Highly-efficient hybrid white organic light-emitting diodes based on a high radiative exciton ratio deep-blue emitter with improved concentration of phosphorescent dopant (Postprint)

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Date: 2017-04-06T00:00:00+00:00

Abstract

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Full Text

Preamble

Highly-efficient hybrid white organic light-emitting diodes based on a high radiative exciton ratio deep-blue emitter with improved concentration of phosphorescent dopant†

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An improved concentration of phosphorescent dopant for highly-efficient hybrid white organic light-emitting diodes based on a high radiative exciton ratio (80%) deep-blue emitter has been developed. The high radiative exciton ratio for the deep-blue emitter was found to result from transfer from the higher triplet state (T5) to the lowest singlet state (S1) via a "hot-exciton" process. Notably, when the concentration of Ir(2-phq)3 is up to 0.9 wt%, the OLED still exhibited white emission with maximum total EQE, CE, and PE of 22.3%, 53.7 cd A ^{1}, and 60.2 lm W ^{1}, respectively. The exciton transfer mechanism in a high concentration of phosphorescent dopant was also discussed. These studies provide a pathway to obtain high-performance F/P hybrid WOLEDs with a simple architecture and improved doping concentration.

Introduction

White organic light-emitting diodes (WOLEDs) have attracted great attention due to their favorable properties of good color rendering, flexibility, and homogeneous large-area emission for lighting sources and full-color OLEDs. In the past two decades, several effective approaches have been used to achieve WOLEDs, including full fluorescent, phosphorescent, and hybrid WOLEDs. Generally, WOLEDs have demonstrated excellent stability. However, these devices show low efficiencies owing to the limit of exciton statistics. Therefore, to obtain high efficiencies, researchers have begun to focus on full phosphorescent WOLEDs, which have been reported with high internal quantum efficiencies up to 100%. Unfortunately, these devices are not stable enough to ensure long device lifetimes due to the limited availability of high-performance blue hosts/emitters. Moreover, an intrinsic exchange energy loss arising from energy transfer from the host’ s singlets to the phosphorescent guest’ s triplets cannot be fully prevented. In view of this, the development of fluorescence (F) and phosphorescence (P) hybrid WOLEDs is considered an ideal solution to achieve high-efficiency and long-lifetime WOLEDs.

Commonly, hybrid WOLEDs can be constructed using two architectures: multi-emissive-layers (multi-EML) or a single-emissive-layer (single-EML) with different color-emitting dopants. The multi-EML structure usually requires complex device fabrication with an interlayer between the fluorophore and phosphor to prevent mutual exciton transfer and quenching processes, which decreases reproducibility and raises fabrication cost. Furthermore, the complicated multilayer structure is also difficult to achieve by solution processing. Compared to the multi-EML counterpart, the single-EML can effectively avoid the accumulation of charges and the generation of a higher electric field at the interfaces, especially for single-dopant single-EML WOLEDs. Therefore, the single-dopant single-EML structure is considered a potential strategy to fabricate hybrid WOLEDs with high efficiencies.

In early work, Leo et al. demonstrated a type of single-dopant single-EML F/P hybrid WOLED by doping a yellow phosphor (Ir(MDQ)2(acac)) into a blue fluorescent host (4P-NPD). Their devices exhibited external quantum efficiencies (EQEs) at a practical brightness of 1000 cd m^{2} that were much lower than the theoretical maximum EQE (EQEmax) of 20%, which can be attributed to the unipolar nature of 4P-NPD. The corresponding power efficiencies were also not yet high enough for practical applications. Meanwhile, Ma et al. reported a bipolar derivative containing triphenylamine and oxadiazole units for single-dopant single-EML WOLEDs, achieving a maximum current efficiency (CE) of 7.9 cd A^{1} (corresponding EQE of 5.2%). Most recently, higher performance with maximum total EQE of 26.6% and total efficiencies of 53.5 cd A^{1} and 67.2 lm W^{1} has been obtained by Zhang’ s group based on a blue fluorophore (DADBT) and orange phosphors (Ir(2-phq)3) hybrid single-dopant single-EML. Nevertheless, the dopant concentration of phosphors in these WOLEDs with single-dopant single-EML structure was very low (0.1%), which was difficult to control by vacuum deposition. Moreover, the color was very sensitive to the dopant concentration of phosphors, as a small variation in dopant concentration would result in a pronounced change in energy transfer between the emitting dopants. Therefore, developing improved dopant concentration design strategies for flexible manipulation has been considered one of the current key challenges for advancing single-dopant single-EML F/P hybrid WOLEDs.

In this work, we demonstrate for the first time an improved dopant concentration through the design and synthesis of an ideal saturated deep-blue fluorophore, N,N-diphenyl-4’ -(1,4,5-triphenyl-1H-imidazol-2-yl)-[1,1’ :4’ ,1’ ’ -terphenyl]-4-amine (DPTPA), which exhibits a high radiative exciton ratio and deep-blue emission. The single-dopant single-EML F/P hybrid WOLED has been fabricated with improved dopant concentration up to 0.9%, which does not follow the conventional principle of doping concentration regulation. The device still shows white emission and excellent electroluminescence (EL) performance with a low turn-on voltage of 2.6 V, maximum total EQE, CE, and PE of 22.3%, 53.7 cd A ^{1}, and 60.2 lm W ^{1}, respectively.

2.1 Synthesis and Crystal Structure

The synthesis of DPTPA (Fig. 1 [Figure 1: see original paper]) proceeded through the important intermediate 4’ -(diphenylamino)-[1,1’ :4’ ,1’ ’ -terphenyl]-4-carbaldehyde (3), which was synthesized by Suzuki coupling of 4-bromo-N,N-diphenyl-[1,1’ -biphenyl]-4-amine (2) and (4-formylphenyl)boronic acid using Pd(PPh3)2Cl2 as catalyst. Compound 2 was prepared by reacting (4-(diphenylamino)phenyl)boronic acid (1) with 1-bromo-4-iodobenzene in the presence of Pd(PPh3)2Cl2, K2CO3, and toluene. Finally, condensation of compound 3 with benzil resulted in the target compound DPTPA with high yield. Single crystals of DPTPA were obtained by layering ethanol onto a mixed solution of dichloromethane and n-hexane. As shown in the crystal structure, DPTPA adopts a highly twisted geometry containing an N-substituted benzene ring with the imidazole plane and triphenylamine (TPA) moiety, with dihedral angles of 97.6° and 23.3°, respectively.

2.2 Electrochemical and Thermal Properties

The electrochemical behavior of DPTPA was studied by cyclic voltammetry (CV). The HOMO level of DPTPA was determined from the onset potential of the first oxidation relative to ferrocene (Fig. 2a [Figure 2: see original paper]), and the LUMO level was calculated by adding the optical band gap to the HOMO level. The results are summarized in Table 1 . The first oxidation event is observed at 0.86 V (vs. Fc/Fc), which translates to a HOMO level of 5.26 eV. The thermal properties of DPTPA were investigated by TGA and DSC under a nitrogen atmosphere, as shown in Fig. 2b, with thermal data summarized in Table 1. The TGA measurement shows that DPTPA possesses high thermal stability above 400 °C, with a 5% weight loss (Td) observed at 469 °C. This value is more than 100 °C above the sublimation temperature and therefore proves high thermal stability. The DSC curve shows a glass-transition temperature (Tg) of 151 °C.

2.3 Theoretical Calculation

The geometry and electronic structures of DPTPA were obtained using density functional theory (DFT) at the B3LYP/6-31G(d,p) level in the Gaussian 03 program. The calculated dihedral angles between the imidazole and N-substituted benzene ring and triphenylamine moieties are 87.8° and 41.5°, respectively. The large twisted geometry configuration might prevent intramolecular extension of -electron delocalization and effectively suppress molecular packing in the solid state, which agrees with the crystal structure results. The electron density distribution of DPTPA is shown in Fig. 3 [Figure 3: see original paper]. The HOMO orbital of DPTPA is populated across the whole molecule, while the corresponding LUMO orbital mainly localizes on the linker and imidazole moieties. Large orbital overlap was found in the molecule, which would benefit charge carrier balance. The HOMO and LUMO levels are 4.9 eV and 2.24 eV, respectively. In contrast, the absolute values of the frontier orbital energies show a 0.36 eV discrepancy between experimental and theoretical data. As DFT calculations were performed only in the gas phase, solvent effects occurring during experimental characterization were not taken into account. Additionally, time-dependent (TD) DFT calculations were used to predict the lowest and higher excited states (triplets and singlets). Two degenerate triplet levels (T3 and T4) of 3.007 eV were found, which is lower than the S1 level (3.267 eV). The lower triplet states (T1 and T2) are 2.614 eV and 2.848 eV, respectively. All these low triplet states are ascribed to transitions from HOMO-1 to LUMO and from HOMO-2 to LUMO+1, both located on the same imidazole unit. The transitions between spatially non-overlapped HOMO-2 and LUMO orbitals are ascribed to S1 and T5, and their energy difference is zero. HOMO-2 and LUMO+2 also do not overlap, and the transitions between them are ascribed to S2 and T9, with their energy difference also being zero. These zero gaps between the excited singlet (Sn $ 1) and triplet (Tm $ 3) state may offer some advantage with respect to increasing the radiative singlet-state population from the nonradiative high triplet state generated by the "hot-exciton" process from T5 to S1.

2.4 Absorption and Photoluminescent Properties

The absorption and photoluminescent (PL) spectra in film and solution are shown in Fig. 4 [Figure 4: see original paper]. The absorption spectra in solution and film are nearly identical, implying small dipolar changes in the ground state in different environments. The absorptive spectra are characterized by an absorption maximum at max,abs = 351 nm, while the onset is determined at = 397 nm, which can be attributed to the → * transition. A weaker absorption band is observed at = 305 nm, assigned to the n → * transition. A small bathochromic shift occurs in the solid state. The PL spectra in solution feature similar profiles to those in film, with peaks located at ~435 nm and FWHM of 58 nm in solution and 46 nm in film, respectively. Moreover, DPTPA exhibits a pronounced Stokes shift of almost 80 nm, characteristic of large reorganization in the excited state. Additionally, PL spectra in various polarity solvents were recorded and are shown in Fig. 5 [Figure 5: see original paper]. With increasing solvent polarity from petroleum ether to acetonitrile, the PL spectra exhibit larger red shifts and broader shapes. Obviously, a large difference in dipole moment between the excited state and ground state indicates a CT state with large polarity that is usually stabilized in polar solvents, consistent with a transition of the excited state from the LE-state to an excited state with strong CT character. The dipole moments were calculated according to the Lippert-Mataga theory to be 21.7 and 5.4 D, respectively. These results are very similar to the pioneering work by Ma and colleagues. Considering that the two compounds in this work have similar molecular structures, we can confirm these emitters also show excited states of hybridized local and charge-transfer (HLCT) character. Furthermore, the phosphorescent spectra and PL decay characteristics were investigated, and the lifetime of DPTPA was observed to be 1.49 ns, which could be assigned to ordinary fluorescence.

2.5 Electroluminescent Properties

To investigate DPTPA’ s electroluminescence (EL) characteristics as a saturated deep-blue emitter, a device with the configuration ITO/HAT-CN (5 nm)/NPB (40 nm)/TCTA (5 nm)/DPTPA (20 nm)/TPBI (40 nm)/LiF (1 nm)/Al was fabricated. Here, ITO (indium tin oxide) and LiF/Al serve as the anode and cathode, respectively; 4,4’ -bis[N-(1-naphthyl)-N-phenylamino]biphenyl (NPB) and 1,3,5-tris(N-phenylbenzimidazol-2-yl)benzene (TPBI) are employed as the hole-transporting layer (HTL) and electron-transporting layer (ETL), respectively; while 4,4’ ,4’ ’ -tris(N-carbazolyl)triphenylamine (TCTA) serves as the electron-blocking layer (EBL).

The electroluminescent (EL) spectra, current density-voltage-luminance (J-V-L) characteristics, and CE-current density-PE plots of the device based on DPTPA are shown in Fig. 6 [Figure 6: see original paper], and key performance parameters are summarized in Table 2 . The device shows saturated deep-blue emission solely from DPTPA (peaked at 438 nm) with Commission Internationale de l’ Éclairage (CIE) coordinates of (0.15, 0.09). The EL spectra show little change as brightness increases from 10 to 10,000 cd m ^{2}. Furthermore, a low turn-on voltage (voltage required to give 1 cd m ^{2}) of 2.8 V was observed, mainly attributed to efficient charge injection and balanced charge recombination in the emitter layer. For EL efficiencies, the device exhibits impressive maximum EQE, CE, and PE of 3.81%, 3.45 cd A ^{1}, and 3.52 lm W ^{1}, respectively, and these efficiencies remain at 3.78%, 3.43 cd A ^{1}, and 3.12 lm W ^{1} at a brightness of 100 cd m ^{2}. Even at 1000 cd m ^{2}, these efficiencies still remain at 3.40%, 3.07 cd A ^{1}, and 1.88 lm W ^{1}. Considering the low fluorescent quantum yield (23.8%) and high device external quantum efficiency (3.81%), the performances are among the best for saturated deep-blue fluorescent OLEDs.

The corresponding theoretical value of the radiative exciton ratio was calculated by the following equation: _ext = · _r · _PL · _out, in which _r is the radiative exciton ratio, _ext is the external quantum efficiency, _out is the light out-coupling efficiency (ca. 20%), _PL is the intrinsic photoluminescence efficiency (ca. 23.8%), and is the recombination efficiency of injected holes and electrons, which is ideally 100% only if holes and electrons are fully balanced and completely recombined to form excitons. Thus, the _r value of the DPTPA device was calculated to be 80%, which breaks through the 25% limit of the radiative exciton ratio for conventional FOLEDs. Since no delayed fluorescence was observed from transient PL, and the EL luminance displayed a linear increase with increasing current density, the high radiative exciton ratio does not seem to be in accordance with mainstream mechanisms such as thermally activated delayed fluorescence (TADF) or triplet–triplet annihilation (TTA).

The hybrid WOLED with a single-EML architecture was fabricated with the same construction as the previous DPTPA-based device, with the only difference being that the DPTPA layer contains different concentrations of Ir(2-phq)3 from 0.1 to 0.9 wt%. As shown in Fig. 7a [Figure 7: see original paper], all devices with different Ir(2-phq)3 concentrations show extremely low turn-on voltages of 2.6 V and low driving voltages of 4.2 V (0.1 wt%), 3.8 V (0.3 wt%), and 3.5 V (0.65 and 0.9 wt%) at a practical brightness of 1000 cd m 2. The EL efficiencies are illustrated in Fig. 7b and c and listed in Table 2 . When the Ir(2-phq)3 concentration is ~0.9%, the hybrid WOLED exhibits maximum total EQE, CE, and PE of 22.3%, 53.7 cd A 1, and 60.2 lm W 1, respectively. As the Ir(2-phq)3 concentration decreases, the efficiencies reduce gradually. When the concentration is down to 0.1%, the efficiencies decline to 13.2% and 25.8 cd A 1, respectively, indicating that about 50% IQE could be harvested at this concentration.

As shown in Fig. 7d, warm white light emission can be realized when the Ir(2-phq)3 concentration is up to 0.9 wt%. The corresponding CIE coordinates show a moderate blue shift from (0.51, 0.41) to (0.43, 0.34) when brightness increases from 10 to 10,000 cd m 2. When the doping concentration was decreased to 0.6 wt%, the CIE coordinates changed from (0.49, 0.40) to (0.39, 0.31), with the white emission approaching standard white light at high brightness (ESI, S1†). When the concentration was reduced to 0.35 wt%, the CIE coordinates changed from (0.45, 0.36) to (0.29, 0.22), thus cold white light could be obtained at high luminance (ESI, S2†). At a concentration of 0.1%, which follows the conventional principle of doping concentration regulation, the spectra cannot show sufficient phosphorescence emission even at high luminance (ESI, S2†).

2.6 Discussion of Exciton Transfer Mechanism in High-Concentration Phosphorescent Dopant Devices

To clearly demonstrate the exciton transfer mechanism in high-concentration phosphorescent dopant WOLEDs, we combined the results of monochrome devices and WOLEDs. Considering the improved dopant concentration in the single-EML-SD F/P hybrid WOLEDs, it is suspected that the relatively high doping concentration should result from the blue emitters with HLCT character and thus "hot exciton" energy transfer from T5 to S1. Due to partial transfer from the triplet excited states to the singlet excited states of the blue emitter, the phosphorescent dopant cannot obtain sufficient energy to emit orange-red light in traditional devices with the same doping concentration, as shown in Fig. 8 [Figure 8: see original paper]. At the 0.9 wt% doping concentration, the fluorescence emission utilizes about ~60.8% of excitons, with about 55% of excitons coming from the triplet state T5 via the hot-exciton process, while the excitons transferred from singlets of the fluorescent to phosphorescent emitters can be calculated from the ratio of their fluorescent lifetimes (_F/ _P)^0.5 in the mixed film, giving a value of 19.2% from singlets of the fluorescent to phosphorescent emitters. The energy transfer from the triplet excited states of DPTPA to those of Ir(2-phq)3 is found to be only 20%. Accordingly, more than 55% of triplet excitons cannot transfer from DPTPA to Ir(2-phq)3. This means that insufficient energy can be transferred to the orange-red dopant, and the doping concentration should be improved to a high level. Furthermore, these results can not only make future mass production much easier to control accurately, but also represent a great breakthrough in device mechanism and design strategy.

3. Conclusions

In summary, we have presented a novel imidazole-based emitter material for high-efficiency fluorescent OLED and single-EML F/P hybrid WOLED devices. Notably, the WOLED still exhibited white emission with maximum total EQE, CE, and PE of 22.3%, 53.7 cd A ^{1}, and 60.2 lm W ^{1}, respectively, when the Ir(2-phq)3 concentration is up to 0.9 wt%, which does not follow the conventional principle of doping concentration regulation. The exciton transfer mechanism in high-concentration phosphorescent dopant devices was also discussed. Meanwhile, the devices with efficiencies of 16.9%, 38.3 cd A ^{1}, and 33.2 lm W ^{1} at 1000 cd m ^{2} also showed low efficiency roll-off, representing among the best results for EML-simplified F/P-WOLEDs. These studies provide a pathway to obtain high-performance F/P hybrid WOLEDs with a simple architecture and high doping concentration for easy manipulation.

4.1 Synthesis

All reagents, unless otherwise specified, were obtained from Alfa Aesar or Sigma-Aldrich and used without further purification. Some solvents were further purified before use (THF from sodium/benzophenone, methanol from CaH2). ^{1}H and ^{13}C NMR spectra were measured using a Bruker AV-400. Chemical shifts are expressed in parts per million (ppm), and splitting patterns are designated as s (singlet), d (doublet), m (multiplet), and br (broad). Coupling constants J are reported in Hertz (Hz). Elemental analyses were performed on a Vario EL elemental analysis instrument (Elementar Co.).

4.1.1 Synthesis of Compound 2

(4-(Diphenylamino)phenyl)boronic acid (2.89 g, 10.0 mmol), 1-bromo-4-iodobenzene (1.415 g, 5 mmol), and Pd(PPh3)4 (50 mg, 0.1 mmol) were suspended in toluene (30 mL) and K2CO3 (10 mL of 2 M aqueous solution), and the reaction was stirred at 90 °C for 24 h. The orange solution was extracted with CH2Cl2 (40 mL × 3 times), washed with water (20 mL × 3 times), dried over MgSO4, and evaporated to dryness. After drying under vacuum, the product was purified by silica gel column chromatography using CH2Cl2-petroleum ether (60-90 °C) (1:15) as eluent to afford a white solid. Yield: 1.68 g, 84%. MS (EI): m/z 399.1, 401.3 (M), which was in agreement with the literature.

4.1.2 Synthesis of Compound 3

Compound 2 (2 g, 5 mmol), 1-bromo-4-iodobenzene (0.75 g, 5 mmol), and Pd(PPh3)4 (50 mg, 0.1 mmol) were suspended in toluene (30 mL) and K2CO3 (10 mL of 2 M aqueous solution), and the reaction was stirred at 90 °C for 24 h. The orange solution was extracted with CH2Cl2 (40 mL × 3 times), washed with water (20 mL × 3 times), dried over MgSO4, and evaporated to dryness. After drying under vacuum, the product was purified by silica gel column chromatography using CH2Cl2-petroleum ether (60-90 °C) (1:1) as eluent to afford a yellow solid. Yield: 1.83 g, 86.1%. ^{1}H NMR (400 MHz, CDCl3) 10.01 (s, 4H), 7.99 (d, J = 7.86 Hz, 2H), 7.83 (d, J = 8.42 Hz, 2H), 7.75-7.70 (m, 4H), 7.55 (d, J = 8.42 Hz, 2H), 7.32-7.28 (m, 5H), 7.19-7.16 (m, 5H), 7.07 (t, J = 7.86 Hz, 2H). MS (EI): m/z 425.2 (M).

4.1.3 Synthesis of Compound DPTPA

A mixture of aniline (0.465 g, 5.0 mmol), benzil (0.21 g, 1.0 mmol), compound 3 (0.425 g, 1.0 mmol), ammonium acetate (0.308 g, 4.0 mmol), and acetic acid (10 mL) was refluxed under nitrogen in an oil bath. After 2 h, the mixture was cooled and filtered. The solid product was washed with an acetic acid–water mixture (1:1, 30 mL) and water, then dried in vacuum. The crude product was purified by silica gel column chromatography using CH2Cl2-petroleum ether (60-90 °C) (2:1) as eluent to afford a yellow solid. Yield: 0.613 g, 88.7%. ^{1}H NMR (400 MHz, CDCl3) 7.65-7.64 (m, 6H), 7.57-7.52 (m, 6H), 7.35-7.21 (m, 13H), 7.17 (d, J = 8.42 Hz, 8H), 7.13-7.11 (m, 2H), 7.06 (t, J = 7.28 Hz, 2H). MS (EI): m/z 692.1. Anal. calcd for C51H37N3: C, 88.54; H, 5.39; N, 6.07; found: C, 88.59; H, 5.22; N, 6.19%.

4.2 Device Fabrication and Measurement

The EL devices were fabricated by vacuum deposition of the materials at 5 × 10 Pa or below onto ITO glass with a sheet resistance of 20 Ω per square. Before device fabrication, the emitters were purified deeply by sublimation. All organic layers were deposited at a rate of 1.0-2.0 Å s ^{1}. The cathode was deposited with LiF (1 nm) at a rate of 0.1 Å s ^{1} and then capped with Al metal (100 nm) through thermal evaporation at a rate of 4.0 Å s ^{1}. The electroluminescence (EL) spectra were measured by a PR705 spectra scan spectrometer. The luminance-current density-voltage characteristics were recorded simultaneously with measurement of the Commission Internationale de l'Éclairage (CIE) coordinates by combining the spectrometer CS200 with a Keithley model 2420 programmable voltage-current source. All measurements were carried out at room temperature under ambient conditions.

Acknowledgements

This work was financially supported by the National Natural Science Foundation of China (21102156, 51273209, 514111004, and 91233116). ZYG and XO greatly appreciate financial support from the External Cooperation Program of the Chinese Academy of Sciences (no. GJHZ1219), Ningbo Natural Science Foundation (2014A610126), and Ningbo International Cooperation Foundation (2013D10013). SJS greatly appreciates financial support from the Ministry of Education (NCET-11-0159) and the Department of Education of Guangdong Province (2012KJCX0008). ZYG greatly appreciates financial support from the State Key Laboratory of Luminescence and Applications.

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Note: Figure translations are in progress. See original paper for figures.