Solution processed naphthalene diimide derivative as an effective electron transport material for enhanced brightness and efficiency in polymer light emitting diodes

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Abstract

Increasing the efficiency and lifetime of polymer light emitting diodes (PLEDs) requires a balanced injection and flow of charges through the device, driving demand for cheap and effective electron transport/ hole blocking layers. Some materials, such as conjugated polyelectrolytes, have been identified as potential candidates but the production of these materials requires complex, and hence costly, synthesis routes. We have utilized a soluble small molecule naphthalene diimide derivative (DC18) as a novel electron transport/hole blocking layer in common PLED architectures, and compared its electronic properties to those of the electron transport/hole blocking small molecule bathocuproine (BCP). PLEDs incorporating DC18 as the electron transport layer reduce turn on voltage by 25%; increase brightness over three and a half times; and provide a full five-fold enhancement in efficiencies compared to reference devices. While DC18 has similar properties to the effective conjugated polyelectrolytes used as electron transport layers, it is simpler to synthesise, reducing cost while retaining favourable electron transport properties, and improves upon their degree for efficiency enhancement. The impact on device lifetime is hypothesized to be significant as well, due to the air-stability seen in many naphthalene diimide derivatives.

Introduction

Polymer light emitting diodes (PLEDs) offer a cheap, flexible alternative to existing lighting display technologies, as they can be fabricated using solution processing techniques for large area devices. [1] While advances in PLED technology have been numerous, they remain hindered by low efficiencies and limited lifetime stability. Since PLEDs require injection of both electrons and holes into the active material, balancing charge flow is essential to increase device efficiency. [2] The majority of electroluminescent polymers used as active materials have higher hole mobilities than electron mobilities, in addition to an incompatibility between their low LUMO levels and the high work function metallic cathodes used. [3, 4] Thus significant interest towards increasing charge flow balance has focused on improving electron injection at the cathode/polymer interface. Early modifications involved inserting a thin layer of low work function reactive metals such as calcium [5, 6] beneath the metal electrode, requiring encapsulation to prevent degradation. However, these metals require high vacuum and temperatures for evaporation, hindering the translation of PLEDs to complete solution fabrication.

Recently, focus has shifted towards the use of solution processed electron injection layers made of self-assembled monolayers (SAMs), [7] conjugated polyelectrolytes (CPEs) or conjugated surfactants. [8-10] These novel organics have excellent solubility in alcohol-based solvents, allowing them to be applied to the active polymer layer for good electrical contact without interfacial mixing. The charged CPE electron transport layers (ETLs) are believed to lower the charge injection barrier between the high work function electrode and the active layer by creating a permanent dipole between the metal and the CPE, in addition to a migration of charged ions into the film. [11-14] Although enhanced efficiency in PLEDs has been demonstrated using CPEs, [8, 15] these polymers require complicated synthesis routes. The most effective polymers have used a hole-transporting polyfluorene backbone, which could hinder their hole-blocking properties. [8, 10, 16-18] An inexpensive alternative to these CPEs would be to replace them with soluble small molecules displaying similar electron-transporting capabilities. Unlike polymers, small molecules are not limited by low product yields or adversely affected by an extensive molecular weight range. Furthermore, delayed response times have been seen

in devices incorporating CPEs, [17] a problem not seen in devices containing their short chain oligomer counterparts. [19]

Naphthalene diimides (NDIs) are a class of compounds with high electron mobilities, and have primarily been used to produce air-stable n-type organic field effect transistors. [20-22] In addition, polymers developed from NDIs have shown promise as novel acceptors in organic solar cells, providing in excess of 1.5% device efficiencies, one of the highest for non-fullerene cells, with potential for extending device lifetimes. [23-25] The high electron mobilities of naphthalene diimides, coupled with their excellent air stability, give them considerable potential as air-stable electron injection layers. It is facile to chemically tune the electrical characteristics of naphthalene diimides using simple organic chemistry to modify either the conjugated backbone or the nitrogen atoms to introduce solubilizing side chains for easy solution processing. [26] For this work we have utilized a non-core modified NDI derivative, N,N'-bis(ethyl-N'',N'',N''-dimethyloctadecyl ammonium)-1,4,5,8-naphthalene diimide (DC18, Figure 1A), as a novel small molecule electron transport material. To our knowledge, this is the first time a NDI derivative has been utilized as such, and illustrates the ease with which n-type active layer materials can be employed as interfacial layers to further enhance organic electronics. We have reported on the synthesis and electroactivity of the DC18 molecule previously. [27] DC18 is expected to be an excellent electron injection layer due to its inherent properties, being a charged cationic species with the high electron transport known to NDIs. From cyclic voltammetry measurements (Supplementary Info), the LUMO value for DC18 was calculated [28] to be -4.2eV, with the HOMO calculated using the optical bandgap to be -7.5eV. These values are in good agreement with similarly structured NDI materials. [21, 29] The deep HOMO value for DC18 ensures it will be an excellent hole-blocking material for many PLED polymers while facilitating electron injection.

Experimental

Patterned ITO substrates ($15 \Omega \text{ sq}^{-1}$) were purchased from Luminescence Technology Corporation, and cleaned using a series of acetone and isopropanol sonications, followed by an oxygen plasma treatment (5 min, 100 W, 20 mbar O_2 , Emitect K1050X plasma cleaner). Poly(3,4-ethylenedioxythiophene) poly(styrenesulfonate) (PEDOT-PSS, Heraeus Clevio P VP AI 4083) was filtered ($45 \mu\text{m}$), mixed in a 25:5 ratio with ethanol, and the 45 nm film was annealed for 10 minutes at 160°C . 100 nm thick active layers were of either poly[2-methoxy-5-(2-ethylhexyloxy)-1,4-phenylene vinylene] (MEH PPV, ADS100RE) in chlorobenzene, or Poly[(9,9-di-n-octylfluorenyl-2,7-diyl)-alt-(benzo[2,1,3]thiadiazol-4,8-diyl)] (F8BT, ADS133YE) in chloroform, from American Dye Source Inc. Both solutions were filtered ($0.2 \mu\text{m}$) prior to use. 2,9-dimethyl-4,7-diphenyl-1,10-phenanthroline (BCP) from Luminescence Technology Corporation was evaporated to form a 5 nm thick layer. $\text{N,N}'$ -bis(ethyl- N'' , N'' , N'' -dimethyloctadecyl ammonium)-1,4,5,8-naphthalene diimide (DC18) was synthesised according to a previously described method, [27] dissolved in ethanol at a 0.5 mg mL^{-1} concentration, and filtered ($0.2 \mu\text{m}$) prior to spincoating to form a 20 nm thick layer. All devices had 80 nm of aluminum evaporated through a shadow mask for pixel areas of 8 mm^2 . All device fabrication was performed inside a glovebox, with the exception of the HTL and DC18. PLEDs were encapsulated prior to removal from the glovebox for testing in air. Current-voltage measurements were taken using a Keithley 2400 source meter, with simultaneous luminescence data collected using a calibrated silicon photodiode. An Ocean Optics USB2000+UV-VIS fiber optic spectrometer was used for electroluminescence spectra. AFM measurements were taken using a Veeco Dimension 3100. Solar measurements were taken using an Abet Technologies 10500 solar simulator, at AM 1.5G solar irradiation.

Results and Discussion

To test DC18's effectiveness as an electron injection/hole blocking layer, PLEDs using a conventional three layer structure (hole-transport layer (HTL) / active layer / electron-transport layer (ETL)) were

fabricated to ascertain changes caused by a single layer substitution. Poly(3,4-ethylenedioxythiophene) poly(styrenesulfonate) (PEDOT-PSS) was used as the hole transport layer for all devices, while two active layer polymers were chosen with different HOMO/LUMO levels to compare ETL charge injection abilities. The structure of the PLEDs was: indium tin oxide (ITO) / PEDOT-PSS (40nm) / active layer (100nm) / ETL / Aluminum (80nm). Poly[2-methoxy-5-(2-ethylhexyloxy)-1,4-phenylene vinylene] (MEH PPV) and poly[(9,9-di-n-octylfluorenyl-2,7-diyl)-alt-(benzo[2,1,3]thiadiazol-4,8-diyl)] (F8BT) were used as the emissive active layer for orange and green (respectively) PLEDs. These polymers were chosen as the well-studied, inexpensive polymers facilitate a selection route for the best ETL to subsequently be transferred to costly and more complex, higher-efficiency devices. Likewise, the straightforward three layer structure (HTL / active layer / ETL) contributes to accurate measurement of the impact seen by changing an individual layer.

The different polymers were chosen to see if the deep lower unoccupied molecular orbital (LUMO) of DC18 at -4.2 eV (shown in Figure 1B) would hinder its effectiveness as an electron injection layer, as MEH PPV has a higher LUMO value in comparison to F8BT (-2.7 eV compared to -3.5 eV). For comparison as a dual-purpose electron-transporting and hole blocking layer, DC18 was compared to a commonly used [30, 31] air stable, evaporated small molecule, 2,9-Dimethyl-4,7-diphenyl-1,10-phenanthroline, known as bathocuproine (BCP). BCP was chosen as a reference ETL due to its air-stability and excellent hole-blocking ability in place of an unstable low work function metal. With a LUMO of -2.9 eV, BCP is well matched for electron injection to the chosen active layer polymers, and its fairly high HOMO of -6.5 eV makes it an excellent hole blocking material, making the comparison with DC18 a confirmation of its usefulness as a novel, solution processed interfacial layer.

Table 1 summarizes typical results for our measured PLEDs, illustrating the enhancement seen in different parameters when switching to DC18. For the F8BT devices, the maximum brightness increased 3.5 times (Figure 2A) when comparing PLEDs with a 5 nm BCP electron transport layer to devices with a 20 nm thick DC18 layer. These devices also see a full volt enhancement in their turn on voltage, defined as the voltage where luminance is 1 cd m^{-2} , dropping from 4.1V to 2.9V,

decreasing by 25%. The efficiencies for the F8BT device improved significantly with the addition of DC18, providing a five-fold improvement in both luminescence efficiency and EQE (Figure 2B). In comparison, the MEH PPV devices have a tenfold increase in maximum brightness when switching from BCP to DC18, with an identical turn on voltage decrease (Supplementary Info). When compared to the reference, both external quantum efficiency (EQE) and luminescence efficiency for MEH PPV devices increased 3.5 times with the switch to DC18 (Supplementary Info). While there is no comparable literature data for NDIs as ETLs, the more than tripled improvement seen in maximum brightness and up to five-fold improvement in device efficiencies seen with DC18 are a greater improvement seen than previously reported for charged oligomers [19] and conjugated polyelectrolytes used as ETLs. [8, 13] Indeed, our device efficiency enhancements improve on those for recently reported non-charged small molecule ETLs. [32, 33] All the measured DC18 inclusive devices maintain this significant efficiency increase over BCP devices across the entire current range.

The overall brightness and efficiency of the reference devices, while not the highest reported, remain consistent with reported results using MEH PPV and F8BT in similar device structures, [34-36] providing evidence that they are a reproducible testing platform for novel injection layers. The brightness and turn on voltages are improved for both active layer polymers with the inclusion of DC18, but the F8BT devices see a larger efficiency improvement compared to the MEH PPV devices. Considering this difference in enhancement, it is possible that even with the improved charge injection due to the dipole effect [11-14] created by the DC18 transport layer, its efficacy with electron injection remains somewhat limited by its deep LUMO value, as the greater energy level difference for the MEHPPV/DC18 junction (1.5eV), to that of the F8BT/DC18 junction (0.7eV) is represented by a lower device efficiency enhancement for the former. Thus it can be inferred that the large energy barrier between LUMO levels does limit electron injection, but this could be offset by an increased hole-blocking ability of DC18, with its deeper HOMO level in comparison to BCP, as well as DC18 having a higher conductivity, allowing for a thicker layer (20 nm) that protects the underlying active layer polymer from thermal damage during the aluminum electrode deposition.

Further investigation into this charge injection barrier was examined by running the PLEDs under solar illumination.

When operated as a solar cell, the open-circuit voltage (V_{oc}) of the PLED reflects the built in potential across the device. [37] As only the ETL was varied in the device structures, it would be solely responsible for any measured V_{oc} shift. The cells photovoltaic response (Figure 3) makes it clear that DC18 increases the V_{oc} for both polymers, from 0.38 V to 0.82 V for F8BT and from 0.92 V to 1.13 V for MEH PPV. A correlation between increased V_{oc} and improved PLED characteristics has been demonstrated by others, relating a higher open circuit voltage with a reduced charge injection barrier into the device. [9] Additionally, other novel electron transport layers using charged materials similar to our DC18 have been shown to increase the V_{oc} in solar cells. [38] This is believed to be a shift of the device's built-in field as it aligns with the interfacial dipole created in the presence of the charged layer at the aluminum interface. [39] A greater V_{oc} shift was observed for the F8BT devices compared to MEH PPV, evidence that there is a lower electron barrier for DC18 with F8BT than when combined with MEH PPV. This is further evidence that a large difference in the LUMO levels can affect DC18's function as an electron injection layer, thus a double ETL using a second material with a better matched energy level to polymers with low LUMOs would enhance efficiency even further. Double ETLs have been used to address energy level mismatch before with excellent results, [40] and we are currently investigating similar methods for our material.

Tapping mode AFM was used for further examination of PLEDs with DC18 to probe if there were any surface morphology effects that could contribute to the differences between the ETLs (Figure 4, shown for MEH PPV). The polymer surface alone has a RMS value of 0.512 nm and a R_{max} of 4.62 nm, indicating a smooth surface in both topography (Figure 4A) and phase contrast mode (Figure 4C). In contrast, the spin cast DC18 film (20 nm) on top of the same polymer presents a rougher surface (Figure 4B) with a RMS value of 0.818 nm and R_{max} of 10.9 nm. While overall the DC18 film is rougher in comparison, the higher R_{max} value indicates distinct 'grains' of DC18 are the main contributor towards this roughness. These grains become more evident in the phase contrast image (Figure 4D) with clear crystallized regions most likely of well aligned stacks of DC18. [27] This order

could facilitate charge transport paths through the film, and contribute to the higher device efficiencies seen for both types of PLEDs. The rougher DC18 surface could foster closer interaction with the evaporated aluminum layer as a means of further enhancing charge injection. Additionally, it is possible that there are bonds being formed between the nitrogen atoms on DC18 and the aluminum further enhancing electrical contact. [41]

A visual comparison of light emission from PLEDs (Figure 5, inset), illustrates the increased brightness when switching to DC18 as the ETL. The electroluminescence spectra (Figure 5) remain essentially unchanged for F8BT devices (leftmost two red spectra), while MEH PPV devices (rightmost two black spectra) produce a modest hypsochromic (blue) shift with the switch to DC18. This shift seen for MEH PPV indicates that quenching of the red-edge of polymer luminescence is occurring with DC18, as hypsochromic shifts have been seen for MEH PPV films with zinc oxide nanoparticles having a similar LUMO level to DC18. [42] With the large LUMO difference between MEH PPV and DC18, a greater degree of quenching is more likely, creating a larger blue shift for the MEH PPV electroluminescence. While this creates potential towards combining the materials in a solar cell, it does explain the lower efficiency enhancement seen for the MEH PPV devices, and this effect could be suppressed by a double layer ETL with better energy level matching. Work is ongoing to pinpoint the exact origin of this blue shift.

Conclusions

We have demonstrated the effectiveness of using an inexpensive, highly soluble charged small molecule with the high electron mobility typical of naphthalene diimides, DC18, as an electron transport/hole blocking layer in a well-studied polymer light emitting diode platform. In spite of the energy level differences between DC18 and the active layer materials, the inclusion of this novel ETL resulted in increased brightness, significantly higher device efficiencies and reduced turn on voltages. These enhancements are attributed to a combination of an interfacial dipole created from the charged DC18, its deep HOMO ensuring it is an improved hole-blocking layer, and effective charge injection

from the aluminum cathode. The inclusion of DC18 provides up to a five-fold efficiency enhancement and has considerable potential for long term device stability. Additional research is on-going to produce double layer ETL devices to address any energy level mismatch occurring with the active layer material, and to combine DC18 in devices with higher stability hole transport layers as a means of further extending device lifetimes. [43-45]

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Figures **Figure 1.** A) DC18 chemical structure and B) energy levels (in eV) of the materials used in this study. **Figure 2.** A) Current density and luminance intensity for a F8BT PLED incorporating BCP and DC18. B) Luminance efficiency and EQE (%) for the same devices.

Figure 3. Photovoltaic response curves of MEH PPV devices incorporating BCP and DC18, and F8BT devices with BCP and DC18.

Figure 4. AFM topography images ($1 \mu\text{m}^2$) of (A) a MEH PPV film, and (B) 0.5mg/mL DC18 on top of a MEH PPV film. The corresponding phase contrast images for MEHPPV (C) and DC18 (D) show the high degree of crystallization found in the DC18 film.

Figure 5. Normalized EL spectra for F8BT with BCP (open red circle) and DC18 (closed red circle), and MEH PPV with BCP (open black triangle) and DC18 (closed black triangle). Inset: photos of PLEDs with BCP (i) and DC18 (ii).

Tables

Table 1 Summary of enhancement seen in PLED outputs with DC18

Active layer	ETL layer	Turn on Voltage (V)	Max Luminance (cd/m ²)	Max EQE (%)	Max Luminance efficiency (cd/A)
MEH PPV	BCP	4.0	89.7	0.012	0.048
MEH PPV	DC18	3.0	970.7	0.036	0.142
F8BT	BCP	4.1	981.7	0.137	1.337
F8BT	DC18	2.9	3395.1	0.709	6.899