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# Solution-processed sodium hydroxide as electron injection layer in inverted bottom-emitting organic light-emitting diodes

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**ABSTRACT:** We present inverted bottom-emitting organic light-emitting diodes (IBOLEDs), consisting of tris-(8-hydroxyquinoline) aluminum ( $\text{Alq}_3$ ) as the emissive layer and an ultrathin layer of sodium hydroxide (NaOH) on top of indium tin oxide (ITO) as the electron injection layer. The devices with NaOH treated by water vapor and  $\text{CO}_2$  and annealing show higher current efficiency than those with NaOH untreated. The current efficiency (6 cd/A) of the optimal devices with treated NaOH layer is improved. The enhancement is attributed to the reduction in barrier height for electron injection due to the dipole formation caused by the conversion of NaOH to sodium carbonate.

**KEYWORDS:** organic light-emitting devices, interface dipole, inverted bottom-emitting, sodium hydroxide

## 1. Introduction

Organic light-emitting devices (OLEDs) have been attractive in the past decades due to the potential applications in large-area flat panel displays and lighting. For large-area displays, it is necessary to develop the active matrix driving method. In order to take advantages of the existing mature technology of n-type amorphous silicon thin film transistor (a-SiTFT) or organic TFTs compatible with active matrix displays,<sup>1-4</sup> inverted OLEDs (IOLEDs) with bottom cathode are highly desirable. The bottom cathode of IOLEDs can be transparent or reflective, which is determined by the direction of the emitting light. On one hand, the top-emitting IOLEDs with light emitted from top electrode have a transparent indium tin oxide (ITO) as anode, most of which has to be sputtered on top of organic layers leading to the decrease of device performance and stability due to the damage caused by high power sputtering.<sup>5-7</sup> On the other hand, the device with light emitted from the bottom substrate is defined as inverted bottom-emitting OLED (IBOLED).<sup>8</sup>

The challenge of building IBOLEDs is to explore an effective approach to make the bottom electrode (ITO) easy for electron injection, i.e. reduce the electron injection barrier (EIB) from ITO (4.7 eV) to the lowest unoccupied molecular orbital (LUMO) level of organic electron transport layer (ETL).<sup>9-10</sup> Modifying the organic/metal interface is an efficient way to reduce the EIB between organic active layer and metal cathode. Various modifications at the organic/metal interface have been reported, including the deposition of an ultrathin layer of alkali metals, alkaline-earth metals, metals and metallic compounds with low work function,<sup>4, 8, 11-16</sup> organic self-assembled monolayer,<sup>12, 17</sup> polyelectrolytes,<sup>18</sup> as well as n-type metal oxides.<sup>19-20</sup>

The mechanism of lowering the work function of ITO by the interfacial modification is generally attributed to the formation of dipoles on ITO surface. Nüesch et al. reported that the work function of ITO is sensitive to acid and base,<sup>21</sup> showing that a base can decrease its work function. Although ITO treated with base has been exploited as anode in OLEDs,<sup>3, 22</sup> there has been no report on IBOLEDs with sodium based base-treated ITO as cathode.

In this report, we demonstrate IBOLEDs with solution-processed sodium hydroxide (NaOH) on ITO as electron injection layer, which is treated under different conditions. The results show that the devices with NaOH treated by a combination of water vapor and carbon dioxide (CO<sub>2</sub>) obtain a higher current efficiency of 6.0 cd/A than the device with NaOH untreated (4.5 cd/A). The significant enhancement in the device performance is attributed to the reduction in electron injection barrier due to the dipole formation on ITO.

## 2. Experimental section

*Device fabrication:* the commercial ITO-coated glass substrates have a sheet resistance of 10  $\Omega$ /sq. The ITO substrates were rinsed with detergent, and then sequentially cleaned by Decon 90 detergent, deionized (DI) water, acetone, and ethanol in ultrasonic bath (Shu mei KQ-300DE) for 15 min. They were dried by a nitrogen flow before usage. ITO substrates were directly heated at 120°C for 10 min or treated by UV-ozone (UVO cleaner 42-220, Jelight Company Inc.) for 15 min. Then the NaOH or sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>) was spin coated on top of ITO at 3000 rpm for 1 min, followed by different annealing treatments. Finally, all of the devices were finished by thermally evaporating tris(8-hydroxyquinolino)aluminum (Alq<sub>3</sub>, 60 nm), N,N'-Di(1-naphthyl)-N,N'-diphenyl-(1,1'-biphenyl)-4,4'-diamine (NPB, 60 nm), MoO<sub>3</sub> (8 nm), and aluminum (Al) (150 nm) in a high-vacuum system ( $<5 \times 10^{-4}$  Pa) in the same running. The active area of the

devices is  $16 \text{ mm}^2$ , defined by the overlap between ITO and Al electrode. The IBOLEDs have a structure of ITO/NaOH (x% vol)/Alq<sub>3</sub>/NPB/MoO<sub>3</sub>/Al as shown in **Figure 1(a)**. Specifically, For Group A, the devices have the NaOH layer with varying concentration of solution and annealed at 120°C for 10 min in air. For comparison, the control device was made without NaOH or Na<sub>2</sub>CO<sub>3</sub>. For Group B devices, Device B has 0.4wt% NaOH on ITO. A Petri dish of water was heated at 120 °C to produce water vapor, and then the NaOH-coated ITO was dispersed into it, followed by blowing of CO<sub>2</sub> from a needle of the syringe for 1 min. CO<sub>2</sub> was produced by the reaction of Na<sub>2</sub>CO<sub>3</sub> and hydrochloric acid (HCl). Finally, the treated layer was heated at 120°C for 10 min in air. Device A, C, and D were fabricated by spin-coating NaOH solution on ITO in air, Na<sub>2</sub>CO<sub>3</sub> solution in air, and NaOH solution in the glove box, respectively, all of which were followed by annealing at 120°C for 10 min in their corresponding environment. Two groups of devices are summarized as below:

Group A:

ITO/NaOH(0.3%wt,0.4%wt,0.5%wt,0.6%wt)/Alq<sub>3</sub>/NPB/MoO<sub>3</sub>/Al

Device E: ITO/Alq<sub>3</sub>/NPB/MoO<sub>3</sub>/Al

Group B:

Device A: ITO/NaOH(0.4wt%)/Alq<sub>3</sub>/NPB/MoO<sub>3</sub>/Al (NaOH annealed without blowing CO<sub>2</sub> in air )

Device B: ITO/NaOH(0.4wt%)/Alq<sub>3</sub>/NPB/MoO<sub>3</sub>/Al (NaOH treated with blowing CO<sub>2</sub> and water vapor)

Device C: ITO/Na<sub>2</sub>CO<sub>3</sub>(0.2wt%)/Alq<sub>3</sub>/NPB/MoO<sub>3</sub>/Al (Na<sub>2</sub>CO<sub>3</sub> made in air)

Device D: ITO/NaOH(0.4wt%)/Alq<sub>3</sub>/NPB/MoO<sub>3</sub>/Al (NaOH made in glove box)

*Device characterization:* The layer thickness was controlled by the frequency counter, and calibrated by Dektak 6M Profiler (Veeco). The current density-voltage-luminance (*J-V-L*) characteristics were measured by using a Keithley source measurement unit (Keithley 2400 and Keithley 2000) with a calibrated silicon photodiode. All devices were measured at room temperature without encapsulation.

### 3. Results and discussion

**Figure 1(a)** illustrates the device architecture of IBOLEDs with NaOH-modified ITO used for the electron injection and MoO<sub>3</sub> used for the hole injection. The energy level diagram of the IBOLED is shown in **Figure 1(b)**. In order to form a uniform layer of NaOH on ITO, the methanol was employed as the solvent due to its low boiling point and the solution was coated at a high spinning speed.

In order to investigate the effect of NaOH layer thickness on the device performance, the devices with varying concentration (0.3wt%, 0.4wt%, 0.5wt%, 0.6wt%) of NaOH solutions (Group A) were compared. **Figure 2(a)** and (b) show their current density-luminance-voltage (*J-L-V*) and current efficiency-luminance (*CE-L*) characteristics for the devices with varying concentration of NaOH solutions, respectively. For comparison, the *J-L* characteristic of Device E is also shown in **Figure 2(a)**. All the devices with NaOH as the electron injection layer exhibit an increase in current density and luminance in forward bias compared to the device without NaOH. It can be seen that Device E hardly emits light, which is attributed to the high electron injection barrier between the work function of ITO (4.7 eV)<sup>23</sup> and the LUMO (~3.3 eV) of Alq<sub>3</sub>.<sup>24</sup> However, the insertion of NaOH layer effectively reduces the work function of ITO from

4.7 eV to 3.71 eV,<sup>25</sup> lowering down the injection barrier to facilitate the electron injection, which improves the charge carrier balance and therefore leads to a superior performance. Obviously, the optimal concentration of NaOH solution is about 0.4 wt% (see **Figure 2**). The optimal device produces a luminance of  $>10,000$  cd/m<sup>2</sup> and a maximum current efficiency of 4.5 cd/A. The results are similar to other reports on insulating layer thickness; either too thin or too thick layer is not good for the tunneling injection mode.<sup>26</sup> We hypothesize that the impact of NaOH film on the electron injection is due to the partial conversion of NaOH to Na<sub>2</sub>CO<sub>3</sub>.

To investigate the effect of NaOH layer with different treatments, the devices of Group B were fabricated, including a control device without annealing treatment on NaOH for comparison. In order to evaluate the electron injection capability of NaOH layer with different treatments, the *J-V* characteristics of a series of electron-only devices of ITO/NaOH(without treatment in air)/Alq<sub>3</sub>(60 nm)/Al (EOD-A), ITO/NaOH(with annealing treatment in the glove box)/Alq<sub>3</sub>(60 nm)/Al (EOD-B), ITO/NaOH(with annealing treatment in air)/Alq<sub>3</sub>(60 nm)/Al (EOD-C), ITO/NaOH(blowing CO<sub>2</sub> and H<sub>2</sub>O)/Alq<sub>3</sub>(60 nm)/Al (EOD-D), and ITO/Na<sub>2</sub>CO<sub>3</sub> (with annealing treatment in air)/Alq<sub>3</sub>(60 nm)/Al (EOD-E) are shown in **Figure 3**, where treated ITO is used as the cathode. EOD-A and EOD-B show much lower electron current. However, the electron current is greatly enhanced as the NaOH layer is annealed. Furthermore, it is clearly seen that the device with NaOH layer being blown by CO<sub>2</sub> and H<sub>2</sub>O vapor and then annealed for 10 min shows higher electron current than that with NaOH treated only by annealing. This indicates that the NaOH layer treated by CO<sub>2</sub> and H<sub>2</sub>O vapor works as a good interfacial layer for better electron injection in IBOLEDs.

The *J-L-V*, *CE-L*, and *power efficiency-L* characteristics of the IBOLEDs in Group B are shown in **Figure 4**(a), (b) and (c), respectively. Device D with NaOH processed and treated in

the glove box exhibits poor device performance with lower current density and efficiency. It can be explained that the nitrogen-filled glove box where NaOH was coated and annealed prevents the conversion of NaOH to Na<sub>2</sub>CO<sub>3</sub> due to lack of sufficient CO<sub>2</sub>. In other words, Device D can be considered as the device with pure NaOH-modified ITO as cathode, leading to lower brightness and efficiency, as well as bigger roll-off. This implies that pure NaOH layer is likely to induce the interaction between alkali and ITO, resulting in a great reduction of device performance.

Device A with NaOH only annealed in air obtains great improvement in device performance. Such annealing may affect the interaction between NaOH and ITO, and NaOH may be partially converted to Na<sub>2</sub>CO<sub>3</sub> during annealing in air.<sup>25, 27</sup> In order to confirm the conversion of NaOH to Na<sub>2</sub>CO<sub>3</sub>, we blowed NaOH layer with CO<sub>2</sub> on purpose. The sample was rinsed in a petri dish with 100 °C water vapor for 1 min, and then heated at 120 °C in air. As seen in **Figure 4**, Device B with NaOH layer treated by CO<sub>2</sub> and water vapor has significant improvement in the luminance and efficiency, achieving a higher current efficiency of 5.9 cd/A and a higher power efficiency of 1.75 lm/W than other devices. Therefore, it is reasonably proposed that NaOH was converted to Na<sub>2</sub>CO<sub>3</sub> upon the treatments of CO<sub>2</sub> and H<sub>2</sub>O vapor. The formation of Na<sub>2</sub>CO<sub>3</sub> via annealing of NaOH in air can facilitate the electron injection, which might be related to the better contact at the ITO/emitting layer interface. Na<sub>2</sub>CO<sub>3</sub> layer has been reported as a better electron injection layer.<sup>13</sup> Thin Na<sub>2</sub>CO<sub>3</sub> layer has the capability of injecting the electrons and blocking the holes, similar to Cs<sub>2</sub>CO<sub>3</sub> used at the cathode/ETL interface.<sup>28</sup>

Furthermore, the conversion of NaOH to Na<sub>2</sub>CO<sub>3</sub> by different treatments can be confirmed by the AFM images in **Figure 5** and X-ray photoelectron spectra (XPS) in **Figure 6**. As shown in **Figure 5**, after NaOH layer is blown by CO<sub>2</sub> and water vapor and then annealed, ITO surface

has more particles than those with NaOH only annealed, and without any treatment, and bare ITO surface, implying the formation of  $\text{Na}_2\text{CO}_3$  by annealing NaOH layer in air. **Figure 6** presents the XPS spectra of a blank ITO, non-treated NaOH/ITO, and  $\text{CO}_2$ -treated NaOH/ITO. The Na bands appear at 1071.9 eV for NaOH, which shift to 1070.9 eV for NaOH/ITO and 1070.18 eV for  $\text{CO}_2$ -treated NaOH/ITO. The binding energies decrease upon the treatment of annealing and  $\text{CO}_2$  blowing, exhibiting lower value for  $\text{CO}_2$  treatment. On the other hand, the C bands of ITO have a shift after the deposition of NaOH. The C1s bands appear at 285 eV for the blank ITO. They shift to 284.5 eV for NaOH/ITO, while they shift to 287.6 eV and 288.9 eV for  $\text{CO}_2$ -treated NaOH/ITO, where 289 eV corresponds to  $\text{CO}_3$ . Therefore, this result further verifies the formation of  $\text{Na}_2\text{CO}_3$ .

In order to assess the effect of treated-NaOH and  $\text{Na}_2\text{CO}_3$  on the device performance, Device C with pure  $\text{Na}_2\text{CO}_3$  layer as electron injection layer was fabricated. 0.2 wt%  $\text{Na}_2\text{CO}_3$  in methanol is spin-coated on ITO in air, which is optimal to modify the ITO. The luminance and efficiency of Device C are higher than those of Device A with NaOH annealed in air, but lower than those of Device B treated with  $\text{CO}_2$  and water vapor. However, in **Figure 4(a)**, the current density of Device C is higher than that of Device B with being blown by  $\text{CO}_2$  and water vapor. It is possible that the device with direct  $\text{Na}_2\text{CO}_3$  treatment has a larger leakage current as a result of non-uniform and rougher  $\text{Na}_2\text{CO}_3$  film as shown in **Figure 5(d)**. This is a general issue that  $\text{Na}_2\text{CO}_3$  is not easy to be dissolved in the volatile organic solvent. However, only by annealing, NaOH layer cannot be completely converted to  $\text{Na}_2\text{CO}_3$ , as seen from **Figure 5(a)**.

It has been proved that introducing NaOH and  $\text{Na}_2\text{CO}_3$  can greatly reduce the work function of ITO by ultraviolet photoelectron spectra (UPS) and the mechanism is demonstrated by XPS.<sup>25</sup> UPS results show that forming NaOH layer in a glove box can reduce the work function more

significantly than that in air. However, the work function of  $\text{Na}_2\text{CO}_3$ -treated ITO is 3.80 eV, even slightly lower than that of NaOH-treated ITO. Un-treated NaOH layer results in lower work function of ITO; however it does not exhibit higher performance. This also confirms our above assumptions that alkali NaOH is more likely to interact with ITO, similar to the report.<sup>21</sup> The reason that alkaline can reduce the work function of ITO is because  $\text{OH}^-$  can be associated with the metal ions in ITO,<sup>21</sup> indicating that the conversion of NaOH to  $\text{Na}_2\text{CO}_3$  is properly effective to reduce the work function of ITO.

#### 4. Conclusion

We have demonstrated a simple method to fabricate efficient IBOLEDs. A thin NaOH interlayer has been introduced between ITO and the electron transport layer by spin-coating, followed by exposure to  $\text{CO}_2$  and  $\text{H}_2\text{O}$  vapor and annealing in air. This process can significantly reduce the work function of ITO, leading to the decrease of the energy barrier between ITO and  $\text{Alq}_3$ . The conversion of NaOH to  $\text{Na}_2\text{CO}_3$  by our method efficiently inhibits the reaction of NaOH and reduces the leakage current caused by the non-uniform film of  $\text{Na}_2\text{CO}_3$ . The formation of  $\text{Na}_2\text{CO}_3$  layer enhances electron injection and blocks holes, resulting in improved balance of electrons and holes in the emitting layer. Finally, high-efficiency IBOLED reaches a maximum current efficiency of 5.95 cd/A. Our results indicate that forming uniform  $\text{Na}_2\text{CO}_3$  film by converting solution-processed NaOH provides an effective approach to obtain efficient electron injection layer for high-performance IBOLEDs.

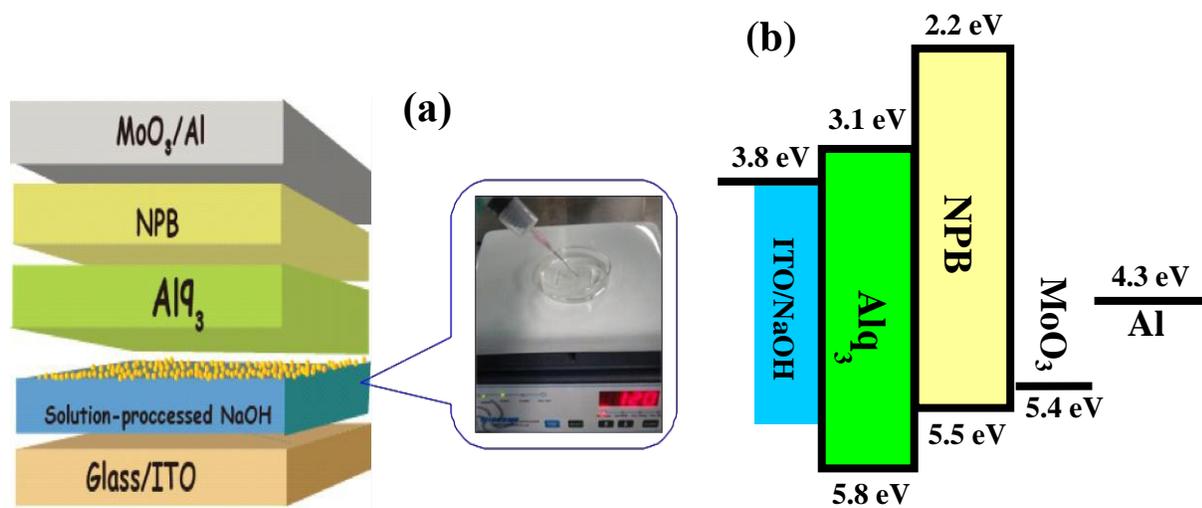


Figure 1 (a) Device architecture of IBOLEDs with NaOH-treated ITO as cathode, (b) Schematic energy diagram of the device. Treatment process of NaOH is also shown in the inset of Figure 1(a).

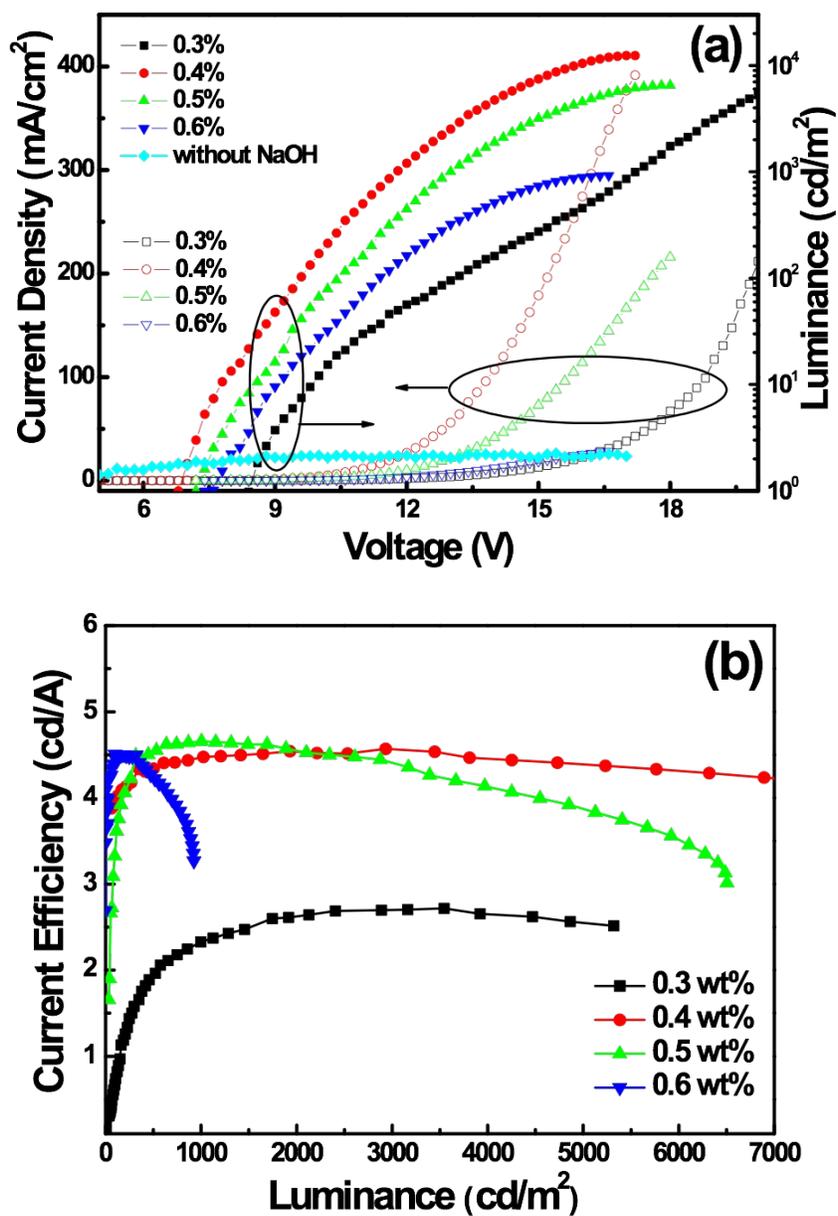


Figure 2. (a)  $J-L-V$  characteristics, (b) Current efficiency-Luminance characteristics of the devices with varying concentration of NaOH solution (Group A).

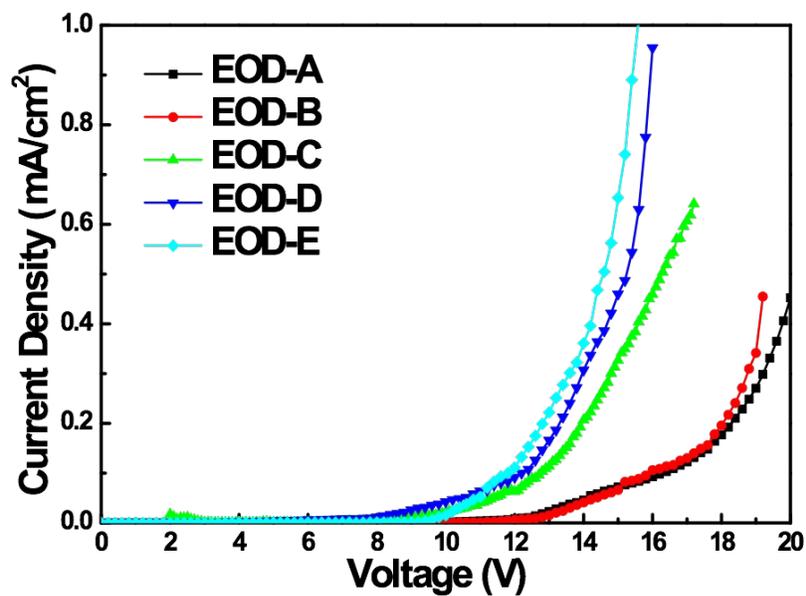
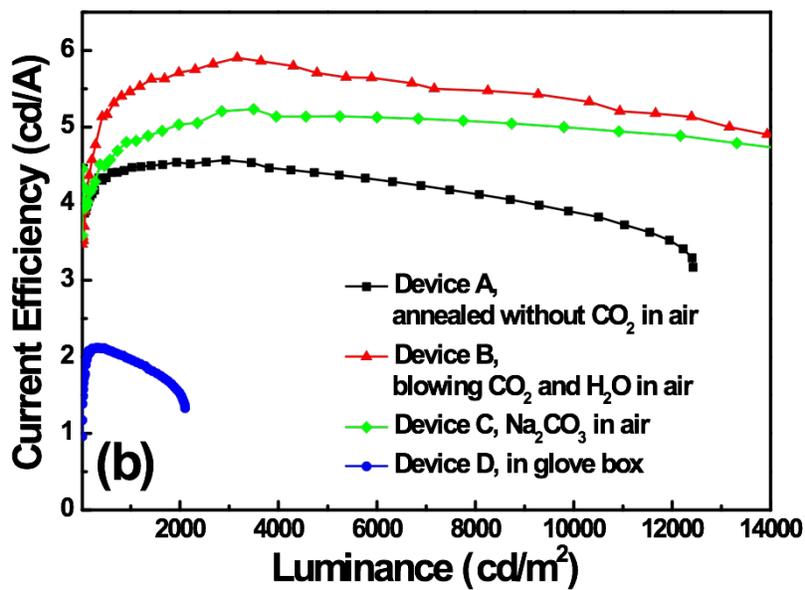
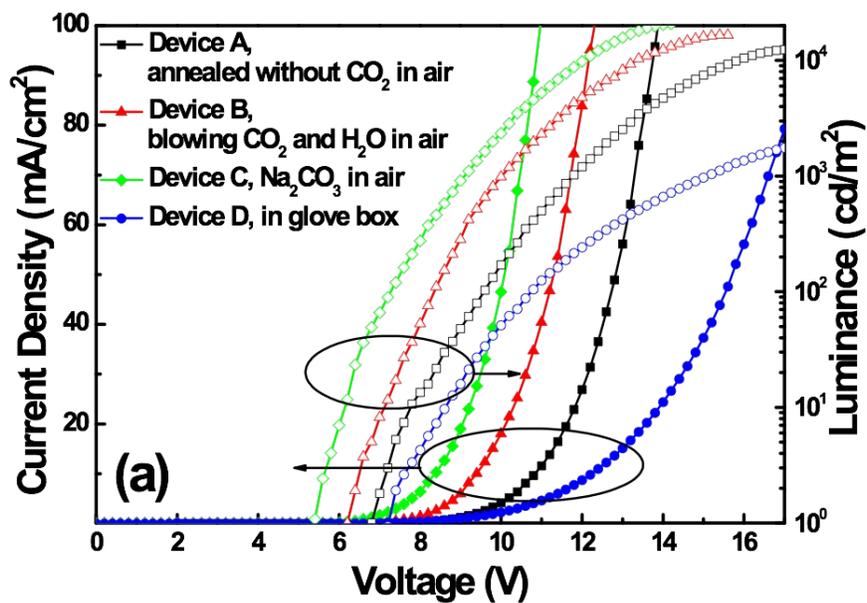


Figure 3.  $J$ - $V$  characteristics of electron-only devices: EOD-A, EOD-B, EOD-C, EOD-D, and EOD-E.



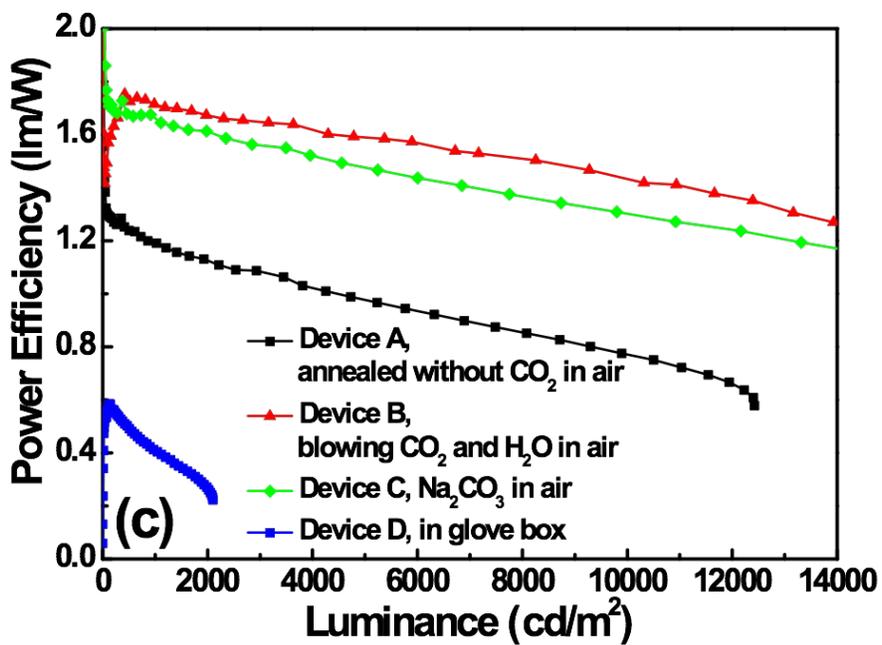
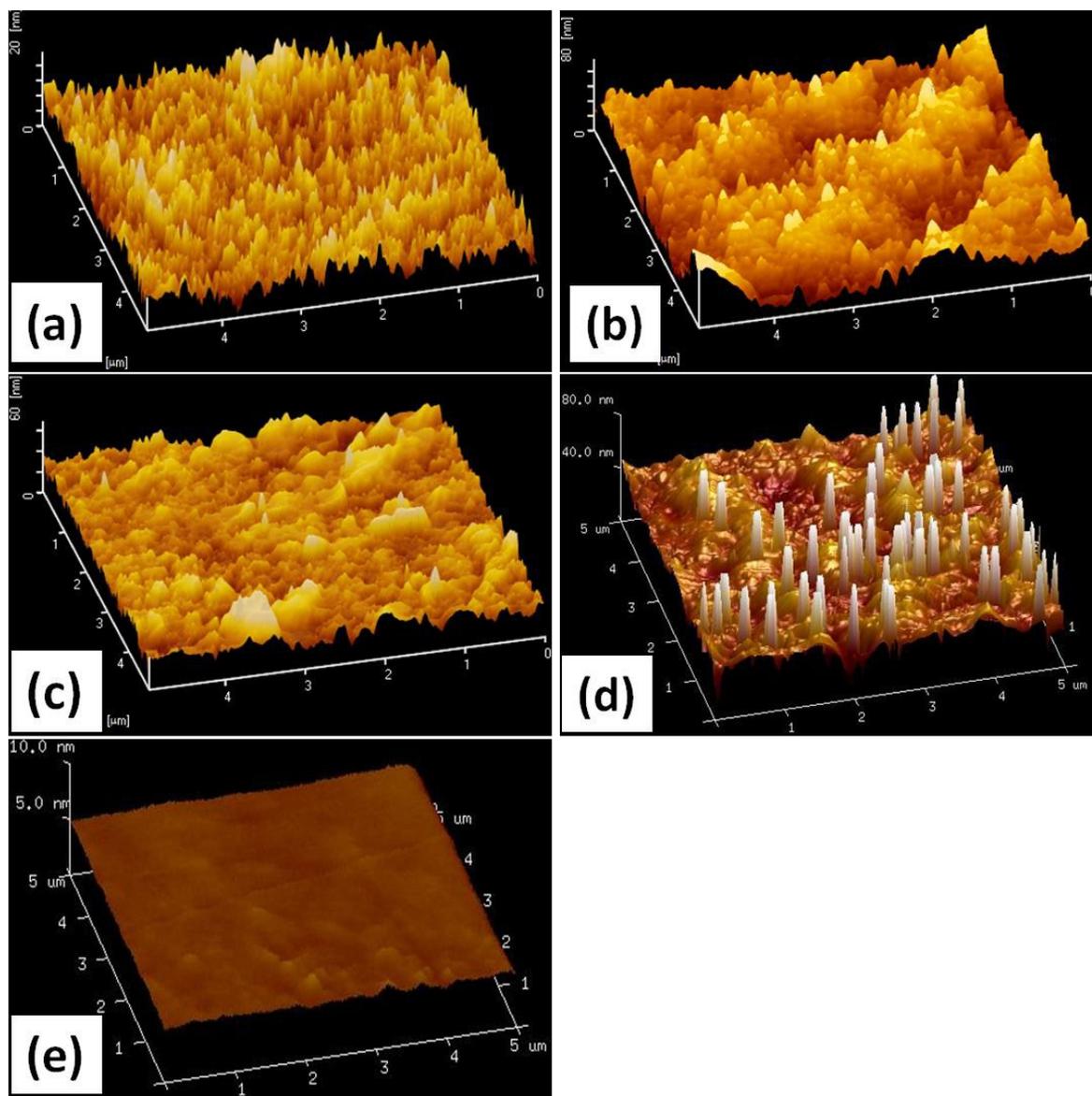


Figure 4. (a)  $J$ - $L$ - $V$  characteristics, (b) Current efficiency-Luminance characteristics, (c) Power efficiency-Luminance characteristics of the devices in Group B.



**Figure 5.** Tapping-mode AFM images of NaOH/ITO or Na<sub>2</sub>CO<sub>3</sub>/ITO. (a) annealed at 120 °C for 10 min in air, (b) blow CO<sub>2</sub> and H<sub>2</sub>O vapor for 1 min and then annealed at 120 °C for 10 min in air, (c) 0.4 wt% NaOH without any treatment, (d) Na<sub>2</sub>CO<sub>3</sub>, and (e) bare ITO. The size of the images is 5×5 μm<sup>2</sup>.

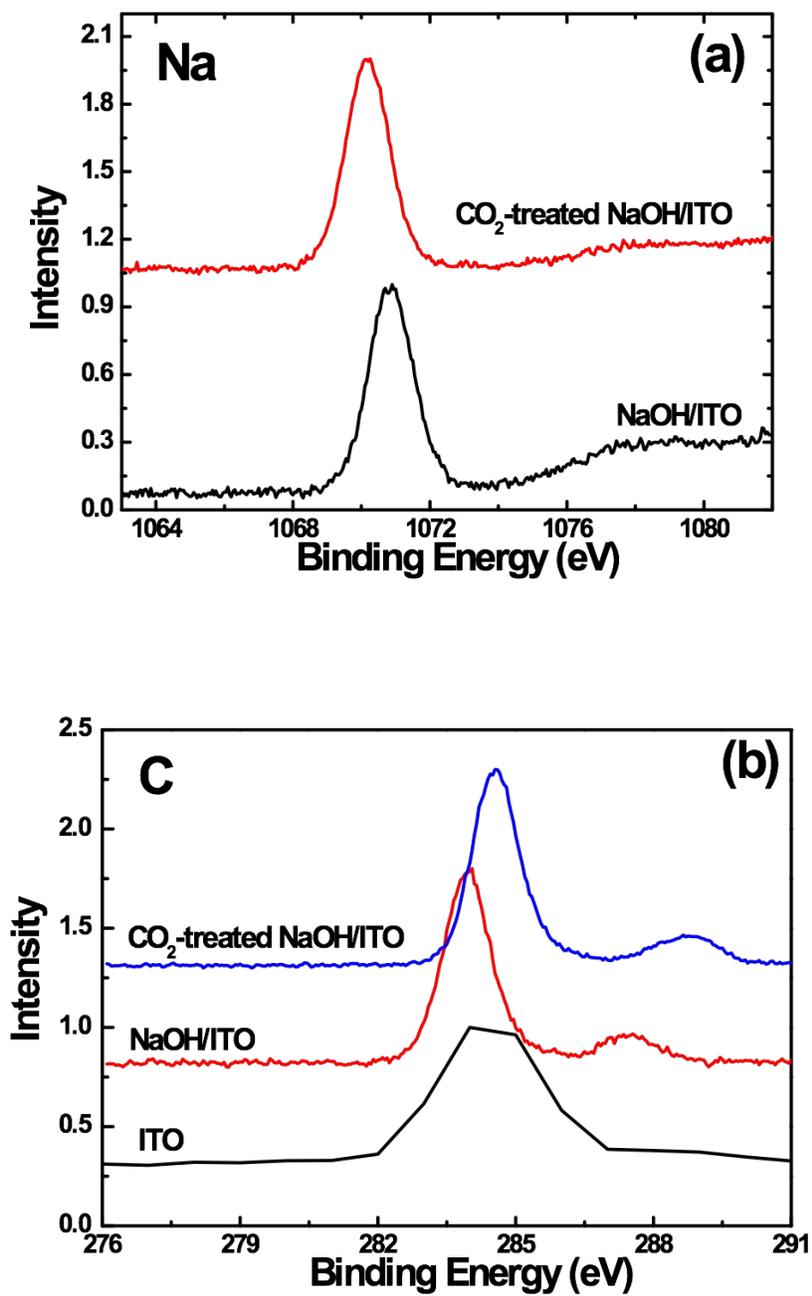


Figure 6. XPS spectra for (a) Na and (b) C of  $\text{CO}_2$ -treated NaOH/ITO, NaOH/ITO, and ITO.

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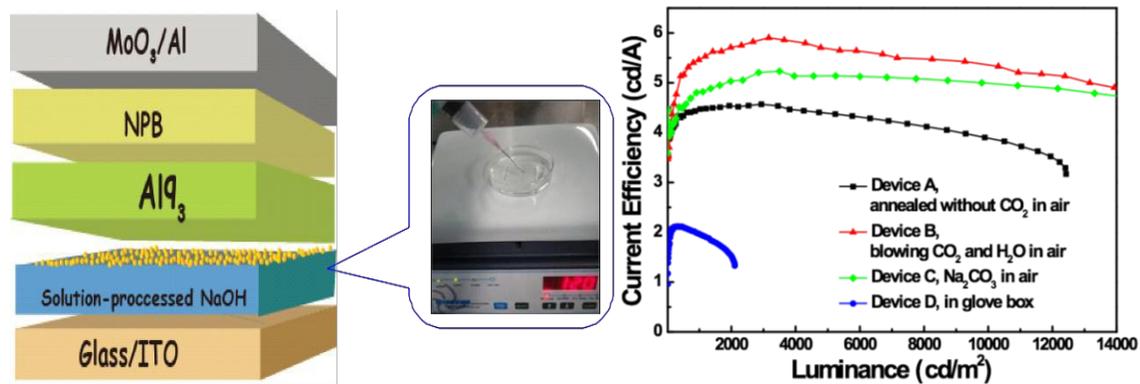
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Thin sodium hydroxide treated by blowing CO<sub>2</sub> and water vapor in inverted organic light-emitting diodes exhibits improved efficiency.