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PAPER

Pyrene-cored dendrimer with carbazole derivatives as dendrons: synthesis, properties and application in white light-emitting diode†

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A new dendrimer using pyrene as core and carbazole derivative as dendron has been successfully prepared *via* Suzuki coupling reaction. Its chemical structure was confirmed through ¹H NMR, elemental analysis and MALDI-TOF MS methods. The dendrimer synthesized possessed excellent thermal stability with initial decomposition temperature over 470 °C and high fluorescence quantum yield of 86%. The luminescence spectra showed that, relative to the solution sample, the emission peaks of the solid dendrimer film were apparently broadened and red-shifted, indicating the strong π - π stacking effect between the pyrene moieties. By doping 1.5% of the dendrimer in 4,4'-bis[*N*-(1-naphthyl)-*N*-phenylamino]biphenyl (NPB), a light-emitting diode device was fabricated in the ITO/NPB/NPB:dendrimer (1.5%)/TPBI/Mg:Ag configuration, which emitted a white color with Commission Internationale de L'Eclairage (CIE_{x,y}) coordinates of (0.29, 0.34) and a maximum brightness of 1300 cd m⁻², exhibiting promising potential in white light-emitting diode application.

Introduction

As one of the important aromatic chromophores, pyrene has been modified through a variety of synthetic strategies into numerous organic and polymeric luminescence materials.^{1–3} Typically, pyrene-based compounds possess high photoluminescence efficiency⁴ and high carrier mobility,^{5–7} which allow them to be used in various optoelectronic applications such as organic or polymeric light-emitting devices^{8–10} and field-effect transistors.¹¹ For example, Gingras *et al.* synthesized a class of polysulfurated pyrene-cored dendrimers with appended poly(thiophenylene) dendrons.¹² Tao *et al.* reported the blue-light-emitting fluorene derivatives with the pyrene group at the C2, C7 positions.¹³ The copolymers based on pyrene and 3,4-ethylenedioxythiophene (EDOT) were prepared *via* electrochemical method by the direct oxidation of pyrene/EDOT mixtures.¹⁴

Because of the large planar conjugative structure, pyrene-containing compounds usually exhibit strong intermolecular π - π interaction. Previously, the broad emission peaks and red-shifted emissions owing to the aggregating between the pyrene moieties has been regarded as a disadvantage for the

light-emitting devices.¹⁵ Nevertheless, the recent studies reveal that the supramolecular π - π stacking effect may play a positive role, and offer a new route to develop white electroluminescence materials. Indeed, Huang *et al.* fabricated a single-layered white light-emitting diode using 9-(49-(20-ethylhexyloxyphenyl))-2,9-dipyranylfluorene (2PPPF) and poly(2-methoxy-5-(2'-ethylhexyloxy)-*p*-phenylenevinylene) (MEH-PPV) blend as the bulk emitter. By doping MEH-PPV into 2PPPF host, the emission peak was significantly broadened, leading to the device strong white-light-emitting.¹⁶

On the other hand, carbazole has been long recognized to be a luminescence compound with high hole-transporting ability arising from the lone pair electrons in nitrogen atom.^{17–19} Over the past decade, carbazole have been incorporated into the linear polymer backbone, or acted as dendrons to construct dendrimer materials, in which carbazole groups are employed as effective energy harvesting moieties, and the singlet and triplet energy can be transferred to the neighboring conjugative group or core of a dendrimer, affording a high luminescence efficiency.^{20,21}

Among the light-emitting diodes, white light emitting devices are currently most pursued owing to their potential applications for the full-color display or as backlight for liquid-crystal displays and solid-state lighting.^{22,23} White-light emission can be obtained by applying three primary colors (red, green, and blue) in physically separated layers,^{24,25} or doping two monochromatic fluorophores or phosphors in the same host.²⁶ However, the drawback of these approaches is that the multi-layer architecture or the introduction of

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dopants in the system are subject to light scattering across layer junctions and self absorption.²⁷ As an alternative method, white-light emission can also be produced by mixing the host material and dopant in a single emission layer. In this way, the devices are easily fabricated, and consequently more cost-effective. Moreover, the problems involving the light scattering can be effectively resolved.

With the above considerations in mind, herein, we designed and synthesized a novel dendrimer consisting of a pyrene core and carbazole derivatives dendron. The combination of pyrene and carbazole is expected to provide the improved thermal stabilities and good charge-transportation ability. As well known, dendrimers possess accurately defined chemical structure with highly branched topology, well controlled size and batch to batch reproducibility, which characteristics are advantageous for them to achieve excellent electroluminescent properties. Using this dendrimer as a fluorescent dopant emitter and blue-light-emitting 4,4'-bis[*N*-(1-naphthyl)-*N*-phenylamino]biphenyl (NPB) as a host, a simple three-layer LED device, *i.e.* ITO/NPB/NPB:dendrimer (1.5%)/TPBI/Mg:Ag, was fabricated. Its electroluminescent properties are investigated and evaluated.

Experimental

Materials and instrumentations

Pyrene, dibromobenzene, carbazol and tetrakis(triphenylphosphine)palladium were purchased from Shanghai Regent Corporation Ltd, and used as received. THF was distilled from sodium/benzophenone prior to use. Other commercial reagents were of analytical grade and used without further purification unless otherwise stated. All chromatographic separations were carried out on silica gel (400 mesh).

Melting points were performed using a X-4 melting-point apparatus with microscope. ¹H NMR spectra were recorded in CDCl₃ on a Varian INOVA at 400 MHz. Elemental analysis was carried out on an Elementar Vario EL III elemental analyzer. Molecular mass was determined on a MALDI micro MX laser desorption-ionization time-of-flight mass spectrometer (MALDI-TOF MS). Thermal gravimetric analysis (TGA) was measured on a NETZSCH TG209C in a nitrogen atmosphere at a heating rate of 10 °C min⁻¹. Different scanning calorimetry (DSC) was carried on a NETZSCH DSC204 with a heating rate of 10 °C min⁻¹ in a nitrogen atmosphere. Absorption and photoluminescence (PL) spectra were recorded on a PTI-700 spectrophotometer and a HP854 luminescence spectrometer, respectively. Cyclic voltammetric (CV) curves were recorded using a BSA 100B electrochemical analyzer with a conventional three-electrode configuration consisting of a glassy carbon working electrode, a platinum auxiliary electrode and a non-aqueous Ag/AgNO₃ reference electrode.

Electroluminescence (EL) device fabrication and measurements

Light-emitting diode was fabricated by vacuum deposition on top of ITO (indium tin oxide) glass substrate. Before deposition, the ITO substrate with a sheet resistance of 30 Ω square⁻¹ was cleaned in organic solvents and deionized water, then

dried in an oven at 120 °C for 2 h. The device was fabricated by evaporating organic layer at a rate of 3 Å S⁻¹ with a pressure lower than 1 × 10⁻⁵ mbar. Onto the 2,2',2''-(benzene-1,3,5-triyl)-tris(1-phenyl-1*H*-benzimidazole) (TPBI) layer, the cathode consisting of Mg:Ag alloy was deposited at a rate of 10 Å S⁻¹. EL spectra and CIE color coordinates of the device were measured with Spectrascan PR 705. The electroluminescence properties of the device were measured with a computer controlled Keithley 236 source meter under ambient condition.

Synthesis of 1,3,6,8-tetrabromo-1,9-dihydropyrene (1).

1,3,6,8-tetrabromo-1,9-dihydropyrene was prepared according to a modified procedure given in the literature.²⁸ Liquid bromine (1.29 ml, 25 mmol) was added dropwise to a solution of pyrene (1 g, 4.95 mmol) in nitrobenzene (10 ml) under vigorous stirring. After complete addition, the mixture was heated at 160 °C for 3 h. The resulting solution was poured into acetone, and the precipitate was filtered and washed with ethanol. Drying the precipitate under vacuum gave 2.43 g yellow product with a yield of 94.2%. ¹H NMR (400 MHz, CDCl₃): δ (ppm) = 8.44 (s, 2H), 8.42 (s, 4H).

Synthesis of 9-(4-bromophenyl)carbazole (2).

9-(4-Bromophenyl)carbazole was synthesized in the similar procedure described previously.²⁹ A mixture of CuI (1.14 g, 6 mmol), 18-Crown-6 (0.53 g, 2 mmol), K₂CO₃ (16.6 g, 120 mmol), 1,3-dimethyl-3,4,5,6-tetrahydro-2(1*H*)-pyrimidinone (DMPU) (2 mL), dibromobenzene (14.2 g, 60 mmol) and carbazole (10 g, 60 mmol) was heated at 170 °C for 11 h under nitrogen. After cooling to room temperature, the mixture was quenched with 1 N HCl, and the precipitate was filtered and washed with NH₃·H₂O and deionized water. The grey solid was purified with column chromatography using hexane as an eluant to afford the white product (11.7 g). Yield: 71%. mp: 145–146 °C. ¹H NMR (400 MHz, CDCl₃): δ (ppm) = 8.13 (d, 2 H, *J* = 7.6 Hz), 7.72 (d, 2 H, *J* = 8.8 Hz), 7.45 (d, 2 H, *J* = 8.8 Hz), 7.41–7.37 (dt, 4H, *J*₁ = 6.8 Hz, *J*₂ = 6.8 Hz), 7.30 (t, 2H, *J* = 6.6 Hz).

Synthesis of 4-carbazolyl-1-bromophenylboronic acid (3).

4-Carbazolyl-1-bromophenylboronic acid was prepared in reference to the literature with some modifications.³⁰ 2.5 M *n*-BuLi in hexane (7.2 mL, 18 mmol) was added dropwise to a solution of (1) (4.3 g, 15 mmol) in THF (40 mL) at –78 °C with stirring. After reaction for 1h, triisopropyl borate (32 mL, 18 mmol) was added using a syringe. The mixture was stirred for additional 1 h and then was gradually heated to room temperature and stirred overnight. The clear solution was diluted with ether (100 mL) and washed with deionized water. The organic layer was dried over MgSO₄. After filtration, the solution was concentrated using a rotary evaporator. Flash column chromatography of the residue over silica gel with a mixed solvent of hexane and ethyl acetate (9/1) as a gradient eluent afford 3.3 g white solid with a yield of 87%. mp: 182–183 °C. ¹H NMR (400 MHz, CDCl₃): δ (ppm) = 8.56 (d, 2H, *J* = 8.0 Hz), 8.18 (d, 2H, *J* = 7.6 Hz), 7.80 (d, 2H, *J* = 8.0 Hz), 7.56 (d, 2H, *J* = 8.0 Hz), 7.46 (t, 2H, *J* = 7.2 Hz), 7.33 (t, 2H, *J* = 7.6 Hz).

Synthesis of 9-(4-(3,6,8-tris(4-(9*H*-carbazol-9-yl)phenyl)-3,3-dihydropyren-1-yl)phenyl)-9*H*-carbazole (P1). Tetrakis(triphenylphosphine) palladium (0.034 g, 0.03 mmol), (**1**) (0.155 g, 0.3 mmol) and (**3**) (0.517 g, 1.8 mmol) were added to an air-free two-phase mixture of toluene (15 mL) and 2M K₂CO₃ aqueous solution (12 mL). The resultant system was vigorously stirred under an argon atmosphere at 80 °C for 24 h. The organic layer was separated and the aqueous phase was extracted with diethyl ether. The organic phases were combined and washed with brine and dried over anhydrous MgSO₄. The solvent was evaporated and the residue went through silica-gel column with dichloromethane as an eluant. Elemental analysis: calculated for C₈₈H₅₄N₄, C 90.54%, H 4.66%, N 4.80%; found, C 90.47%, H 4.71%, N 4.90%. ¹H NMR (500 MHz, CDCl₃): δ (ppm) = 8.51 (s, 4H), 8.32 (s, 2H), 8.21 (d, 8H, *J* = 8 Hz), 8.02 (d, 8H, *J* = 7.6 Hz), 7.83 (d, 8H, *J* = 7.6 Hz), 7.62 (d, 8H, *J* = 7.2 Hz), 7.49 (t, 8H, *J* = 7.6 Hz), 7.35 (t, 8H, *J* = 7.2 Hz). MALDI-TOF MS (*m/z*): Calcd. for C₈₈H₅₆N₄ 1168.45, found 1167.44 [M⁺].

Results and discussion

Synthesis and characterization

The chemical structure and synthetic route of the dendrimer are illustrated in Scheme 1. 1,3,6,8-Tetrabromopyrene (**1**) was readily obtained by direct bromination of pyrene using bromine in nitrobenzene solution. The synthesis of 9-(4-bromophenyl)-carbazole (**2**) was prepared by the moderated Ullman reaction between the carbazole and 1,4-dibromobenzene. The obtained compound (**2**) was reacted with *n*-BuLi in THF at −78 °C, followed by the addition of triisopropyl borate and then hydrolysis with 2 N HCl to give the 4-(9*H*-carbazol-9-yl)-phenylboronic acid (**3**). The target dendrimer (**P1**) was prepared *via* Suzuki coupling reaction between (**1**) and (**3**) in

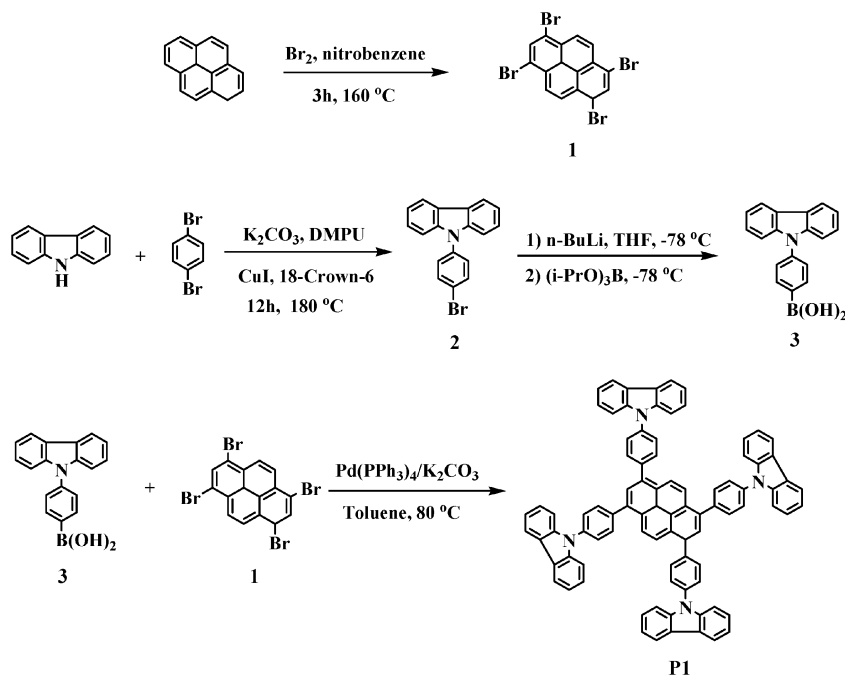
the presence of tetrakis(triphenylphosphine) palladium and K₂CO₃. The chemical structure of **P1** was confirmed by elemental analysis, ¹H NMR (Fig. 1S, Supporting Information) and MALDI-TOF-MS (Fig. 2S, Supporting Information). The peaks at 8.21 (doublet), 8.02 (doublet), 7.83 (doublet), 7.62 (doublet), 7.49 (triplet) and 7.35 ppm (triplet) could be assigned to the dendron protons, whereas the peaks at 8.51 and 8.32 ppm were attributed to the core protons. Moreover, the peak in MALDI-TOF MS spectrum was exactly matching the calculated molecular weight of **P1**.

Thermal properties

The thermal properties of **P1** were investigated by differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) under nitrogen atmosphere. For the first heating-run, the DSC plot exhibited a sharp melting point up to 338 °C (Fig. 1). However, when the sample was quenched and reheated for the second time, the former melting peak almost completely disappeared. The above results indicated that the melting peak in the first-heating curve was due to the strong π - π stacking effect between the pyrene cores, which was broken at the high temperature. As a result, it exhibited amorphous nature in the second heating curve. In addition, no glass transition temperature was detected for the two heating runs. The reason may be that the structure of **P1** is so rigid that its glass transition occurs at the higher temperature beyond the measuring range. In the TGA curve (Fig. 2), the **P1** has the initial decomposition temperature at 470 °C and 5% weight loss at 580 °C, exhibiting excellent thermal stability.

Photophysical properties of solution and film samples of P1

The absorption spectra of **P1** for the dilute solution sample in tetrahydrofuran (THF) and solid film sample on quartz are



Scheme 1 Synthetic route and chemical structure of the dendrimer.

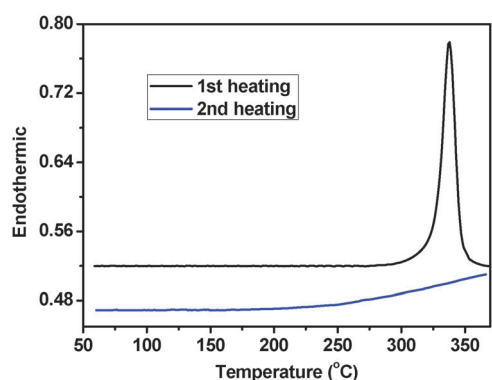


Fig. 1 DSC traces of **P1** recorded at a heating rate of $10\text{ }^{\circ}\text{C min}^{-1}$.

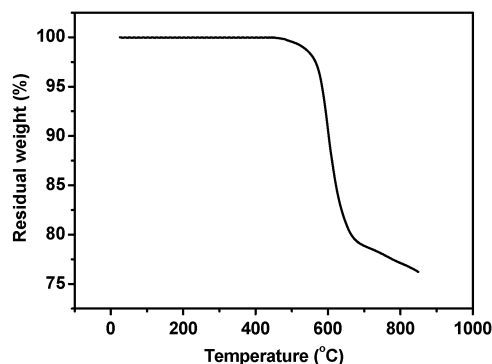


Fig. 2 TGA curve of **P1** measured at $10\text{ }^{\circ}\text{C min}^{-1}$.

presented in Fig. 3. For the solution sample, the absorption spectrum of **P1** displayed three major absorption bands. The lowest-energy absorption band appearing at 394 nm was from the π - π^* transition of the core,³¹ the weak absorption peaks observed around 327 and 340 nm could be attributed to the n - π^* transition of the carbazole moieties,³² while the highest-energy absorption band at 293 nm was ascribed to the π - π^* transition of the carbazole moieties.^{33,34} For the solid film, the absorption spectrum of **P1** was nearly identical to that in solution with only a slight red-shift. The optical band gap (E_g) of **P1** measured from the onset wavelength in its film absorption spectrum was 2.7 eV. The photoluminescence spectra of **P1** in THF solution (Fig. 4) exhibited one emission peak at 447 nm and two shoulders at 470 and 514 nm, which

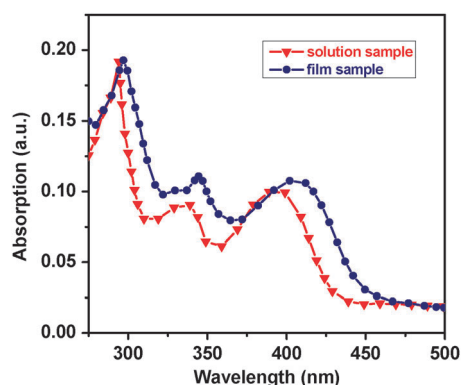


Fig. 3 Absorption spectra of **P1** in solid film and dilute solution.

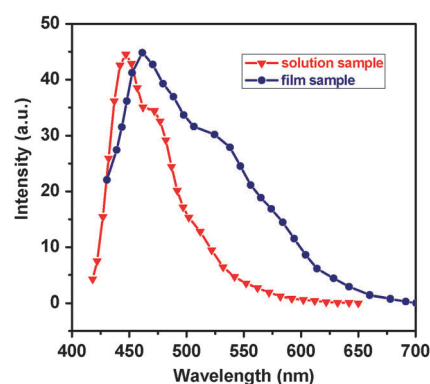


Fig. 4 Photoluminescence spectra of **P1** in solid film and dilute solution.

originated from the pyrene core.³⁵ No emission from the carbazole dendrons could be observed, implying that the excitation energy absorbed by carbazole dendrons had been efficiently transferred to the pyrene core. The emission peaks of **P1** in the solid state were obviously red-shifted relative to that in solution, indicating the strong aggregation of pyrene group, which was well consistent with the DSC result. The fluorescence quantum efficiency of the **P1**, obtained at room temperature in dilute THF solution using quinine sulfate (0.1 N in H_2SO_4) as a calibration standard, was as high as 86%, suggesting that the dendrimer with carbazole moieties attached to the pyrene core has good fluorescence properties.

Electrochemical behavior of **P1** film

The electrochemical property of **P1** was examined by cyclic voltammetry (CV) method. The **P1** film obtained by drop-casted on a glass carbon electrode was scanned both positively and negatively in a 0.10 M anhydrous acetonitrile solution of tetrabutylammonium hexafluorophosphate (Bu_4NPF_6), using Ag/Ag^+ as the reference electrode calibrated with ferrocene/ferrocenium (Fc/Fc^+) (4.8 eV below the vacuum level).³⁶ As depicted in Fig. 5, **P1** gives two onset oxidation potential (E_{ox}) at 0.5 and 1.58 V, respectively, as well as one onset reduction potential (E_{red}) at 2.25 V, indicating that these two redox curves are assigned to two separate oxidation process. The first reversible process should be corresponded to the oxidation at the pyrene,⁸ while the second irreversible process could be

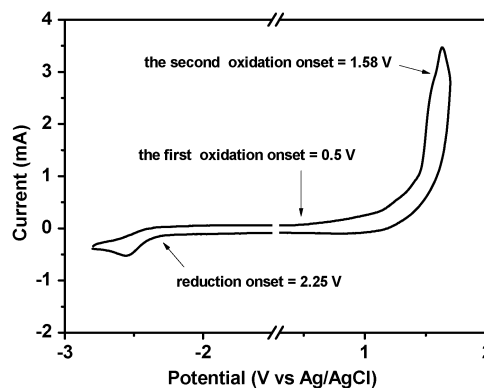


Fig. 5 Cyclic voltammetry curve of the **P1** film (the inset shows the anodic run).

attributed to the oxidation of the 3,6-position on carbazole units.³⁷ It was obvious that the introduction of the carbazole could help to enhance the hole transportation ability of the dendrimer and thus to facilitate the hole injection into film. The HOMO and LUMO energy levels as well as the electrochemical energy gap of the **P1** were calculated according to the following equation: $\text{HOMO} = -E_{\text{ox}} - 4.8 \text{ eV}$, $\text{LUMO} = -E_{\text{red}} - 4.8 \text{ eV}$, and $E_{\text{g}} = E_{\text{ox}} - E_{\text{red}}$, respectively. From the empirical formula above, the HOMO and LUMO energy level of **P1** were estimated to be -5.3 eV and -2.55 eV , respectively, which were close to the energy levels of NPB,³⁸ implying easy hole and electron injections from NPB host into the guest based on **P1**. The electrochemical band gap of **P1** was 2.75 eV , which was higher than that measured from the absorption spectrum.

Properties of optoelectronic device

To evaluate the electroluminescence (EL) properties of **P1**, a three-layer device with a structure of ITO/NPB (40 nm)/NPB : **P1** (30 nm, 1.5%)/TPBI (60 nm)/Mg : Ag was fabricated by vacuum deposition method. In this device, ITO and Mg : Ag were used as anode and cathode, respectively; 4,4'-bis[*N*-(1-naphthyl)-*N*-phenylamino]biphenyl (NPB) was used for hole-transporting layer (HTL) and host for the dendrimer dopant, whereas TPBI served as an electron-transporting material (ETL). Fig. 6 showed that the EL spectrum of **P1**-based device was very broad, covering a range from 400 to 650 nm, in which the emission peaks at 480, 566 and 612 nm were from the dendrimer emitter and the short-wavelength peak at 430 nm was originated from the NPB host layer.³⁸ In comparison with the PL spectrum, the EL spectra bands of **P1** were red-shifted and the intensity of blue peak at 480 nm increased apparently. As revealed in the absorption spectra, the red-shifted EL emission was also caused by the close stacking of pyrene-based dendrimer.³¹ The inset of Fig. 7 depicts the schematic energy level diagram of the white organic light-emitting device. The highest occupied molecular orbit (HOMO) and the lowest unoccupied molecular orbit (LUMO) levels of NPB are 5.7 and 2.2 eV,³⁸ respectively, whereas the HOMO and LUMO of **P1** are 5.3 and 2.55 eV, respectively. The HOMO level of NPB lies about 0.4 eV lower and LUMO about 0.35 eV higher than that of **P1**, meaning that both holes and electrons could be potentially trapped by **P1**. On the other

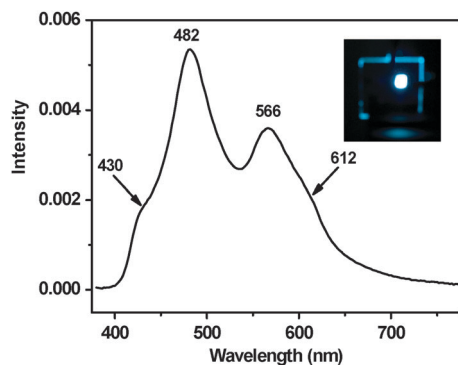


Fig. 6 Electroluminescence spectrum of the device (the inset shows image of the device under operation).

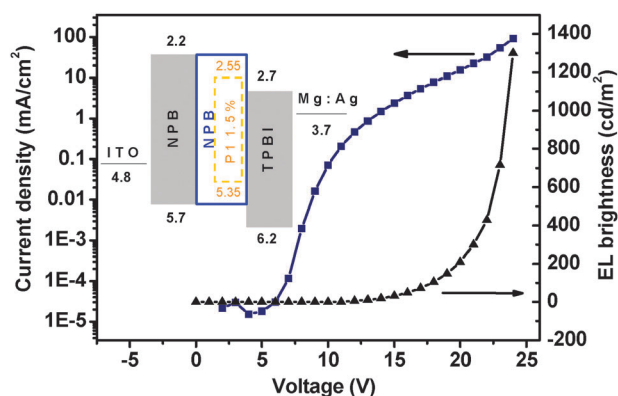


Fig. 7 Curves of current density-voltage and brightness-voltage for the device (the inset shows schematic device configuration of the device).

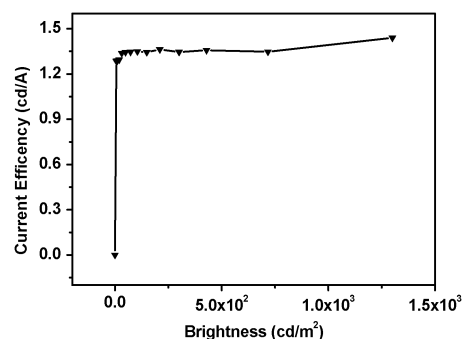


Fig. 8 Plot of brightness versus efficiency of the device.

hand, doping the **P1** into NPB further resulted into broadened and red-shifted peaks in the EL spectra, thus leading to the white emission. The device displayed efficient white-light emission with Commission Internationale de L'Eclairage (CIE_{x,y}) coordinates at (0.29, 0.34). The Current density-voltage-brightness characteristics and brightness versus efficiencies plots of the device are shown in Fig. 7 and 8, respectively. The turn-on voltage of device is 12 V, the maximum brightness is 1300 cd m^{-2} and the peak efficiency can reach 1.44 cd A^{-1} at a driving voltage of 24 V. It is important to note that the current efficiency of the device shows a slightly enhanced response and no drop is observed with the increase of brightness.

Conclusions

We have successfully synthesized a new dendrimer consisting of a pyrene core and carbazole-based dendrons. The dendrimer exhibited high thermal stability with initial decomposition temperature up to 470°C . Of interest was the observation that the π - π stacking effect between the pyrene cores resulted into a sharp melting peak in the DSC curve, which completely disappeared in the second heating-run by the quenching treatment. Moreover, the strong supramolecular interaction was further proved by the significantly broadened and red-shifted fluorescence emission peaks for the dendrimer solid film compared to those of the solution sample. The dendrimer had high fluorescent quantum efficiency of 86%. When the

pyrene-containing dendrimer was doped into NPB, the fabricated device exhibited intense white light (CIE: 0.29, 0.34) with the maximum brightness of 1300 cd m⁻². The investigation on the higher generations of dendrimers is still undertaken and will be published subsequently.

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