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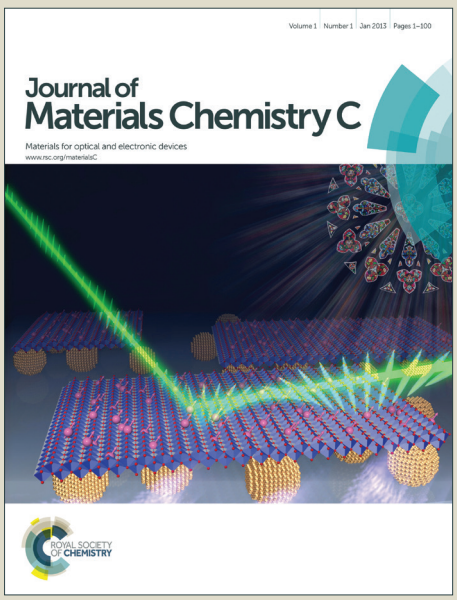

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Combination Effect of Polar Solvent Treatment on ZnO and Polyfluorene-Based Polymer Blends for Highly Efficient Blue-Based Hybrid Organic-Inorganic Polymer Light-Emitting Diodes

Cite this: DOI: 10.1039/x0xx00000x

Received 00th January 2012,
Accepted 00th January 2012

DOI: 10.1039/x0xx00000x

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Highly efficient blue-based hybrid organic-inorganic polymer light-emitting didoes were demonstrated by combination effect of polar solvent treatment on zinc oxide (ZnO) and hole trap using poly(9,9-dioctylfluorene) (F8):poly (9,9-dioctylfluorene-co-N-(4-butylphenyl)-diphenylamine (TFB) blend to balance the charge carrier transport, leading to maximize the recombination of charge carriers and enhance the device efficiency.

Great interest for polymeric light-emitting diodes (PLEDs) has been increasing because of low cost and large area fabrication by solution processing and mechanical flexibility for their potential use in solid-state lighting and flexible display applications.[1-3] The balanced Red-Green-Blue (RGB) based device efficiencies and stability are required to realize white PLEDs specially for solid-state lighting and full-color display. However, the efficiencies of blue-based PLEDs fall far behind, compared to those of the green and red PLEDs. Therefore, highly efficient blue-based PLEDs should be developed to realize RGB display application.[4]

Inverted-type polymer light-emitting diodes (IPLEDs) have been studied for the substitute of conventional-type PLEDs due to exceptionally good air-stability of devices using metal-oxide as charge transport layers and gold (Au) with high work function as an anode.[5-16] However, the efficiencies of IPLEDs without surface modifications on solution processed n-type metal-oxide layer are very low due to the unbalanced electron and hole carrier injection/transport without charge blocking behavior.[5-8] Previous studies of IPLEDs demonstrated that molybdenum oxide (MoO_3) provides ohmic hole injection from Au anode to organic semiconductor and electron injection is more difficult due to larger energy barrier between n-type metal-oxide and active layer in PLEDs.[17] Therefore, efficient electron injection with blocking the hole carriers is required to maximize the recombination of charge carriers near the interface of metal-oxide and emissive layer and enhance the efficiency in IPLEDs.[5] For excellent hole blocking as well as electron injection efficiency, surface modifiers such as conjugated polymer electrolyte (CPE),[11,12] barium hydroxide ($Ba(OH)_2$),[14] and cesium carbonate (Cs_2CO_3)[8,16] on n-type metal-oxides have been utilized to balance charge carrier injection/transport and maximize the recombination of holes and electrons near the interface of metal-oxide and emissive polymer, leading to improved efficiency of IPLEDs. However, there are still room for enhancement of the efficiency of IPLEDs by reducing significant exciton quenching near the interface of metal-oxide and emissive polymer with excellent hole blocking and electron injection efficiency.[14] Moreover, addition of a trap material into emissive layer for highly efficient PLEDs has been demonstrated to balance charge-carriers injection and transport with blocking one of charge-carrier due to the difference energy levels between two materials, leading to improvement of device efficiency.[18-21]

In this study, we demonstrate the significant enhancement of blue-based IPLEDs efficiency by using amine-based polar solvent treatment on zinc oxide (ZnO) and introducing a hole trap material into emissive polymer. The ethanolamine/2-methoxyethanol (EA+2-ME) co-solvent treatment on ZnO layer was done to improve minority carriers of electron injection with blocking majority carriers of hole, balance and maximize the recombination of charge carriers, and enhance the device efficiency. Poly (9,9-dioctylfluorene-co-N-(4-butylphenyl)-diphenylamine (TFB) was added as a hole trap in emissive polymer material and PLED efficiency increased without influencing optical properties.[21]

ZnO film preparation. ZnO solution was prepared by mixing 0.4 mL of diethylzinc solution and 0.8 mL of tetrahydrofuran (THF) in glove box. The solution was filtered by 0.2μm□hydrophilic filter. The ZnO solution was spin-cast at 3,000 rpm on cleaned FTO (fluorine doped tin oxide) substrates in air and then dried at 110 °C for 10 min. The details are shown in ref [15].(삭제요청)

IPLED fabrication. FTO substrates were washed by ultra-sonication in dilute water, acetone and isopropyl alcohol. ZnO layer was deposited on the FTO substrates by spin coating method. The ZnO films on substrates were treated by UV-ozone for 12 min. Subsequently, EA +2-ME co-solution was spin-coated at 2,000 rpm on ZnO layer and annealed at 110°C for 10min. Co-solvents was prepared by mixing 2.85mL of 2-methoxyethanol (2-ME) and

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0.15mL of ethanolamine (EA). Poly(9,9-dioctylfluorene) (F8) emissive semiconductor was spin-casted at 2,000 rpm (12 mg mL^{-1} *p*-xylene solutions) and annealed at 90 °C for 1 hour in glove box. Finally, a 10 nm-thick MoO_3 was thermally evaporated at deposition rate of 0.01 nm s^{-1} and a 100 nm-thick Au was thermally evaporated at deposition rate of 0.01 nm s^{-1} to 10 nm and 0.05 nm s^{-1} from 10 nm to 100 nm.

Loss Coefficient Experiment. For loss coefficient measurement, the third-harmonic of an Nd:YAG laser (Quanta Ray INDI series, Spectra-Physics Co. Ltd.,) was used for an optical excitation at λ = 355 nm. The repetition frequency and purse width were 10 Hz and 6 ns, respectively. The rectangular-shaped laser beam (2 mm * 370 μm) was focused on the edge of sample surface and the output emission was detected from sample edge by a multi-channel spectrometer (HR 2000+, Ocean Optics, Inc).[22] In particular, loss coefficient measurement was conducted by measuring the emission intensity with changing the unpumped region between the edge of the excitation stripe and the substrate edge. [23]

Time-Correlated Single Photon Counting (TCSPC) Characterization. The exciton lifetime was determined by the time-correlated single photon counting (TCSPC) technique. The details are shown in ref [3].

Photoluminescence (PL) Charaterization. The photoluminescence spectra of F8 films with or without polar solvent treatment on a quartz substrate were obtained on a Cary Eclipse spectrofluorometer with a xenon lamp as an excitation source (Edinburgh Instruments, Ltd.).

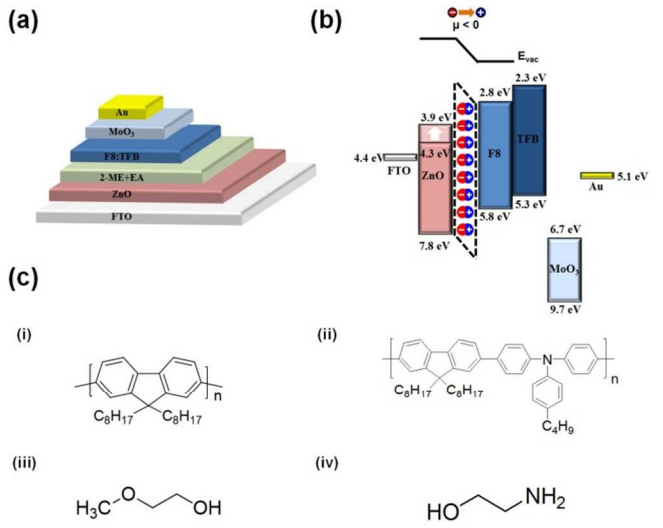

Fig. 1 (a) IPLED structure, (b) energy level diagram for all the layers in IPLED. (c) Chemical structures of (i) F8, (ii) TFB, (iii) 2-methoxyethanol and (iv) ethanolamine.

In Fig. 1 (a)-(c) represents the device schematic, energy level diagram of IPLEDs using polar solvent treatment including amine groups on ZnO and the blend of TFB and F8 and the chemical structures of (i) F8, (ii) TFB, (iii) 2-methoxyethanol and (iv) ethanolamine. In particular, a significant energy barrier (~ 1.5 eV) between the lowest unoccupied molecular orbital (LUMO) of F8 and the conduction band (CB) of ZnO layer exists and interfacial engineering are required to reduce the energy barrier and balance hole–electron injection/transport in hole-dominant IPLED with MoO_3 layer.[24,25] Recently, B. R. Lee et al. has showed that amine-based polar solvent treatment on ZnO layer tuned work functions of ZnO layer by creating interfacial negative dipole and then CB of ZnO was well matched with the LUMO of polymeric semiconductor with reduction of electron extraction barrier and suppression of bimolecular recombination leaded to the enhanced inverted-polymer solar cell (IPSC) efficiency.[26] In our IPLEDs, polar solvent treatment using ethanol amine on ZnO was conducted and the reduction of energy barrier between emissive polymer and ZnO was expected by negative dipolar effect, as shown in Fig. 1 (b). Moreover, small amount of TFB into F8 with large energy gap (~ 0.5 eV) between the highest occupied molecular orbital (HOMO) level of TFB and F8 was used as a hole trap to balance charge carrier and enhance the device efficiency.[27,28]

The IPLEDs with polar co-solvent treatment on ZnO were optimized through differnet volume concentration and the optimized volume concentraion of EA in 2-ME is 5.00 vol. % in Fig. S1(a)-(e) and Table S1, which is volume ratio of EA to 2-ME is 1:19. From the result, it was confirmed that interfaical dipole layer of optimized IPLED was more thicker than that of optimized IPCS.[26] Thicker interfacial layer effectively blocked hole carriers and reduced exciton quenching near the interface of ZnO and emissive layers even though electron injection between ZnO and emissive layer was not maximizely enhanced. For efficient IPLED, both excellent hole blocking with suppression of exciton quenching and efficient electron injection should be considered. The optimized IPLED with 5.00 vol. % of polar co-solvent treatment showed the highest device performance with a luminance of 1,640 cd/m^2, luminous efficiency of 1.55 cd/A, and external quantum efficiency of 1.28 %. Luminous and external quantum efficiency of optimized IPLED with 5.00 vol. % of polar co-solvent treatment increased by forty-four and thirty-six times respectively by using interfacial dipole effect by polar co-solvent treatment on ZnO in comparison with the reference IPLEDs, as shown in Table S1. Remarkable thing is available by observing the electroluminescence (EL) spectra of the reference IPLED and the IPLEDs with polar co-solvent treatment at 6V in Fig. S1 (f). The EL spectra of the IPLEDs with polar co-solvent treatment do not change in comparison with the EL spectrum of reference IPLED and EL spectra of optimized IPLED (FTO/ZnO/2-ME+EA (5.00 vol. %)/F8/MoO_3/Au) do not change even though operating voltage increases up to 10 V, as shown in Fig S4 (a).

The current density and luminance versus voltage (*J-V-L*) and device efficiency of polar solvent treated (5.00 vol. %) IPLEDs with different concentration of TFB into F8 are presented in Fig. 2. The thickness of active layer is 1,000 nm. *J-V-L* curves show that driving voltage increases the amount of TFB into F8 due to the existence of a hole trap resulting from difference between HOMO level of F8 and TFB. Device efficiency with addition of small amount of TFB into F8 was enhanced by comparison with that of the reference IPLEDs due to maximized charge-carrier balance by a hole trap, as shown in Fig. 2 (c)-(e). Hole current density of device decreased with increasing wt. % of TFB in F8 and the efficiency of IPLEDs with addition of TFB into F8 was optimized through differnet concentration and the optimized concentration of TFB in F8 is 5.00 wt. %, which is weight ratio of TFB to F8 is 1:19. The optimized IPLED with 5.00 wt. % of TFB in F8 showed the highest device efficiency with luminous efficiency of 3.42 cd/A, and external quantum efficiency of 3.29 %, representing more two folds increased luminous efficiency compared to that of IPLED without addition of TFB in F8. The improved device efficiency is originated from maximizing charge carrier balance by using TFB as a hole trap. The detailed IPLED performance is shown in in Table 1. The EL spectra

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of the polar solvent treated IPLEDs without and with addition of TFB in F8 at 15 V are shown in Fig. 2 (f) and the EL spectra of the IPLEDs with addition of TFB in F8 are almost the same as the EL spectrum of IPLED without addition of TFB in F8, which means that addition of TFB as a hole trap into F8 does not influence the EL spectrum of F8. Moreover, EL spectra of device (FTO/ZnO/2-ME+EA (5.00 vol. %)/F8:TFB (5.00 wt. %)/MoO_3/Au) also do not change significantly even though operating voltage increases up to 25 V, as shown in Fig S4 (b).

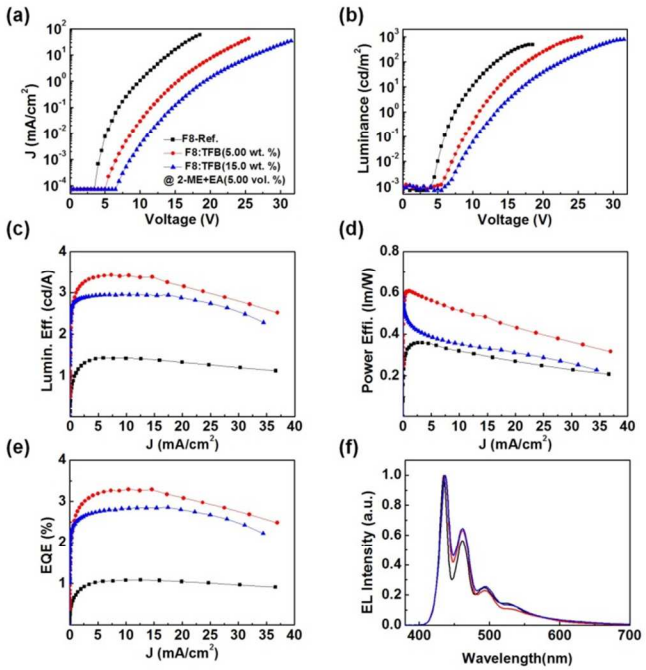

Fig. 2 *J-V-L* characteristics of FTO/ZnO/2-ME+EA (5.00 vol. %)/F8:TFB/MoO_3/Au with different concentration ratio of TFB in F8 .(a) Current density versus voltage, (b) luminance versus voltage, (c) luminous efficiency versus current density, (d) power efficiency versus current density, (e) external quantum efficiency versus current density and (f) electroluminescence spectra.

Table 1. Summarized device performances of FTO/ZnO/2-ME+EA (5.00 vol. %)/F8:TFB/MoO_3/Au with different concentration ratio of TFB in F8.

Device configuration (Thickness of F8: 1μm)	L_{max} [cd/m^2] @ bias	LE_{max} [cd/A] @ bias	PE_{max} [lm/W] @ bias	EQE_{max} [%] @ bias
FTO / ZnO / 2-ME+EA (5.00 vol. %) / F8 / MoO_3 / Au	490 (18.5 V)	1.42 (14.0 V)	0.36 (12.0 V)	1.09 (14.5 V)
FTO / ZnO / 2-ME+EA (5.00 vol. %) / F8:TFB(5.00 wt. %) / MoO_3 / Au	960 (25.5 V)	3.42 (20.0 V)	0.61 (15.5 V)	3.29 (21.0 V)
FTO / ZnO / 2-ME+EA (5.00 vol. %) / F8:TFB (15.00 wt. %) / MoO_3 / Au	790 (31.5 V)	2.95 (26.0 V)	0.55 (13.0 V)	2.86 (28.5 V)

Hole-only devices were fabricated to confirm hole-trap due to difference between HOMO level of F8 and TFB. *J-V* characteristics of hole-only device with F8 and F8:TFB blends are shown in Fig. S2. Hole current density of hole-only device with F8:TFB blends was significantly reduced with increasing weight ratio of TFB in F8. Lu et al. demonstrated that the electron trasport of F8 is very poor, compared with the hole trasport, and current density of electron was three orders lower than that of hole in IPLEDs with ITO/ZnO/CS_2CO_3/Aryl-F8/MoO_3/Au.[21] Therefore, introduction of hole trap was required to reduce the current density of hole in comparison with the current density of electron and CS_2CO_3 interfacial layer is required to enhance the electron injection with hole-blocking behavior to balance the charge carriers for maximizing the recombination of the charge carriers. As the same results as ref [21], hole current density of hole-only device decreased with increasing wt. % of TFB in F8 and the use of optimized small amount (5.00 wt. %) of hole-trap in active polymer increased higher device efficiency by balacing hole and electron and maximizing the recombination of two charge carriers in our IPLEDs.

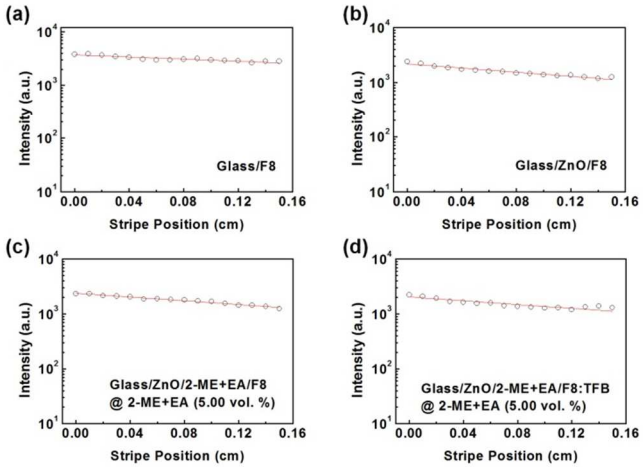

Fig. 3 Dependence of the emission intensity at λ_{ASE} on the length of the unpumped region between the edge of the excitation stripe and the substrate edge of the waveguide. The data is fitted with equation $I = I_0 \exp(-\alpha x)$.

Table 2. Loss coefficient of F8 and F8:TFB films with different configurations.

Sample configuration	Glass / F8	Glass / ZnO / F8	Glass / ZnO / 2-ME+EA / F8	Glass / ZnO / 2-ME+EA / F8:TFB
Loss coefficient (cm^{-1})	2.29	4.34	3.96	3.97

Radiative excition can be generated near the interface of metal-oxide and emissive layer due to hole-dominant IPLED with MoO_3 layer and the reduction of exciton quenching near the interface between ZnO and emisive layer is critical for highly efficient IPLEDs. To characterize more the influenece of optical properties of films with polar solvent treatment and the addition of TFB, we utilized shifting excitation stripe measurement, which is the method to measure the emission intensity as a function of lateral displacement away from the edge of substrates.[29] The emission intensities of samples were measured by changing the length of the unpumped region between the substrate edge of the waveguide and the edge of the excitation stripe. The loss coefficient data for F8 and F8:TFB films in different configuration are shown in Fig. 3. The loss coefficient for F8 and F8:TFB films are summarized in Table 2.[29] The value of loss coefficient for F8 (2.29 cm^{-1}) in this study is lower than that of loss coefficient for F8 (9.7 cm^{-1}) in ref [22] because of

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different experimental conditions such as different batch and thickness of F8, and different Nd:YAG laser with shorter pulse width. The loss coefficient of glass/ZnO/2-ME+EA (5.00 vol. %)/F8 (3.96 cm^{-1}) was also lower than that of glass/ZnO/F8 (4.34 cm^{-1}) due to the reduction of optical losses by solvent treatment on ZnO layer. This shows that 2-ME+EA treatment on ZnO decreases optical losses caused by exciton quenching because defect sites such as oxygen vacancy and the roughness of surface by solution processed ZnO layer decrease by 2-ME+EA treatment. The results of exciton lifetime measured by time-correlated single photon counting (TCSPC) and photoluminescence (PL) intensity measurements also support that polar solvent treatment on ZnO layer supresses exciton quenching by observing longer exciton life time and higer PL intersity for ZnO/2-ME+EA (5.00 vol. %)/ F8 sample, as shown in Figure S3 and Table S2. Moreover, the loss coefficient of glass/ZnO/2-ME+EA (5.00 vol. %)/ F8:TFB was almost the same as the loss coefficient of glass/ZnO/2-ME+EA (5.00 vol. %)/ F8 and it also indicated that the addition of TFB did not change optical properties.

It was also confirmed that electron injection was enhanced by 5.00 vol. % of polar co-solvent treatment on ZnO layer by comparing the magnitude of current densities of electron only devices (ITO/ZnO/F8/LiF/Al) with and without 5.00 vol. % of polar co-solvent treatment at the same voltage, as shown in Fig. S5.

Long-term operational stability is also important issue for commercialization of PLEDs. The stability of PLEDs can be determined by various factors such as dark spot caused by diffusion of oxygen and moisture into the device or morphological defects by phase separation.[30,31] In particular, It is well known that the stability of blend device is quite low due to phase separation between the polymers caused by thermal degradation. In this study, the device stability was investigated by monitoring the change of luminance and luminous efficiency at fixed luminance of 100 cd/m^2 for about 300 hours in Fig. 4. The operational stability test of the encapsulated device was conducted under ambient air conditions. The F8-based and F8:TFB blend-based device performances with polar solvent treatment showed almost the same as initial values even after 300 hours. It indicates that the interaction between polar solvent and ZnO is still consistent and no degradation occurs by phase separation between polymer blends after long time operation.

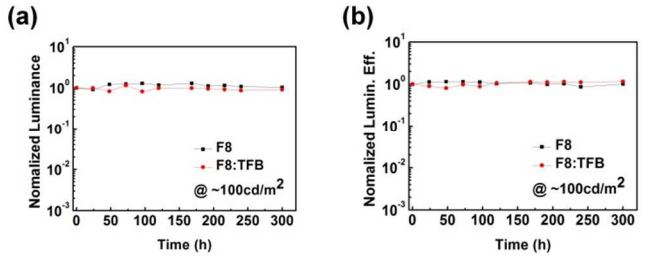

Fig. 4 Normalized (a) luminance and (b) luminous efficiency of FTO/ZnO/2-ME+EA (5.00 vol. %)/F8/MoO_3/Au (closed-rectangular black-line) and FTO/ZnO/2-ME+EA (5.00 vol. %)/F8:TFB (5.00 wt. %)/MoO_3/Au (closed-round red-line) as a function of storage time for IPLEDs with 2-ME+EA co-solvent treatments on ZnO layer in air under ambient conditions with encapsulation.

Conclusions

We demonstrated a highly efficient blue-based hybrid organic-inorganic IPLED with 2-ME+EA polar solvent treatment on ZnO layer and introduction of small amount of TFB as a hole trap in F8 emissive polymer. It was confirmed that device efficiency was enhanced by introducing amine-based polar solvent treatment on ZnO and optimized F8:TFB blend by observing increase of luminous efficiency by forty-four times using interfacial dipole layer and more than double times with the addition of TFB into F8. Amine-based polar solvent treatment on ZnO layer effectively blocked majority carriers of hole, reduced exciton quenching and enhanced the electron injection by reducing contact barrier with interfacial dipole effect, leading to higher device efficiency. Moreover, the introduction of adequately small amount TFB as a hole trap into F8 emissive layer maximized the recombination of charge carriers and also increased the device efficiency. In addition, optical properties of interfaces between ZnO and emissive polymer were analyzed measuring optical loss coefficient, exciton life time, and PL intensities measurement and the loss coefficient of F8 film with polar co-solvent treatment on ZnO decreased, compared to those of F8 film without polar co-solvent treatment ZnO, while the introduction of small amount of TFB into F8 did not affect optical properties with almost the same values of loss coefficient as those of F8. We also confirmed that the device performance with solvent treatment and introduction of TFB was maintained under ambient air condition for a long time. This verifies that the interaction between polar solvent molecules and ZnO is still constant and there is no phase separation between polymer blends caused by thermal degradation in blend device.

We thank Cambridge display Technology (CDT) for providing us with F8BT and TFB polymer. This work was supported by Korea Institute of Materials Science (KIMS) and the National Research Foundation of Korea (NRF) grant (No.2012R1A2A2A06046931, 2009-0093020) by the Ministry of Science, ICT & Future Planning (MSIP), Korea. This study was also financially supported by the KIST-UNIST partnership program (2.130400.01). M. H. Song thanks the support by UNIT-Samsung Display Co. OLED center.

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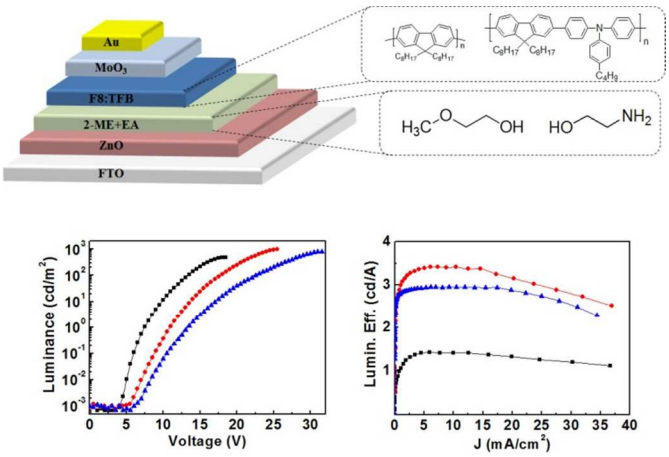

Device efficiency is enhanced by combination effect of polar solvent treatment on ZnO and hole trap using F8: TFB blend.

Journal of Materials Chemistry C Accepted Manuscript