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Low-Temperature Cross-linkable Small Molecules for Fully Solution-Processed OLEDs

Sungkoo Lee* and Min Hye Seo

Abstract: Cross-linkable hole-transporting, host, and electrontransporting materials with a new cross-linking group, uracil, are designed and synthesized. These compounds exhibited good solubility in common organic solvents and excellent solvent resistance after cross-linking at a low temperature of 120 °C. The OLED was fabricated by all-solution processing using cross-linkable synthetic compounds, except for the electrodes. This device exhibited a current efficiency of 39.2 cd/A and a power efficiency of 15.3 lm/W.

Organic light-emitting diodes (OLEDs) have attracted attention due to their excellent performance characteristics, such as low weight, low power consumption, wide viewing angle, and short response time.^[1-3] Generally, OLEDs for display and lighting applications are fabricated by vacuum deposition or solution processing. Solution processes are more desirable for large-size or flexible displays because of the possibility of employing costeffective large-area manufacturing processes, such as inkjet, rollto-roll, and screen printing.^[4-7] It is also applicable to various substrates including plastic substrates.^[8,9] To date, the best way to improve the performance of OLEDs is to form the OLEDs into a multilayer structure. The multilayer structure of OLEDs is typically fabricated by continuously depositing materials using the thermal evaporation process. In solution processing of OLEDs, when the new layer is coated, the underlayer can be dissolved by the solvent of the upper layer, which makes it difficult to fabricate a multilayer structure. As a result, few reports have been made on the fabrication of multilayer OLEDs using the solution-process, and all currently commercialized OLEDs have been fabricated by the thermal deposition process.^[10,11]

In order to solve the problem of producing a successive layer in solution processing of OLEDs, two methods have been used: orthogonal solvents or cross-linkable materials. The first method uses materials that can be coated with orthogonal solvents. That is because the orthogonal solvent dissolves only the upper layer and not the underlayer. The multilayer OLEDs can be fabricated by using materials soluble in orthogonal solvents such as water, alcohols because organic materials used in OLEDs are usually soluble in organic solvents. Nevertheless, this method has the disadvantage that only a limited number of materials are compatible with this solubility criterion. The second approach to fabricate multilayer OLEDs is the use of cross-linkable materials

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that can be cross-linked by heating or light irradiation and that are not soluble in organic solvents after cross-linking.^[10] In the upper layer coating, the cross-linked underlayer is not affected by the solvent of the upper layer, and it makes the fabrication of multilayer OLEDs possible. The cross-linkable groups chosen for previous studies include thermally and/or photochemically initiated moieties such as acrylates, benzocyclobutenes (BCBs), trifluorovinyl ethers, oxetanes, norborneol, or styrenes.^[12-17] The cross-linking materials can be divided into two process types: photochemical and thermal. The photochemical process needs photochemical initiators and ultraviolet (UV) radiation for crosslinking, which add the problems of quenching of excitons from radical initiators and causing photodamage to the OLED materials by UV light, respectively.

On the other hand, thermally cross-linking materials can be a better choice because they do not cause similar problems in OLED operation. Thermally cross-linkable materials generally have functional groups such as styrene, benzocyclobutene, and trifluorovinyl ether.^[18,19] Ma et al. reported an OLED that had a maximum external quantum efficiency of more than 10% using a BCB-based cross-linked copolymer as the hole-transport layer. This cross-linking process was accomplished by two hours of heating at 200 $^\circ\text{C}.^{\text{[20]}}$ Tang et al. utilized styryl groups to modify carbazole and 1,2,4-triazole moieties to create a cross-linkable bipolar host material.^[19] The host material with styryl groups can be thermally cross-linked between 172-190 °C. Most of the thermally cross-linkable materials mentioned above need curing at temperatures around 200 °C. Although cross-linking was achieved, the thermal cross-linking process requires high temperatures and/or several hours. However, since most plastic materials that can be used as substrates for flexible OLEDs are unstable at temperatures above approximately 150 °C, fabrication processes at low temperatures are crucial. As a result, lowering of the thermal cross-linking temperature is an important requirement in the development of flexible, solution-processed OLEDs, and a large amount of research is currently being dedicated to the development of cross-linkable materials capable of cross-linking at low temperatures; however, research on OLED materials for low-temperature cross-linking has rarely been reported.

Lee et al. recently reported that hole-transporting materials with a bicarbazole core and alkoxystyrene-based cross-linking functionalities were thermally cross-linked at 120 °C.^[21] Solution-processed multilayer OLEDs using physically cross-linking materials were first reported by C. C. Cheng and co-workers.^[22,23] They demonstrated that uracil-functionalized poly(3-thiophene) was able to form a physically cross-linked layer, resulting in high thermal stability and excellent hole injection/transporting capabilities in the solid state. Cheng et al. also synthesized an adenine-based polymer and used it as a hole-transporting

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material, thereby providing a stable hole-transporting layer to OLED devices. $\ensuremath{^{[24]}}$

Solution processing also has the advantage of being compatible with a much wider range of materials, including small molecules and polymers, as well as dendrimers. Polymers can show relatively good morphological stability and resistance to molecular diffusion compared to small molecules. The drawback of solution processing for the polymer is that it is generally more difficult to purify these materials, thereby causing critical problems in achieving long-lived devices.^[11,25] On the other hand, small molecules tend to undergo morphological changes, such as recrystallization after solvent removal, but they have the advantage of achieving high efficiency and long-term stability due to a well-defined structure with high purity. However, there have been many reports on solution-processed OLEDs using polymers, but not many reports on using small molecules.^[6,26-28]

The purpose of this paper is the development of OLED small molecules for solution processing that are cross-linked at low temperatures and are well soluble in organic solvents. For lowtemperature cross-linking, it is necessary to introduce a new cross-linking group which is not a known high-temperature crosslinking group. In this study, we have introduced uracil group as a cross-linking group. There are many functional groups being capable of hydrogen-bonding such as the pyrimidine, acrylate, urethane, et al.^[29,30] Uracil has the advantage of being capable of hydrogen bonding between uracils and forming strong hydrogen bonds. Uracil can also be used as a self-healing material using hydrogen-bonding property.^[31,32] Therefore, we selected the uracil group as a hydrogen bonding functional group. The synthesized compounds based on the uracil group are capable of being solution processed and thermally cross-linked at temperatures as low as 120 °C. As a result, we synthesized small molecules with the uracil functional group, which has been used as a holetransporting, host, and electron-transporting material in OLED devices. We chose triphenylamine as the core of the holetransporting material and carbazole and pyridine as that of the host material and electron-transporting material, respectively. The optical properties and photophysical properties of the synthesized compounds were fully investigated. We also fabricated an OLED device containing four solution-processed layers (hole-injection layer, hole-transporting layer, emitting layer, and electrontransporting layer) and then characterized the electroluminescence properties of this device. The relationship between molecular structure and the electroluminescence properties of the OLED device was systematically investigated herein.

The synthesized chemical structures and hydrogen-bonded structures are shown in Figure 1. A series of uracil-functionalized side-chain compounds were synthesized based on small molecules previously reported in the literature, which were used as the hole-transporting layer, the emitting layer, and the electron-transporting layer. All compounds were designed to have different cores; the hole-transporting material, u-TCTA, utilized a triphenylamine core, whereas the host material, u-CzTP, had a carbazole core, and the electron-transporting material, u-BmPyPB, had a pyridine core. u-TCTA had the structure in which the uracil group was bonded to the side chain of the TCTA, which was a triphenylamine-based OLED material. u-CzTP and u-BmPyPB had the structure in which the uracil group was bonded to the side chain of the CzTP and BmPyPB, which were carbazole- and pyridine-based OLED materials. TCTA, CzTP,

and BmPyPB are generally known as materials used for the holetransporting layer (HTL), emitting layer (EML), and the electrontransporting layer (ETL), respectively, in thermally evaporated OLEDs. As a result, u-TCTA was synthesized as the holetransporting material, u-CzTP as the host material, and u-BmPyPB as the electron-transporting material.



u-TCTA





Figure 1. Cross-linked u-TCTA, u-CzTP, and u-BmPyPB.

u-TCTA was synthesized at 32% yield by an aminodehalogenation reaction between 1-(3-((9*H*-carbazol-2yl)oxy)propyl)pyrimidine-2,4(1*H*,3*H*)dione and tris(4bromophenyl)amine. u-CzTP and u-BmPyPB were prepared at 32% and 47% by a Suzuki reaction and an amide reaction, respectively. The chemical structures of the synthesized compounds were confirmed by ¹H NMR. The synthetic routes are

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shown in Scheme S1 (Supporting Information). The synthesized compounds showed better solubility than the thermal-evaporated compounds with similar structures. For each of the synthesized compounds, the uracil group was connected to the OLED core structure, which endows good solubility in common solvents, facilitating film fabrication using solution-processing techniques. The solubility of synthesized compounds is summarized in Table S1.

The photophysical properties of the three synthetic compounds were investigated by room-temperature absorption and photoluminescence measurements of solid films. The synthesized compounds had short wavelengths of maximum light absorption (λ_{max}) due to limited conjugation in the molecular structures. The absorption peaks of u-TCTA, u-CzTP, and u-BmPyPB were observed at 354 nm, 300 nm, and 304 nm, respectively. The synthesized compounds exhibited strong absorption bands in the range of 240-350 nm that can be attributed to the $\eta-\pi^*$ and $\pi-\pi^*$ transitions of the whole molecules. The photoluminescence emission of u-CzTP was observed at 390 nm, which was overlapped by the absorption of Ir(mppy)₃ in the range of 350-430 nm; therefore, efficient energy transfer from u-CzTP to Ir(mppy)₃ can be expected. T1 energy levels of the synthetic compounds were determined from phosphorescence spectra. T1 levels of u-TCTA, u-CzTP, and u-BmPyPB were estimated to be 2.38 eV, 2.74 eV, and 2.67 eV, respectively. The u-CzTP as a host material had a higher triplet energy than the 2.54 eV of Ir(mppy)₃. Therefore, the triplet exciton can be confined in the emitting layer, and the triplet exciton quenching of Ir(mppy)₃ at the HTL/EML and/or EML/ETL interfaces can be minimized.

 $\label{eq:table_transform} \begin{array}{l} \textbf{Table 1. Photophysical and electrochemical properties of u-TCTA, u-CzTP, \\ and u-BmPyPB. \end{array}$

Compound	λ _{max} (abs) [nm]	T₁ [eV]	HOMO [eV]	Eg [eV]	LUMO [eV]
u-TCTA	354	2.38	5.47	3.40	2.07
u-CzTP	300	2.74	5.62	3.54	2.08
u-BmPyPB	304	2.67	6.74	3.80	2.94
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Electrochemical properties of the synthetic compounds were examined using cyclic voltammetry, with ferrocene as an internal standard and the cyclic voltammograms of them are shown in Figure S3. The synthesized compounds showed a reversible oxidation behavior, indicating that they can be used in OLEDs. The HOMO energy levels of u-TCTA, u-CzTP, and u-BmPyPB were estimated from an oxidation onset potential to be -5.47 eV, -5.62 eV, and -6.74 eV, respectively. The LUMO energy levels of the u-HTL, u-EML, and u-ETL were calculated to be -2.07 eV, -2.08 eV, and -2.94 eV, respectively, by adding the optical band gap to the HOMO energy level. The properties of the synthesized compounds are summarized in Table 1. The HOMO/LUMO energy of TCTA, CzTP, and BmPyPB were reported to be 5.7 eV/2.4 eV, 5.9 eV/2.5 eV, and 6.6 eV/2.6 eV, respectively. The HOMO/LUMO energies of the synthesized compounds with uracil groups are similar to those of the compounds with the same core structure. This indicated that the uracil group did not affect the photophysical and electrochemical properties of the core structure.

The thermal properties of the synthesized compounds were examined by differential scanning calorimetry (DSC) at a heating rate of 10 °C/min and the thermogravimetric analysis (TGA) in a nitrogen atmosphere at a scanning rate of 10 K/min. DSC thermograms of compounds are shown in Figure S4. In the synthesized compounds, the glass transition temperature (T_g) was not observed. The thermograms of u-TCTA and u-BmPyPB had only the endothermic peak of 287 °C and 284 °C, respectively. That is assigned to the melting temperature. u-CzTP had the crystallization temperature of 153 °C and the melting temperature of 190 °C, respectively. u-HTL, u-EML, and u-ETL exhibited high thermal stability with decomposition temperatures (T_d) (corresponding to 5 % weight loss) of 416 °C, 419 °C, and 547 °C, respectively (Figure S5).



Figure 2. Solvent resistance results of cross-linked u-TCTA, u-CzTP, and u-BmPyPB.

The solvent resistance of the thermally cross-linked films was investigated using UV/Vis spectroscopy. Solvent resistance test samples were prepared by dissolving 30 wt% of the synthesized compounds in chlorobenzene and then spin coating the solutions onto a glass substrate. The films were cross-linked at 120 °C for 30 min under a nitrogen atmosphere. After measuring the UV/Vis absorption of the cross-linked films, the films were dipped in chlorobenzene for 5 min. After removing the solvent, the UV/Vis spectra of the washed films were measured again. The comparison of the UV/Vis absorbance before and after dipping provided us with the solvent resistance created by cross-linking the films. The solvent resistance results for the cross-linked films are shown in Figure 2. Figure 2 shows that all of the compounds cross-linked at 120 °C had almost 100% solvent resistance. It is expected that the difference in the solvent resistance of crosslinked materials at low temperatures is due to the difference in solubility of each material. The absorption spectra of the crosslinked compounds are almost the same before and after the dipping process, indicating that the cross-linked compounds with uracil have complete solvent resistance. This also indicated that the hydrogen bonding between the uracil groups gave effective solvent resistance, such that the OLED materials were not soluble in the organic solvents. The detailed absorption spectra of the synthetic compounds with different cross-linking temperatures

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before and after dipping in the solvent are shown in Figure S6. As shown in Figure 1, the uracil groups of the synthesized compounds are hydrogen-bonded to each other by heating, so that the coated synthetic compounds form thin films having chain structures or network structures. As a result, the thin films are not dis-solved in organic solvents due to the solvent resistance provided by these structures. The cross-linking of these compounds can be analyzed using Fourier transform infrared (FT-IR) spectroscopy as shown in Figure S7. In the FT-IR spectra, noncross-linked films showed free NH stretching vibration peaks around 3,400~3,470 cm⁻¹; however, after cross-linking the intensities of the free NH vibration peaks at about 3,400~3,470 cm⁻¹ were decreased and the intensities of the NH stretching peaks at about 3180 cm⁻¹ were increased, indicating that the interaction occurred through hydrogen bonding between the uracil groups.



Figure 3. (a) Current density and luminance as a function of voltage and (b) current efficiency and power efficiency as a function of luminance for the solution processed PhOLED.

To further investigate the actual device aspects of the synthesized compounds, we fabricated OLEDs by solution processing of all the layers, except for the electrode, using the synthesized compounds. A fully solution-processed multi-layer phosphorescent OLED (PhOLED) using cross-linkable small

molecules was fabricated with the following structure: ITO/poly(3,4-ethylenedioxythiophene):poly(styrenesulfonate) (PEDOT:PSS) (30 nm)/u-TCTA (20 nm)/u-CzTP + Ir(mppy)₃ (8%) (40 nm)/u-BmPyPB (30 nm)/LiF (1 nm)/Al. In device, all of the organic layers were solution processed, and the LiF and Al were vacuum deposited to form a cathode. The PEDOT:PSS layer, acting as a hole-injection layer, was spin coated onto the ITO glass. The emitting layer was composed of 92 wt% u-CzTP as a host material and 8 wt% of the green phosphorescent dopant, Ir(mppy)₃. u-TCTA was used as the hole-transporting material. u-BmPyPB was used as the exciton-blocking and electron-transporting material. LiF and Al were thermally deposited on the top of the u-BmPyPB layer, successively. For the solution-process of all the organic layers in OLEDs, the uppermost layer, u-BmPyPB in this case, does not need to be cross-linked, only solution-process is sufficient. However, we used cross-linked u-BmPyPB in order to show that the uracil group could be applied to all layers of OLEDs. The current density-voltage, luminance-voltage, current efficiency-luminance, and power efficiency-luminance characteristics are shown in Figure 3. As shown in the inset of Figure 3(b), the EL spectrum of the device shows the emission peak of Ir(mppy)₃ at 510 nm, without a trace of the host emission at around 390 nm. This indicates that efficient energy transfer occurred from u-CzTP to the dopant Ir(mppy)₃, and that the two transporting materials, u-TCTA and u-BmPyPB, well-confined the triplet exciton within the emitting layer. The relatively low turn-on voltages of 4.6 V was observed for device. At a given constant luminance of 5,000 cd/m², the current and power efficiencies were 35.5 cd/A and 10.1 lm/W, respectively. The maximum current and power efficiencies were 39.2 cd/A and 15.3 lm/W, respectively. The solutionprocessed device exhibited a lower driving voltage, indicating that the charge injection and transport were relatively efficient. This means that the HOMO energy level of u-TCTA matched the energy level of PEDOT:PSS. The relatively low driving voltage of the solution-processed device might be attributed to the relatively low energy barrier between PEDOT/PSS and u-TCTA as shown in Figure S8. The device fabricated using the cross-linkable small molecules exhibited comparable device performance to the device fabricated by the thermal evaporator. This indicates that the new cross-linkable materials can serve as good alternatives to vacuum-deposition materials.

Since the device configuration has not been optimized and manufacturing environment is not good in the present work, further performance improvement may be expected by finetuning of device structure and improvement of the manufacturing environment. Future research will more examine the lifetime characteristics of devices with the finetuning of the device.

In summary, low molecular mass OLED materials with uracil that are capable of cross-linking have been successfully synthesized. The uracil group improved the solubility of the small-molecule compounds and made them usable for the solution process. The thermal cross-linking of OLED materials with the uracil group occurred at approximately 120 °C without the addition of an initiator and the production of byproducts. In the film state, the hydrogen bonding between uracil groups results in the enhancement of solvent resistance, and as a result, it has become possible to manufacture multilayer thin

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films using a solution process. The OLED was fabricated by all-solution processing using cross-linkable synthetic compounds, except for the electrodes. This device exhibited a current efficiency of 39.2 cd/A and a power efficiency of 15.3 Im/W. These results demonstrate that OLED materials with the uracil group have the potential to be used for solution processing of OLEDs that require multilayer thin films.

Keywords: thermal-crosslink, OLED, solution-process, uracil

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Low Temperature Cross-linkable small molecules for Fully Solution-Processed Multilayer OLEDs

OLED materials with uracil that are capable of cross-linking have been successfully synthesized. The hydrogen bonding between the uracil groups gave effective solvent resistance, such that the OLED materials were not soluble in the organic solvents. The OLED was fabricated by all-solution processing using cross-linkable synthetic compounds, except for the electrodes. The device fabricated using the cross-linkable small molecules exhibited comparable device performance to the device fabricated by the thermal evaporator. This indicates that the new cross-linkable materials can serve as good alternatives to vacuum-deposition materials.

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Figure 1. Cross-linked u-TCTA, u-CzTP, and u-BmPyPB.

Figure 1 showed hydrogen-bonded structures of the synthesized materials. This figure shows the hydrogen-binding between the uracil groups that occurs between the oxygen of the carboxyl group and amine hydrogen.

Figure 2. Solvent resistance results of cross-linked u-TCTA, u-CzTP, and u-BmPyPB.

Cross-linking studies of the synthesized materials were performed by varying the curing temperatures. Figure 2 provided the solvent resistance created by the cross-linked films with the comparison of the UV/Vis absorbance before and after washing at varying the curing temperature.

Figure 3. (a) Current density and luminance as a function of voltage and (b) current efficiency and power efficiency as a function of luminance for the solution processed PhOLED

Figure 3 shows (a) Current density and luminance curves and (b) current efficiency and power-efficiency curves of solu-tion-processed OLED and vacuum deposited OLED.

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