



## Article

# Nozzle Printed-PEDOT:PSS for Organic Light Emitting Diodes with Various Dilution Rates of Ethanol

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**Abstract:** In this study, we investigated the ink formulation of poly(3,4-ethylenedioxythiophene) polystyrene sulfonate (PEDOT:PSS) as the hole injection layer (HIL) in an organic light emitting diode (OLED) structure. Generally, in a PEDOT:PSS solution, water is incorporated in the solution for the solution process. However, the fabrication of thin film which contained the water, main solvent, could not easily form by using printing technology except spin-coating process because of the high surface tension of water. On the other hand, mixing PEDOT:PSS solution and ethanol (EtOH), a dilution solvent, could restrain the non-uniform layer that forms by the high surface tension and low volatility of water. Therefore, we printed a PEDOT:PSS solution with various concentrations of EtOH by using a nozzle printer and obtained a uniform pattern. The line width of PEDOT:PSS diluted with 90% (volume ratio) ethanol was measured as about 4 mm with good uniformity with a 0.1 mm nozzle. Also, imaging software and a scanning electron microscope (SEM) were used to measure the uniformity of PEDOT:PSS coated on a substrate. Finally, we fabricated a green phosphorescent OLED device with printed-PEDOT:PSS with specific concentrations of EtOH and we achieved a current efficiency of 27 cd/A with uniform quality of luminance in the case of device containing 90% EtOH.

**Keywords:** nozzle printing; PEDOT:PSS; ethanol; dilution rate; OLED; grey scale method

## 1. Introduction

Printed electronics are an important new technology for emerging markets. A printing process can be applied for various low-cost organic electronic device applications such as organic thin film transistors (OTFTs) [1], organic solar cells (OSCs) [2], radio frequency identification (RFID) [3], organic sensors [4], and organic light emitting diodes (OLEDs) [5]. Printing technology such as inkjet and nozzle printing process-based OLEDs have received huge interest from many companies and research groups. In particular, many researchers have tried to fabricate printed OLEDs by inkjet printing [6,7], electrohydrodynamic (EHD) jet [8], organic-vapor jet [9], and so on. Typically, various processes with chemical vapor deposition (CVD), sputtering, vacuum thermal evaporation, and spin-coating are used for the fabrication of organic thin films. The above process includes the steps of deposition, exposure, and developing to fabricate a pattern on a substrate. The nozzle printing process for manufacturing electronic devices, in particular, has many advantages including a fast and direct printing method on a substrate in a general atmosphere by using a simple ink solution. The nozzle printing process deposits the material selectively and simply and it can be applied to a large area and even a flexible substrate for the fabrication of OLEDs.

Although printed based OLED technology has many advantages including a simple process, low cost, and the possibility of large area processing, there is a critical bottleneck of film uniformity.

In relation to this, Han et al. investigated a method to prevent coffee ring and pinhole defects by using a co-solvent system with a different de-wetting velocity mechanism [10]. Poly(3,4-ethylenedioxythiophene) polystyrene sulfonate (PEDOT:PSS) is currently the most popular and important hole injection layer (HIL) for the fabrication of solution process based OLED device structures [3]. Generally, it is difficult to fabricate a pristine PEDOT film in a neutral state because pristine PEDOT cannot be dissolved in most conventional organic solvents. Therefore, to apply a solution process to printing, a stable PSS blend system that can approach to process easily was developed [11,12]. The development of PEDOT:PSS, HIL materials has been studied extensively in the solution based OLED industry. Kido et al. successfully fabricated an all solution tandem white OLED by using various types of PEDOT:PSS [13].

In this study, we suggested a deposition methodology for PEDOT:PSS with various concentrations of EtOH dilution solvent by using a nozzle stream jet printer. In addition, we considered the pattern quality about defect free printing. To prevent defects such as a de-wetting zone and pinholes, we considered the properties of the solution, specifically, the ink formulation, viscosity, surface tension, evaporation rate, and residual containing solvent volume in the PEDOT:PSS solution. Finally, to analyze the film uniformity of the PEDOT:PSS/EtOH solution, we used imaging software and a scanning electron microscope (SEM) measurement system. We then fabricated a green phosphorescent OLED device with nozzle printed-PEDOT:PSS containing various amounts of EtOH, and obtained high I-V-L efficiency of an OLED owing to enhanced film uniformity of the PEDOT:PSS layer.

## 2. Experimental

### 2.1. Materials

A sputtered-indium tin oxide (ITO) glass substrate with sheet resistance of  $20 \Omega/\square$  was used as the anode electrode for observing the nozzle printed PEDOT:PSS pattern. To fabricate a typical HIL, we purchased the PEDOT:PSS (CH8000) from Clevios<sup>TM</sup> in Heraeus, Germany. Also, we purchased the following various organic materials for OLED devices: 4,4'-bis(N-carbazolyl)-1,1'-biphenyl (CBP), tris[2-(p-tolyl)pyridine]iridium(III) [Ir(mppy)<sub>3</sub>] as the host/dopant, green emission materials, and 2,2',2''-(1,3,5-benzinetriyl)-tris(1-phenyl-1-H-benzimidazole) [TPBi] as the electron transport layer (ETL) from OSM Corporation, Korea. Furthermore, 99.5% ethanol (HPLC grade) was purchased from Samchun Chemical Korea as the dilution solvent for blending with PEDOT:PSS.

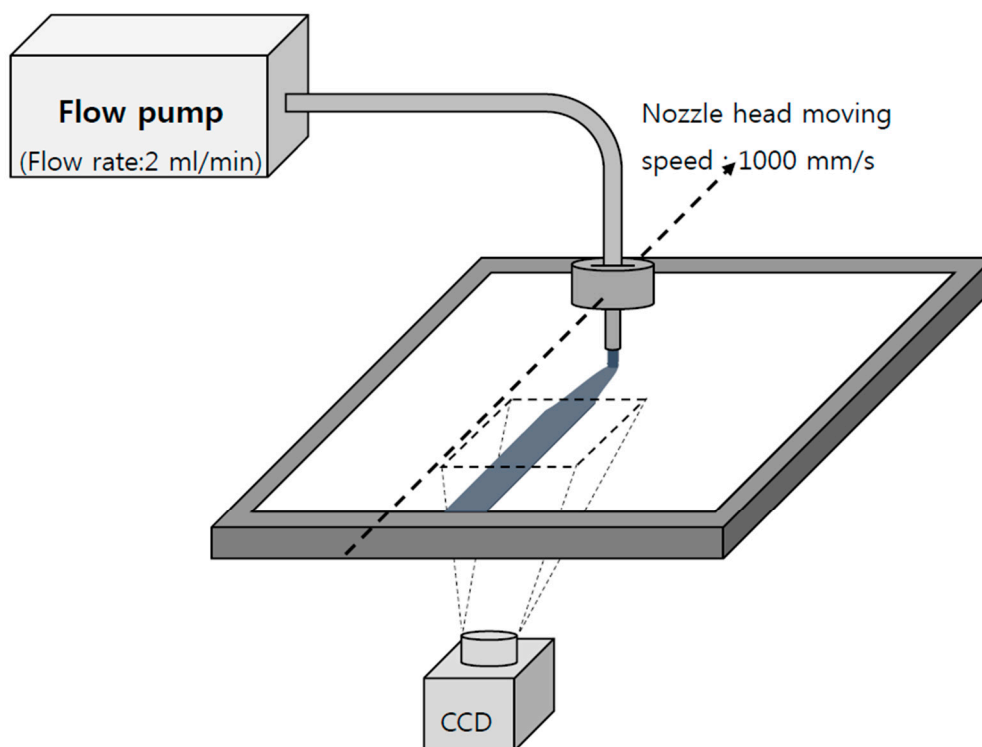
### 2.2. Cleaning Substrate

To fabricate the PEDOT:PSS pattern by using a nozzle printer and observe the variation of the film behavior on top of the ITO substrate, a cleaning process is critically important. First, the ITO/glass substrates were washed carefully by a surface-active agent solution. They were then cleaned by an ultra-sonication process in acetone and isopropanol of 1:1 (*v/v* ratio) for a 1 h. Furthermore, they were boiled in refreshed isopropanol solvent at 250 °C for 20 min. Finally, they were baked in a vacuum chamber at 180 °C for 30 min. A patterned-ITO glass substrate for the OLED device was also prepared with the same cleaning process as described above.

### 2.3. Fabrication of PEDOT:PSS Line Pattern

The nozzle printer (prototype made by Device Eng, Asan-si, Korea) used in the present study consists of a stage, a motored nozzle head, a liquid pump (Havard apparatus-PHD ULTRA<sup>TM</sup>CP, Havard, MA, USA), and a charge-coupled device (CCD) camera (Basler-acA 1300-30 gm, Ahrensburg, Germany), as shown in Figure 1. The stage of the nozzle printer can accommodate a substrate of 100 mm × 100 mm size. The CCD camera is located under the stage to capture the coating process through a transparent substrate such as an ITO/glass substrate. The motored nozzle head can be equipped with various nozzle types and move with a maximum speed of 1000 mm/s to the y-axis direction. The used SUS nozzle has an external diameter of 0.23 mm and an internal diameter 0.1 mm,

and it is connected to the liquid pump that helps to ensure a constant flow through the nozzle. In this experiment, the nozzle moving speed and flow rate of the coating solution were fixed at 1000 mm/s and 2 mL/min, respectively.



**Figure 1.** Schematic diagram of experimental set up with Nozzle printer.

The PEDOT:PSS (Heraeus CH8000) solution employed in this study is one of the most commonly used HIL materials and has high conductivity, excellent stability, and good transparency in the visible region. It was used to evaluate the printing process and thickness uniformity according to the EtOH volume ratio in aqueous solution. In this study, we prepared ink samples of pristine PEDOT:PSS (0% EtOH) and various diluted-PEDOT:PSS samples mixed with EtOH (10, 20, 30, 40, 50, 60, 70, 80, 90%). The ITO glass substrate was then treated by O<sub>2</sub> plasma to improve its wettability. Finally, we fabricated a PEDOT:PSS line pattern on the ITO substrate with the following nozzle printing conditions and ink formulation:

- Printing speed of 1000 mm/s
- Ink injection rate of 2 mL/min
- Stage temperature of 24 °C (±0.5 °C)
- Atmosphere of 50% (±5%) relative humidity
- PEDOT:PSS baking condition of 100 °C/30 min after solvent evaporation

#### 2.4. Fabrication of OLED Device

We fabricated the device including nozzle printed-PEDOT:PSS as HIL in an OLED structure. The PEDOT:PSS, HIL material, was deposited on top of the patterned ITO/glass substrate (substrate size of 1 inch × 1 inch and pixel size of 2 mm × 2 mm) by using the nozzle printing process. In the case of the line patterned PEDOT:PSS for an OLED, the experimental group, we prepared representative samples composed of PEDOT:PSS with EtOH (0, 40, 90%). After the annealing process of HIL, green phosphorescent EML materials consisting of CBP as the host and Ir(mppy)<sub>3</sub> as the dopant (10% doped (30 nm)) were thermally vacuum deposited in a  $6.0 \times 10^{-7}$  Torr atmosphere.

Also, 2,2',2''-(1,3,5-Benzinetriyl)-tris(1-phenyl-1-H-benzimidazole) (TPBi, 25 nm) as the ETL and lithium fluoride (LiF, 0.5 nm):aluminum (Al, 100 nm) as the metal layer were evaporated. The entire thermal vacuum deposition process was performed at a deposition rate of 1 Å/s.

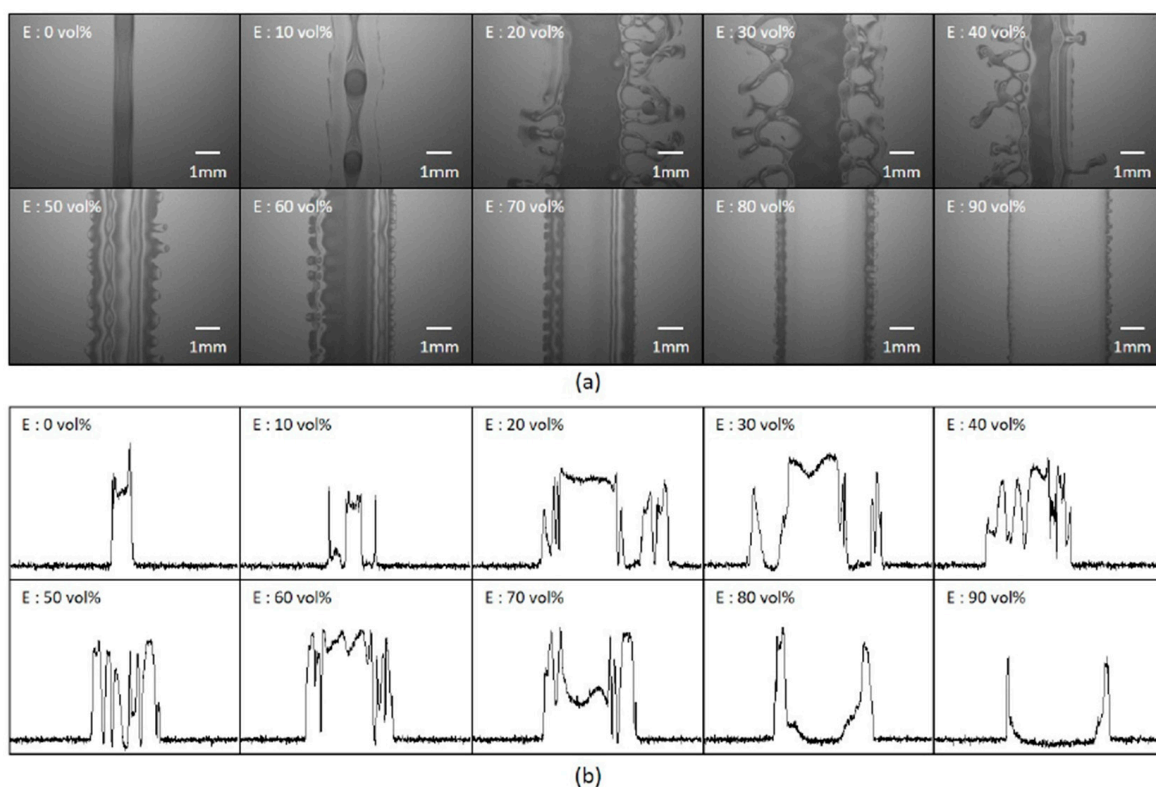
### 2.5. Measurement

To observe the PEDOT:PSS film uniformity, we used the grey-scale method [14] and a SEM measuring system. The I-V-L characteristics of the OLED devices were then analyzed by an IVL measurement system (PR-655, Photo Research Co., New York, NY, USA) and a Keithley 2400 (Tektronix, OR, USA).

## 3. Result and Discussion

In this study, we developed a nozzle printed PEDOT:PSS (as the HIL) wide line pattern for application to an OLED device. To this end, we attempted to determine the optimum PEDOT:PSS material as a HIL thin film with various concentrations of EtOH, a dilution solvent that is added to PEDOT:PSS, by using nozzle printer equipment. We described the behaviors through CCD camera images of the PEDOT:PSS pattern according to the dilution rate of EtOH, as presented in Figure 2a. The images were taken by a CCD camera after the samples were dried completely. As seen in Figure 2a, the narrowest line was formed in the sample with EtOH 0%. Furthermore, when the volume rate of EtOH was increased, the line width was greater than that of the pristine PEDOT:PSS based pattern. Also, we observed pattern contrast of grey color intensity with the pattern width as well as the behavior of the spreading, shrinking, and drying mechanism. As seen in these images, the pattern with 10% EtOH showed a bulging motion through the spreading and shrinking process and different surface tension between hetero-solvent systems. Also, from the point of line edge, we predicted that the shrinking mechanism occurred after the pinning effect. Through the samples with a ratio of EtOH from 20% to 40% showed an instable branched type pattern. Though the above group, the samples with EtOH of 20~40%, had a poor film shape, and the film uniformity of the PEDOT:PSS layer was enhanced with an increase of the volume rate of EtOH (50~90%). Finally, in the case of the 90% EtOH ratio in PEDOT:PSS, we obtained good spreading and a uniform film of PEDOT:PSS pattern. While the coffee ring effect still remains at the line edge, line width of almost 4 mm could cover a 2 mm × 2 mm pixel area in the OLED device, and therefore entire film planarization was not necessary in this study. Also, as seen in the image of Figure 2a, the line width increased the EtOH ratio from 0% to 20%, and the line width reduced when the EtOH quantity ranged from 30% to 40%. Finally, in the case of the 50~90% range, the pattern width increased again. This indicated that when the EtOH volume concentration in the PEDOT:PSS solution was reduced from 90% to 50%, the printed line width was also reduced. However, when the EtOH volume concentration in the PEDOT:PSS solution was decreased from 40% to 0%, the viscosity decreased and the surface tension increased. When the EtOH volume concentration in the PEDOT:PSS solution was decreased from 40% to 20%, the decrease in viscosity was greater than the increase in surface tension, and hence the spreading proceeds faster and the line width was enhanced. However, when the EtOH volume concentration in the PEDOT:PSS solution was less than 20%, the value of the surface tension became larger, and therefore spreading was suppressed and the width was reduced. This phenomenon of variation was induced by the dynamic viscosity of the binary system [15]. Generally, PEDOT:PSS contains water. When a greater amount of ethanol is dissolved into water, ethanol molecules replaced water molecules between inter/intra-molecule of PEDOT:PSS with enhancement of the grain boundary of PEDOT:PSS [16,17]. When with the reason above, the viscosity of PEDOT:PSS would be increased relatively with an increase of EtOH, and concentration ratio of EtOH overcomes the threshold barrier of the covered-EtOH molecule at the surface of water, and thus the viscosity of the hetero-solution might be reduced. We also measured the viscosity and surface tension values of the hetero-solution. The viscosity of the PEDOT:PSS solution diluted with a 0% to 40% volume ratio of EtOH was shown to increase from about 10 cp to 25 cp. The viscosity of the PEDOT:PSS solution diluted with more than 40% was decreased from about 25 cp

to 2.5 cp. The surface tension value of PEDOT:PSS solution (CH8000) was about 70 mN/m before being diluted with EtOH and its surface tension value was decreased to about 22.5 mN/m. In the case of the PEDOT:PSS solution diluted with 90% EtOH, its viscosity value and surface tension value were measured to be about 1.3 cp and 22.8 mN/m, respectively. Therefore, we could predict and describe the behavior of pattern width variation would follow that seen in Figure 2a. Furthermore, we converted and calculated the relative film thickness without absolute scale of the PEDOT:PSS/EtOH pattern shape by using the grey scale method [14]. The grey scale method revealed that the coated pattern can absorb and reflect the light with various thicknesses of the layer, and therefore the pattern contrast of grey color intensity with pattern shape including width has a difference shadow. Thus, we could convert the thickness of the patterns, as shown in Figure 2b. The behavior of the pattern thickness by using the grey scale method was similar to the CCD camera images of the pattern shape. To fabricate a proper PEDOT:PSS line pattern, the remaining water concentration is also important. The evaporation rate change of the 90% EtOH volume concentration in PEDOT:PSS solution in a circular petri dish with an inner diameter of 35 mm and the evaporation rate was measured according to the EtOH volume concentration. The evaporation rate of 90% EtOH volume concentration in the PEDOT:PSS solution in the dish changed with time. Eventually, when the EtOH in the solution was completely dried, the evaporation rate was the same as the water evaporation rate. During the drying process, the measured evaporation rate could be used to calculate the volume concentration and EtOH in the remaining solution and the amount of remaining solution. As in the above result, we found that a uniform pattern, considering line width, depends on the remaining water concentration in the total solution.

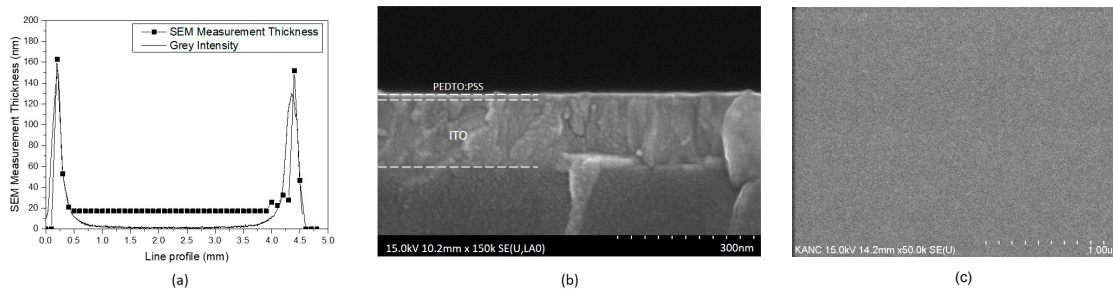


**Figure 2.** (a) Charge-coupled device (CCD) camera images of poly(3,4-ethylenedioxythiophene) polystyrene sulfonate (PEDOT:PSS) pattern with containing various rate of EtOH solvent. (b) Shape of PEDOT:PSS patterns with various conditions by Simulating of Grey scale method.

As a result, the optimized thickness uniformity was obtained at 90% EtOH volume concentration in PEDOT:PSS solution, and SEM was used to measure the actual thickness. To determine the



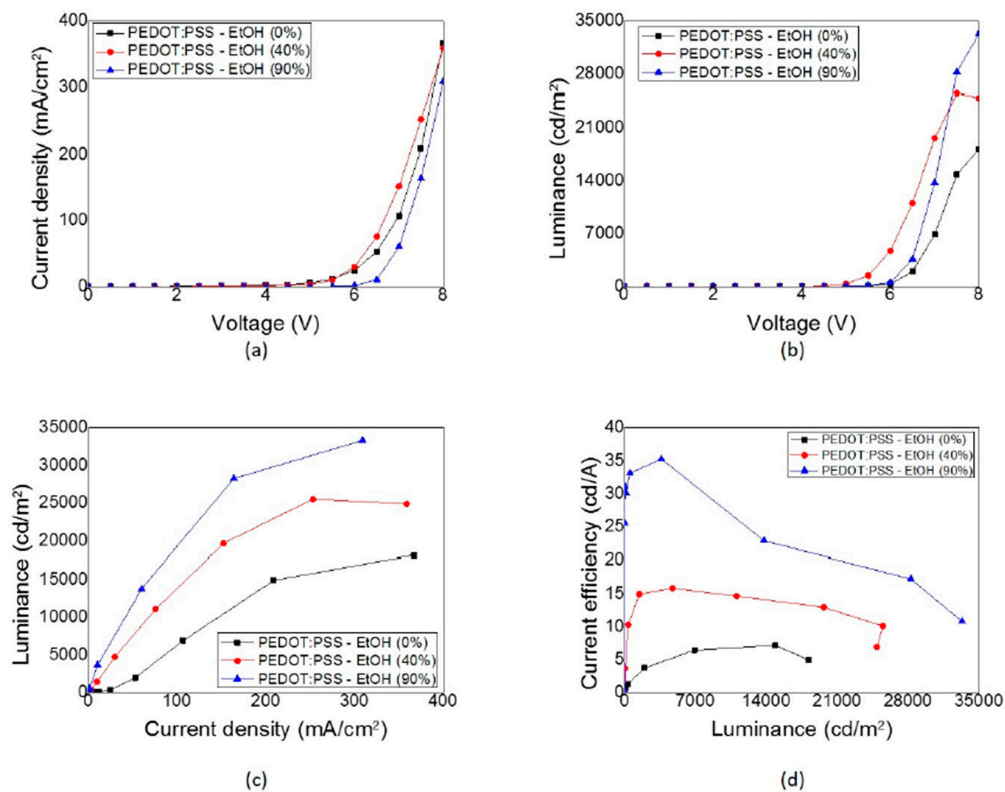
uniformity by grey intensity, the thickness profile of the width was measured at intervals of 100  $\mu\text{m}$  using SEM. Figure 3 shows the line width thickness profile measured by grey intensity and SEM. The thickness of both ends of the line was measured as about 160 nm due to the coffee ring effect and the thickness except for at both ends was measured to be about 18 nm. A thickness of about 18 nm was uniformly measured over about 3.5 mm scanning length. Also, we observed good film uniformity without any pinhole defects in Figure 3c.



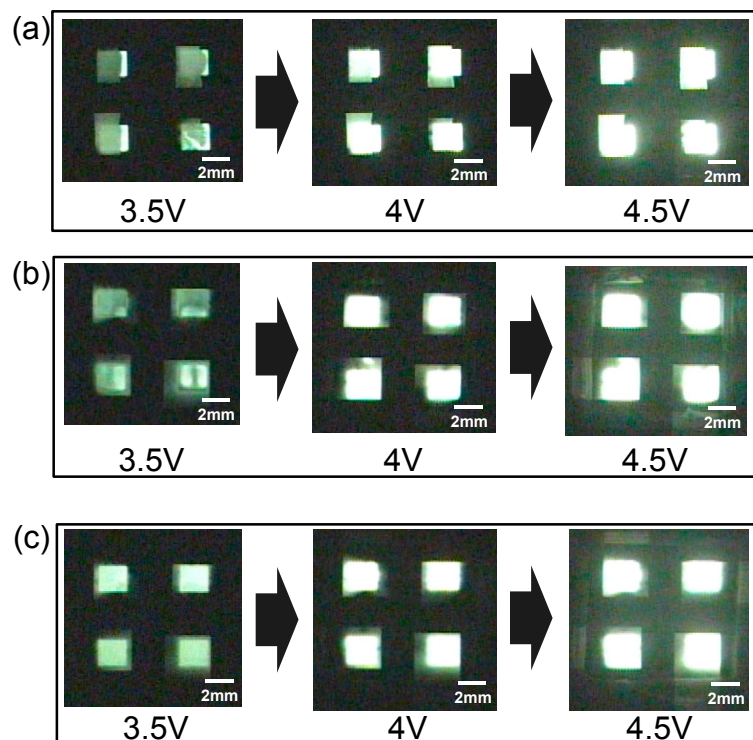
**Figure 3.** (a) Profile data of PEDOT:PSS patterns with including EtOH rate of 90% by measuring of SEM and scaling of Grey scale method. SEM cross-sectional (b) and top-view (c) images of PEDOT:PSS pattern.

Finally, as seen from the results of the above PEDOT:PSS pattern behavior according to the dilution rate of EtOH, we fabricated the OLED device structure as glass/ITO/printed-PEDOT:PSS ( $w/\text{EtOH}$  0%, 40%, 90%)/CBP:Ir(mppy)<sub>3</sub> (10% doped)/TPBi/LiF/Al. The whole group of OLED devices had a spectrum that arose in the electro luminance (EL), where a maximum peak of 515 nm was achieved. Additionally, EL spectrum yielded 1931 Commission Internationale de L'Éclairage (1931 CIE) chromaticity coordinates of (0.296, 0.623) approximately. According to the EL spectrum and CIE chromaticity coordinates, the nozzle-printed HIL based device emitted green light without any color shift. Figure 4 shows the I-V-L characteristics of the OLED devices. From the voltage-current density graph and voltage-luminance characteristics, device operated at almost 5 V (Figure 4a,b). Typically in the OLED device structure of glass/ITO/printed-PEDOT:PSS ( $w/\text{EtOH}$  0%, 40%, 90%)/CBP:Ir(mppy)<sub>3</sub> (10% doped)/TPBi/LiF/Al, the turn on voltage is approximately 4 V. However, as seen in Figure 4a,b, the device samples fabricated by the nozzle printing process shifted to relatively higher voltage because the nozzle printing process had poor overall film uniformity compared with the spin-coating process. Figure 4c shows the current density-luminance data. As seen in the graph, when the EtOH percent was increased, a high value of luminance was shown in the same current density scale. The OLED device based on PEDOT:PSS with EtOH of 90% had 35,000  $\text{cd}/\text{m}^2$  maximum luminance at 300  $\text{mA}/\text{cm}^2$ , and the OLED devices based on PEDOT:PSS with EtOH of 0% and 40% recorded values of about 17,000  $\text{cd}/\text{m}^2$  and 25,000  $\text{cd}/\text{m}^2$  at 300  $\text{mA}/\text{cm}^2$ , respectively. Lastly, in Figure 4d, maximum current efficiency of 35  $\text{cd}/\text{A}$  was recorded in the device sample of PEDOT:PSS-EtOH (90%) while PEDOT:PSS-EtOH (0%, 40%) showed values of 5  $\text{cd}/\text{A}$  and 15  $\text{cd}/\text{A}$ . This difference was caused by the film uniformity of the PEDOT:PSS layer with variation of the EtOH concentration. When a greater amount of EtOH (about 90%) was added to the PEDOT:PSS solution, spreading, shrinking, and drying behaviors were optimized for the formation of a uniform film layer.

Figure 5 presents electro-luminescence (EL) camera images of individual device samples with voltage variation. The OLED device in Figure 5a was fabricated with pristine PEDOT:PSS and those in Figure 5b,c were made with PEDOT:PSS with 40% and 90% EtOH solvent. With bias voltage of 3.5, EtOH of 90% based pixel in OLED showed a clear green light image whereas non-uniform light with a defect pattern was observed for both pristine PEDOT:PSS and EtOH of 40% including in PEDOT:PSS based pixel.



**Figure 4.** Organic light emitting diode (OLED) device performance graph of (a) Current Density-Voltage; (b) Luminance-Voltage; (c) Luminance-Current density; and (d) Current Efficiency-Luminance.



**Figure 5.** Electro-luminescence (EL) images of OLED with various conditions of solvent ratio and voltage variations: (a) pristine PEDOT:PSS based EL image; (b) EtOH of 40% containing ratio in PEDOT:PSS based EL image; and (c) EtOH of 90% containing ratio in PEDOT:PSS based EL image.

#### 4. Conclusions

In this study, we determined the optimal dilution rate of EtOH in PEDOT:PSS solution as about 90 volume percent, and also obtained a uniform PEDOT:PSS pattern with width of almost 4 mm to cover pixels in an OLED by using 0.1 mm diameter nozzle printing technology. We observed the PEDOT:PSS pattern behaviors with various concentrations of EtOH in the total solution. In terms of achieving a proper uniform layer, the EtOH (90% ratio) based experimental group showed the best uniformity and suitable coating thickness of about 20 nm. Finally, we fabricated an OLED device with nozzle printed PEDOT:PSS as the HIL. We obtained a current efficiency of 27 cd/A with uniform quality of luminance in the case of the device containing 90% EtOH. Further study to determine the detailed mechanism for the branched shape at the line edge and the correlation with film uniformity will be performed in the near future.

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**Author Contributions:** K.-T. Kang, M. Kang and D. G. Yoon conceived and design the experiments; D. G. Yoon, M. Kang, J. B. Kim performed the experiments; D. G. Yoon and M. Kang analyzed the data; M. Kang contributed grey scale method and SEM analysis, D. G. Yoon contributed the SEM, AFM and IVL data analysis; D. G. Yoon wrote the paper.

**Conflicts of Interest:** The authors declare no conflict of interest.

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