



3,12-Dimethoxy-5,6,9,10-tetrahydro-7,8-dicyano-[5]helicene as a new emitter for blue and white organic light-emitting diodes



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ABSTRACT

In this work, crystallographic data along with thermal, electrochemical and optical properties of 3,12-dimethoxy-5,6,9,10-tetrahydro-7,8-dicyano-[5]helicene (DTDH) were studied and reported. DTDH has an energy band gap of 2.9 eV which enable it to emit blue light in the fluorescence process. Because of high fluorescence quantum yield, i.e. 0.96, it is suitable to be used as an emissive material in an organic light-emitting diode (OLED). The device with a configuration of ITO/PEDOT:PSS/DTDH/Ca/Al emitted blue light with a peak at 475 nm. The best device showed a turn-on voltage at 4.0 V, maximum brightness at 4954 cd/m², and maximum current efficiency at 2.38 cd/A. However, up on mixing DTDH with N,N'-diphenyl-N,N'-bis(1-naphthyl)-1,1'-biphenyl-4,4'-diamine (NPB), the couple compounds form exciplex when excited resulting in a new emission spectra at lower energy with a peak at 558 nm. Consequently, White OLEDs with the configuration of ITO/NPB/NPB/DTDH(1:1)/DTDH/BCP/Alq₃/LiF/Al, in which NPB/DTDH layer was formed by co-evaporation, were fabricated. Performance and color quality of the white OLEDs can be tuned by varying thickness of the mixture layer (NPB/DTDH) and the pure DTDH layer. The best WOLED with CRI of 72 and CCT of 5096 was obtained. Evidences of exciplex formation between NPB and DTDH were observed in fluorescence lifetime and time-resolved emission spectroscopy (TRES) experiments.

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1. Introduction

Helicenes are polycyclic aromatic hydrocarbons (PAH) composing of benzene or other aromatic rings that fused at the ortho-position resulting in helical shaped molecules. In this work, we focus only on carbohelicenes in which only benzene rings were incorporated into helical shape. In spite of having highly conjugated structure, they present very low fluorescence quantum yields, e.g. 0.04, 0.04, 0.02, 0.01 for [5] [6], [7], and [8]helicenes, respectively [1,2]. Many attempts have been made to improve the quantum yield of [5]helicene as shown in Fig. 1. Stammel et al. [3,4] modified [5] helicene by adding two cyano groups acting as electron acceptor on helicene **1**, **2**, and **3**. One and two methyl groups acting as electron donor were added on helicene **2** and **3**, respectively. Quantum yield of the three helicenes was significantly improved, i.e. up to 0.12. Morrison et al. [5] synthesized 7:8,9:10-dibenzo-1,2,3,4-tetrafluorotriphenylene, **4**, in which four fluorine atoms acting as an electron acceptor were added on a [5]helicene structure resulting in an increasing of the quantum yield to 0.15. Mandal et al.

[6,7] improved a [5]helicene structure by adding two cyano groups, electron acceptor, at 7,8 positions and two methoxy groups, electron donor, at 3,12 positions forming an effective electron push-pull system. The product, 3,12-dimethoxy-7,8-dicyano-[5]helicene (DDH), obtains quantum yield as high as 0.28 making it sufficient to be employed as an emitter material in a OLED [8].

In this work, we report the use of 3,12-dimethoxy-5,6,9,10-tetrahydro-7,8-dicyano-[5]helicene (DTDH) as a novel emitter. DTDH possesses good properties such as high quantum yield, good thermal property, and even large energy band gap that make it suitable to be used as a blue emitter. Additionally, DTDH can form exciplex with N,N'-diphenyl-N,N'-bis(1-naphthyl)-1,1'-biphenyl-4,4'-diamine (NPB) which readily releases orange light. The combination of the blue light emitted from DTDH and the orange light from the exciplex produces a tunable white light emission.

2. Experimental

2.1. Materials

Unless otherwise noted, common commercial reagents were used as received from the commercial suppliers (Aldrich, Fluka and

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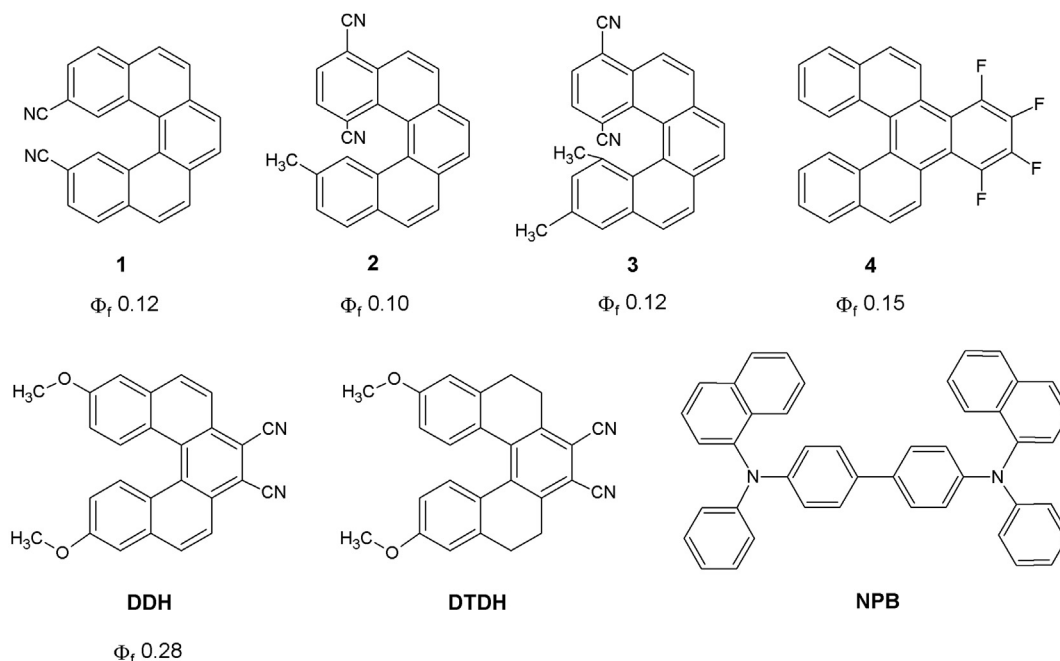


Fig. 1. Structure of [5]helicene derivatives and N,N'-diphenyl-N,N'-bis(1-naphthyl)-1,1'-biphenyl-4,4'-diamine (NPB).

Acros) without further purification. DTDH was synthesized according to the procedure in the literature [7]. Poly(3,4-ethylene dioxythiophene):poly(styrenesulfonate) (PEDOT:PSS, CLEVIOS™ P VP Al 4083) was purchased from Heraeus. N,N'-diphenyl-N,N'-bis(1-naphthyl)-1,1'-biphenyl-4,4'-diamine (NPB), 2,9-dimethyl-4,7-diphenyl-1,10-phenanthroline (BCP), and Tris(8-hydroxyquinolino)aluminium (Alq₃) were purchased from Luminescence Technology Corp. (Lumtec). Lithium fluoride (LiF) was obtained from Kurt J. Lesker company.

2.2. Instruments

UV/vis absorption spectra were taken from a Perkin Elmer Lambda950 UV/vis/NIR spectrophotometer. Photoluminescence spectra (PL) and time-resolved fluorescence spectra were obtained from a Horiba Jobin Yvon FluoroMax®-4 fluorescence spectrometer equipped with time-correlated single photon counting unit (TCSPC). Both experiments were executed at room temperature. A light source for fluorescence lifetime measurement was a 370 nm NanoLED® from Horiba. The thermal gravimetric analyzer (TGA) thermogram was obtained from a Mettler Toledo TGA/SDTA851^e thermogravimetric analyzer. The differential scanning calorimetric (DSC) trace was performed on a Mettler Toledo DSC822^e differential scanning calorimeter. Both thermal properties were studied under nitrogen atmosphere at a heating rate of 20 °C/min. All electrochemical studies were performed by cyclic voltammetry (CV) in a three electrode cell (a glassy carbon working, a platinum counter, and a Ag/Ag⁺ 0.1 M AgNO₃ in acetonitrile non-aqueous reference electrode) in a solution of 0.1 M tetrabutylammonium tetrafluoroborate (Bu₄NBF₄) in acetonitrile. All measurements were scanned to negative and positive directions at a scan rate of 50 mV/s which controlled by an Autolab PGSTAT12. All reported potentials were quoted vs. the Ag/Ag⁺ reference electrode. The same CV experiment was done on ferrocene for correction and the half wave potential of ferrocene/ferrocenium ion (Fc/Fc⁺) was 0.04 V (vs. Ag/Ag⁺). The potential of the Ag/Ag⁺ reference electrode, thus, can be estimated to 0.36 V (vs. SCE). Single-crystal x-ray experiment was performed on a Bruker APEXII CCD diffractometer, equipped with a

CCD detector, using Mo K α radiation ($\lambda = 0.7107$ Å).

All electroluminescence (EL) spectra of OLEDs were measured using an Ocean optics USB4000 spectrometer. Current density-voltage-luminance (J-V-L) characteristics were recorded by Minolta LS-110 as a luminance detector and Keithley 2420 source measure as a power supply and multimeter. The J-V-L measurements of OLEDs were carried out at room temperature under ambient atmosphere within 2 h after OLEDs were fabricated.

2.3. OLED fabrication

A substrate which is a patterned indium-tin oxide (ITO) coated-glass slide was cleaned by sonication in a detergent aqueous solution, acetone and isopropanol consecutively for 10 min each, then, dried under nitrogen stream. Subsequently, the ITO coated-glass was exposed to UV-ozone (Jelight model 42-220) for 5 min. Poly(3,4-ethylene dioxythiophene):poly(styrenesulfonate) (PEDOT:PSS) was spin-coated at 3000 rpm onto the precleaned ITO substrate yielding a thin layer of ca. 35 nm thickness which determined by a surface profilometer (Dektak 3ST). The PEDOT:PSS layer was baked at 140 °C for 10 min to remove residual solvents. A layer of small organic molecules, for example DTDH, NPB, BCP, and Alq₃, was prepared by thermal evaporation in vacuum at the base pressure of 10⁻⁶ mbar. LiF was employed as electron injection layer. The metal cathode layer was also prepared by thermal evaporation of aluminum film (ca. 100 nm) was coated onto calcium layer. An active area of the OLEDs was 3 × 3 mm². All thermal evaporation experiments were carried out using an Edwards Auto 306 at the base chamber pressure of 10⁻⁶ mbar and the thicknesses of deposited layers were monitored by a quartz crystal microbalance.

3. Results and discussion

3.1. Structure of DTDH

As illustrated in Fig. 1, DTDH contains two nitrile groups acting as an electron acceptor, and two methoxy groups acting as electron donor. The positions of these substitution groups were engineered

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