr-TPA3 or Tr-TPA9 layer. The lines are the fit to the respective experimental data points. (a) Tr-TPA3 and (b) Tr-TPA9.

4. Conclusions

To summarize, two novel solution-processable dendrimers, Tr-TPA3 and Tr-TPA9, were used as HTM in OLEDs. The photophysical properties of dendrimers demonstrate that the films of dendrimers are in good amorphous states. The dendrimers possess excellent solubility and good film forming property. The T_g of dendrimers is higher than that NPB, suggesting better thermal stability of the dendrimers. The Tr-TPA9-based double-layer device exhibited similar EL brightness, slightly higher maximum current efficiency and lower turn-on voltage comparing with the NPB-based device. Therefore, the triphenylamine-based dendrimers with truxene as cores can be good candidates for HTM in OLEDs.

Acknowledgements

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.orgel.2009.04.024.

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