

Full-color OLEDs based on conjugated materials

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Abstract Since 1987, the possibility of realizing a new generation display based on organic light-emitting diodes (OLEDs) has inspired much interest in both academic and industrial groups. This review elucidates recent process in materials for OLEDs, approaches to improve electroluminescent properties of devices, and recent works based on conjugated materials in our laboratory.

Keywords organic light-emitting diodes (OLEDs), electroluminescence (EL), conjugated, display

1 Introduction

Electroluminescence (EL) of organic materials was first reported for anthracene single crystals by Pope's group in 1963 [1]. Weak blue fluorescence was produced under the high bias voltage of 400 V. Due to high voltage, low luminance and poor efficiency, organic light-emitting diodes (OLEDs) did not inspire much interest from the 1960s to the 1980s. The development of OLEDs was revived by the milestone work, which was reported by Tang and VanSlyke in 1987 [2]. They fabricated a double-layer EL device, containing indium-tin oxide (ITO) used as anode, aromatic diamine as hole-transporting layer (HTL), tri(8-hydroxyquinoline) aluminium (Alq₃) as both electron-transporting and light-emitting layer, and magnesium-silver alloy as cathode. The maximum luminance was 1000 cd/m² at applied voltage of 10 V, and the quantum efficiency was 1% (1.5 lm/W). Since then, OLEDs have received considerable attention because of their superior characteristics, such as self-light-emission, high luminance, super-thin width and low weight [3–7].

2 Concept and materials for OLEDs

2.1 Mechanism of OLEDs

The mechanism of electroluminescence in OLEDs may be explained as follows. The organic layers are sandwiched

between anode and cathode. In order to reduce energy barriers between electrodes and organic materials, the work function of anode material should be as high as possible and that of cathode should be as low as possible. Under bias voltage applied at two electrodes, holes are injected from fermi level of anode to the highest occupied molecular orbital (HOMO) of EL material, and electrons transport from fermi level of cathode to the lowest unoccupied molecular orbital (LUMO) of EL material. The electrons and holes migrate in opposite directions. A hole and an electron encounter each other while drifting through an emissive layer, and recombine to form exciton, including excited singlet and triplet states (so-called). The spin wavefunction of the exciton is either singlet ($S = 0$) or triplet ($S = 1$), where S is the spin quantum number, and the fraction of excited singlet and triplet states is 25% and 75%. Fluorescence only comes from decay of singlet excitons, and triplet excitons decayed by triplet-triplet annihilation or phosphorescence [8].

EL wavelength depends on energy band gap between HOMO and LUMO of emissive materials. Holes and electrons hop in organic materials from one molecule to another, and it was described well by the Poole-Frenkel equation in a wide variety of organic layers.

$$\mu = \mu_0 \exp(\beta E^{1/2}),$$

where μ is the drift mobility of charge carrier, μ_0 is the zero field mobility, β is the Poole-Frenkel factor and E is the electric field intensity [5].

Efficiency depends on the equilibrium of hole and electron mobility, and when hole and electron mobility are equal in OLED, device efficiency will reach its maximum.

Current efficiency and quantum efficiency are two of the most important critical figures of merit for OLEDs. Current efficiency η_L is defined as

$$\eta_L = L/J,$$

where L is luminance of device, and J is the corresponding current density.

Quantum efficiency is a parameter of the degree of the conversion of the current into light, including internal

quantum efficiency η_{int} and external quantum efficiency η_{ext} . η_{int} of an OLED, defined as the ratio of the number of photons emitted per electrons injected, is given by $\eta_{\text{int}} = \gamma r_{\text{st}} q$, where γ is the ratio of the number of excitons in OLEDs to the number of electrons within circuit, r_{st} is the fraction of singlet excitons, and q is efficiency of decay of singlet excitons. However, η_{ext} is defined as the ratio of the number of photons emitted from device per electrons injected. So η_{ext} is always lower than η_{int} for photon loss within device.

2.2 Classification of EL materials

The EL materials can be classified into different categories according to the different classifications. In the following, we introduce organic EL materials briefly. According to molecular weight, the materials can be classified to small molecule and polymer.

Small molecules: Since the report of superior EL characteristics of Alq₃ [2], many scientists have dedicated their effort to the research of small molecules for OLEDs. Various color-emitting EL devices including blue, green, red and white were achieved based on small molecules. For example, TPFH, THPF, DPF, Liq, LiMeq, etc. were reported to be used as blue emitter [9–15], MQPTZ, PQPTZ, TT3, TDPFPA, PAQ-NEt₂, etc. as green emitter [16–19], and DCM, Rhodamine, DCJTb, TC3, etc. as red emitter [20–23]. Figure 1 shows structures of the common EL small molecules.

Alq₃, Liq and LiMeq are also called metal-coordinated EL materials, and they have the characteristics of high quantum efficiency and good stability. The structures of metal-coordinated EL materials contain ligands and metal ions. The metal ions often used in metal-coordinated EL materials are Li⁺, Al⁺, Mg²⁺, Be²⁺, Re³⁺, Eu³⁺, Tb³⁺, Er³⁺, etc. When rare earth ions are used as metal ions, the materials are called rare earth complex EL materials. Rare earth complex Tb(acac)₃ was first reported to be used as emitting layer in OLED. The EL wavelength was 545 nm, and the full width at half maximum (FWHM) was as narrow as 10 nm [4]. Due to the merit of high color-purity, rare earth complex EL materials are promising candidates for OLEDs.

The white EL emission requires mixing of red, green and blue emission, or yellow and blue emission [20,24–27]. Small molecules have the advantages of high purity, long life and high stability [15,28], which is beneficial to their application in OLEDs. The films of small molecules are frequently deposited by vacuum-depositing and spin-coating methods. However, the shortcoming of small molecules is their serious aggregation which deteriorates their EL properties.

Polymer materials: PPV is the first polymer which was reported to be used in polymer-LED [3]. The fabrication of devices based on PPV is usually difficult due to the insolubility of PPV in common solutions. Upon that,

various soluble PPV derivatives were synthesized, such as MEH-PPV, TPA-PPV, etc [6,29,30]. Figure 2 shows their molecular skeletons. Polyfluorene (PF) is another important sort of EL polymer, which was first reported in 1991 [31]. After modification of backbone or side chain of PF, as shown in Fig. 2, the emission color from blue to red can be achieved based on PF and its derivatives [32–37]. Polymers are expected to be suitable for OLEDs due to their less aggregation and simple film fabrication by spin-coating or ink-jet printing techniques. Furthermore, the EL properties can be easily developed by modification of backbone or side chain of polymers. However, multilayer polymer-LED device is difficult to fabricate.

The EL materials can also be classified to hole transporter, electron transporter and emitting material according to their function in the devices.

Hole transporting materials: In order to obtain highly efficient OLEDs, hole transporting layer (HTL) is quite important to be inserted between anode and emissive layer. In Tang's work, diamine was used as a HTL, and hole mobility as well as quantum efficiency was dramatically increased [2]. Hole transporters usually contain electron-donor units [38–42]. For example, the commonly-used hole transporting materials of TPD and NPB [39,40] have electron-donor of triphenylamine groups as component parts, as depicted in Fig. 3.

In order to improve hole-transporting ability, organic or inorganic electron-acceptor, such as C₆₀, WO₃, ReO₃, are doped into HTL [43–45]. Hsieh et al. doped tungsten oxide (WO₃) into NPB layer [43], and increased the hole concentration of NPB layer from 1.97×10^{14} to $1.90 \times 10^{17} \text{ cm}^{-3}$ and decreased the activation energy of the resistance of NPB layer from 0.354 to 0.176 eV with the increase of WO₃ content from 0% to 16%.

Electron transporting materials: As to most OLEDs, electron is a minority carrier and the hole is a majority carrier. The employment of electron transporting layers (ETL) is essential to balance electron and hole mobility and then leads to high quantum efficiency [46,47]. Electron transporters are electron-acceptor molecules. The common electron transporters are Alq₃, Liq, PBD, BPhen, etc [2,10,48–50].

Emitting materials: emitting materials usually have high fluorescent efficiency, superior semiconductor properties, and high stability. Light-emitting layer (LEL) is either pure material or doped material, and sometimes LELs also have hole-transporting or electron-transporting ability. Some emitting materials (especially dyes) have high quantum efficiency in low concentration, and low quantum efficiency in high concentration due to serious concentration quenching effect. Therefore, they are used as guest in doped LEL, and the ratio of guest is usually low enough (about 1%) to get high quantum efficiency. The emitting materials can be classified to fluorescent and phosphorescent materials. As only the singlet excited state of fluorescent molecules contributes to light emission, the

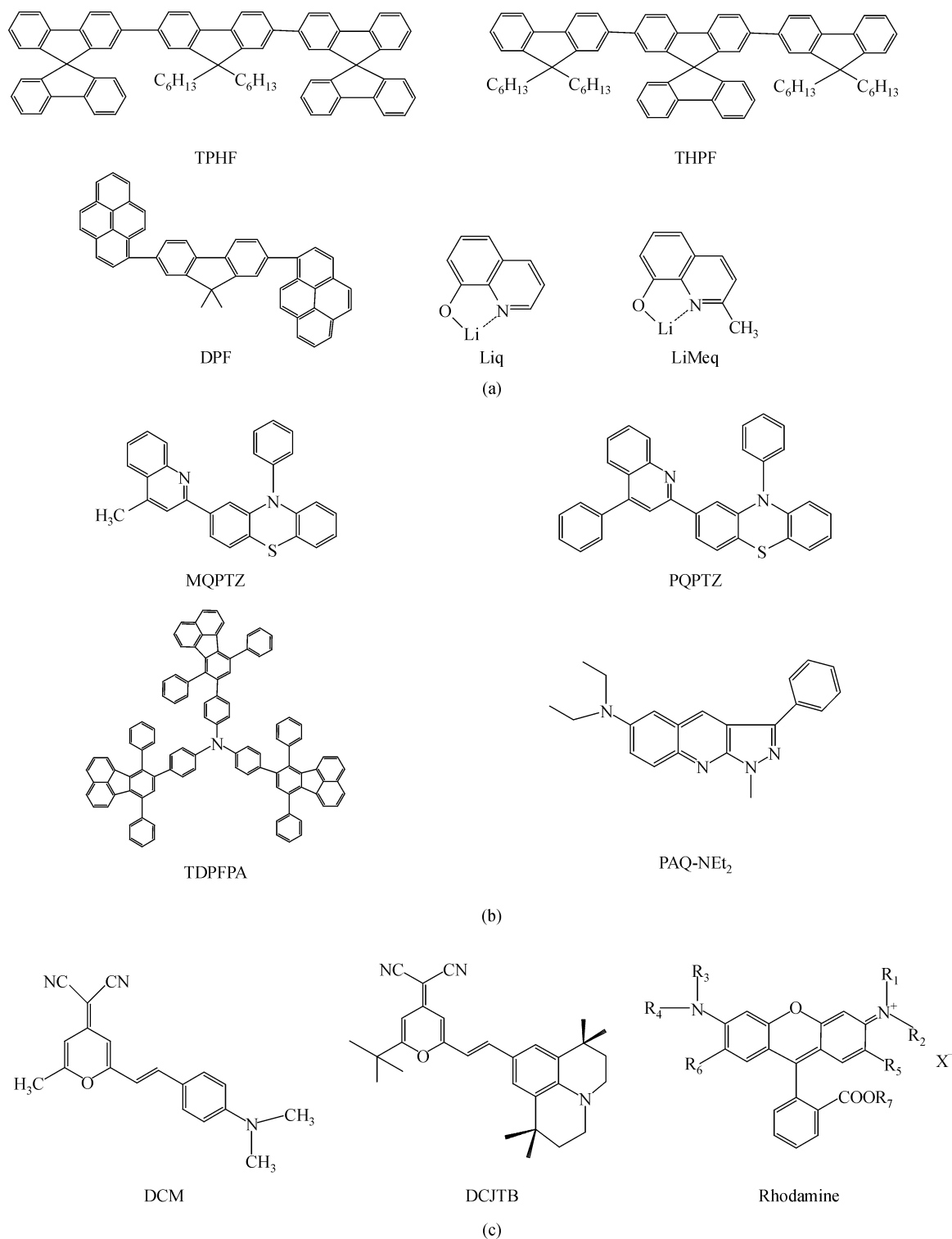


Fig. 1 Structures of common EL small molecules. (a) Blue-emitting materials; (b) green-emitting materials; (c) red-emitting materials

internal quantum efficiency of fluorescence-based OLEDs is limited to 25% [51]. However, phosphorescent materials can harvest both singlet and triplet excitons and obtain a potential maximum internal efficiency of 100%. PtOEP is the first phosphorescent material which was reported to be

used in OLEDs [52]. High-efficiency ($\geq 90\%$) energy transferred from both singlet and triplet states, in a host material doped with PtOEP, and the doped EL devices generated saturated red emission with peak external and internal quantum efficiencies of 4% and 23%, respectively.

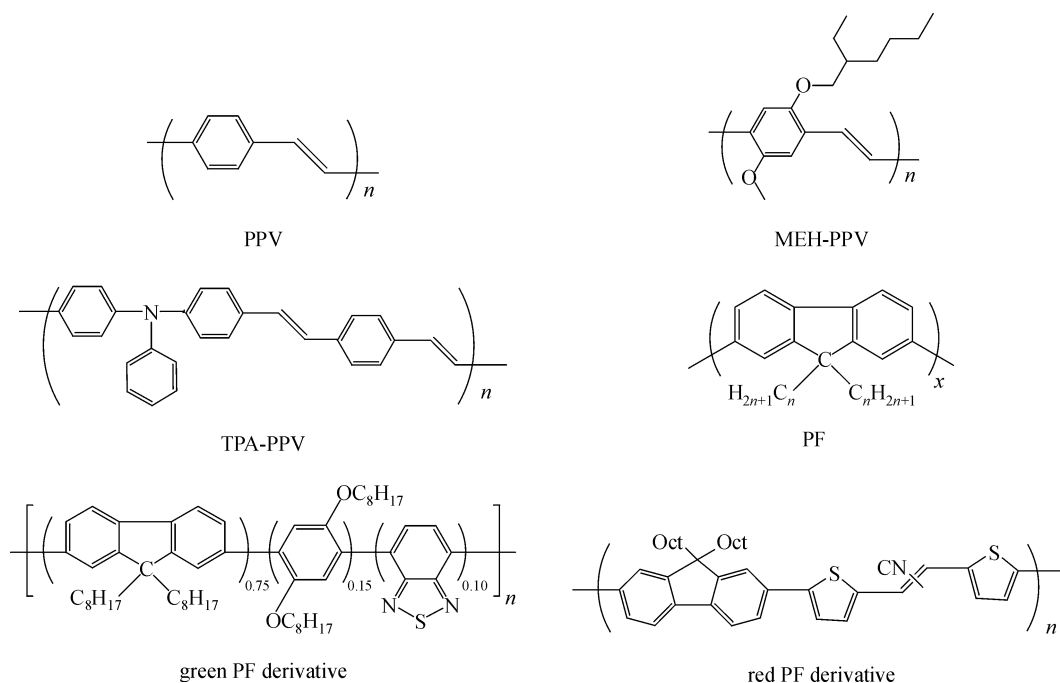


Fig. 2 Structures of PPV, PF and their derivatives

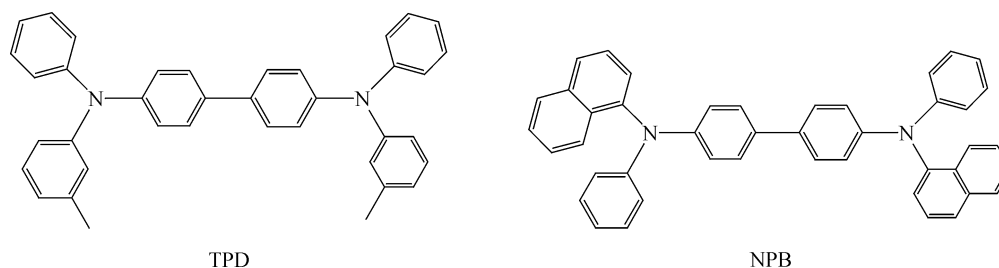


Fig. 3 Skeletons of TPD and NPB

Consequently, much attention has been focused on phosphorescent materials [53–57].

2.3 OLED structures

In addition to effort on development of EL materials, the OLED structure has also been optimized to get higher luminous efficiency. Most organic materials are p-type conductors and major charge carrier is hole. The mobility of holes in OLEDs is usually two orders of magnitude larger than that of electrons. Therefore, holes and electrons recombine in the immediate vicinity of the cathode. Due to serious electrode quenching effect, lifetime and efficiency of device are reduced. In order to balance the mobility of holes and electrons and shift the recombination zone towards the centre of the emissive layer, multilayer OLEDs are introduced. HTL, LEL and ETL are fabricated layer by layer, and the multilayer device is sketched in Fig. 4. Holes and electrons transport in different organic layers; electrons inject into emissive layer efficiently via ETL,

and the mobility of electrons enhances greatly, which is nearly equal to that of holes. Therefore, efficient electroluminescence is achieved based on multilayer structure.

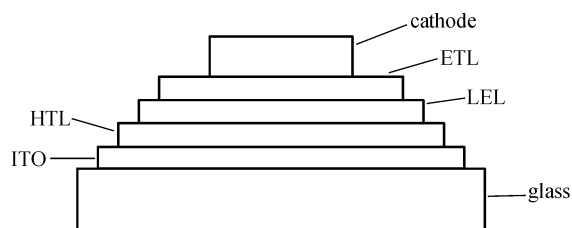


Fig. 4 Sketch of principle OLED structure

Generally speaking, there are two main ways to increase device efficiency. First, buffer layer is inserted in OLEDs to balance the mobility of charge carriers and decrease the leak current. The buffer layer between emitter and electrode can also separate electrode and emitter to reduce electrode-quenching effect. You et al. reported that

molybdenum oxide (MoO_3) as the buffer layer on ITO not only enhanced EL efficiency and stability of devices, but also reduced turn-on voltage to 2.4 V [58]. C_{60} and Mg thin layers could also act as anode buffer layers in OLEDs [59,60]. Hung et al. inserted ultrathin LiF layer between ETL and an aluminum outerlayer, and devices with LiF layer showed enhanced electron injection and high EL efficiency [61]. Furthermore, MgO, lithium manganese oxide and Al_2O_3 can also be used as cathode buffer layers [62–64].

The second improvement is to introduce microstructure into OLEDs to increase outcoupling efficiency. As the difference between refractive indices of air and organic films, light outcoupling efficiency is only about 20% for conventional OLEDs. Therefore, there is considerable potential for enhancing outcoupling efficiency by microstructures in OLEDs [65,66]. Feng et al. introduced two-dimensional (2-D) corrugated structure into OLEDs [65]. The surface plasmon propagating in all lateral directions can be coupled out into free space by the 2-D corrugated structure. The emission at normal direction was increased by a factor of 4 compared with that of OLEDs with one-dimensional (1-D) corrugated metal cathodes and was also higher than that observed from transparent substrate side in uncorrugated OLEDs.

3 Full-color OLEDs based on conjugated materials

Generally speaking, the defining feature of π -conjugated compounds is the alternation of carbon–carbon single and double bonds, respectively, in both π -conjugated small molecules and π -conjugated polymer backbones. Since the first report of electroluminescence from organic conjugated material PPV in 1990 [3], a great deal of attention has therefore been focused on OLEDs based on conjugated materials.

Conjugated small molecules have attracted much

attention over recent years owing to their high purity and high stability [9,15,28]. Zhang et al. reported two conjugated small molecules, TPHF and THPF (Fig. 1), which can be used as pure-blue-emitting materials [9]. The main EL peaks of the two materials were 428 and 404 nm respectively, which belonged to the deep blue region. The device based on TPHF got the maximum current efficiency of 1.52 cd/A, which was a high value among nondoped devices. Therefore, TPHF and THPF are promising conjugated small molecules for blue-emitting OLED.

Lin et al. reported a sky blue conjugated small molecule BUBD-1 doped in a high-band-gap host material, 2-methyl-9,10-di(2-naphthyl)anthracene (MADN) [12]. Energy can be effectively transferred from host material MADN to guest BUBD-1, due to significant spectral overlap between ultraviolet-visible (UV) absorption spectrum of BUBD-1 and photoluminescence (PL) spectrum of MADN. They used the mixed film as emissive layer and the current efficiency of the devices was as high as 13.2 cd/A.

Compared to small π -conjugated molecules, the main advantage of using conjugated polymers is their potential for large scale and low cost processing. The π (bonding) and π^* (antibonding) orbitals help holes and electrons transport in conjugated polymers. π -conjugated polymers have their semiconducting properties from delocalized π -electron bonding along their chain.

Donat-Bouillud et al. reported the synthesis of novel fluorene-based π -conjugated polymers PPF, PBPF, PTF, and PBTF, and the structures of the polymers are shown in Fig. 5 [67]. The experimental results showed that the alternated incorporation of phenylene or thiophene moieties led to tunable electroluminescent properties and the light-emission varied from blue to green (or yellow). Nowadays, more efforts focus on fluorene-based π -conjugated polymers [68,69].

PPV and its derivatives are another kind of conjugated polymers. As elucidated above, soluble PPV derivatives, such as MEH-PPV and TPA-PPV were synthesized, and

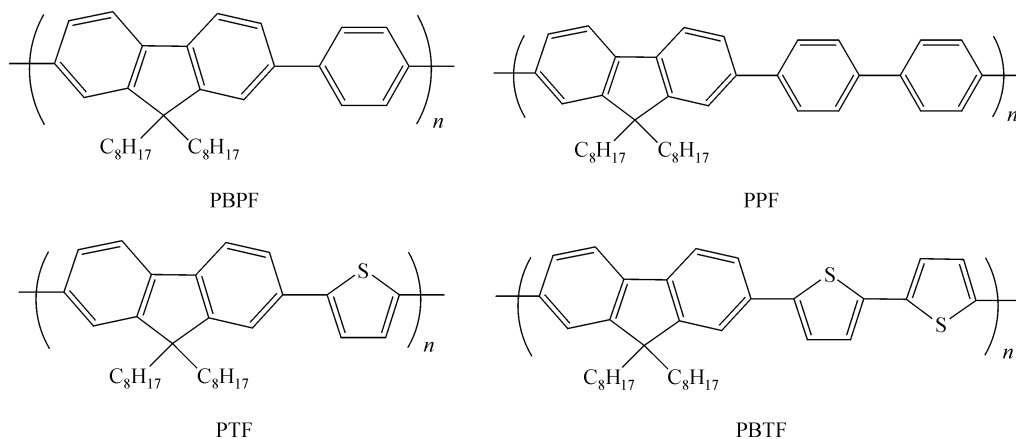


Fig. 5 Structures of fluorene-based π -conjugated polymers

the electroluminescent properties of OLEDs based on conjugated PPV derivatives were acceptable [6,29,30].

McGehee et al. obtained pure red-emitting OLEDs with a quantum efficiency of 1.1% by doping conjugated polymer CN-PPP with $\text{Eu}(\text{dnm})_3\text{phen}$ [70]. They also pointed out that blue- and green-emitting OLEDs could be made using CN-PPP and CN-PPP doped with coumarin 6. Figure 6 shows the skeleton of CN-PPP.

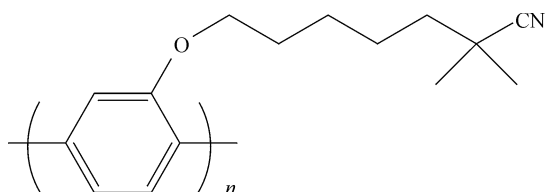


Fig. 6 Skeleton of CN-PPP

Tasch et al. reported an efficient white light-emitting OLEDs based on a blend of two conjugated polymers, and the white-emission was composed of a broad blue emission of ladder-type (polyparaphenylene) (m-LPPP) and a red-orange emission of poly(perylene-co-diethynylbenzene) (PPDB). White emission was obtained when the concentration of PPDB in the blend was 0.05% [71]. However, due to the ultra low doping ratio of PPDB, it is difficult to accurately control the doping process.

Recently, more attention has been paid to OLEDs providing single-color emission (three primary colors: blue, green and red) and white emission [3,9,12,32,33,41, 72–91]. Our group has dedicated our efforts to the investigation of OLEDs based on conjugated materials, and tricolor, yellow as well as white emission were obtained from the devices.

In the following text, recent main works for OLEDs in our lab are reviewed.

3.1 Blue-emitting OLEDs based on fluorene, polyfluorene and their derivatives

9,9-Dibutyl-N,N,N,N-tetraphenyl-9H-fluorene-2,7-diamine (DTFD) was synthesized via Ullmann condensation [48], and the synthetic route for DTFD is shown in Fig. 7. DTFD had good hole transporting property due to triphenylamine moiety, and blue emission of device ITO/DTFD/2,2-[1,2-phenylenebis(oxy)]bis(N,N-diphenylacetamide) (PBD)/Alq₃/LiF/Al came from the exciplex emission between DTFD and PBD.

We also synthesized a novel dendritic polyfluorene

derivative (PDFA), as shown in Fig. 8, containing a hole-transporting triphenylamine group [92]. PDFA had many advantages compared with polyfluorene, such as high hole-transporting capability, low aggregation and high thermal stability. Therefore, maximum luminance and quantum efficiency of single and double-layer devices based on PDFA were almost twice than those of other devices based on polyfluorene. All the experimental data prove that PDFA was a promising blue-emitting material for OLEDs.

With PDFA or poly(9,9-dibutyl)fluorene as light-emitting layer, we inserted an inorganic/organic (SiO_2 /triphenylamine) hybrid hole-transporting layer (HHTL) between anode (ITO) and emissive layer [93]. The introduction of HHTL balanced the combination of holes and electrons in light-emitting layer, and the EL performance of devices was improved.

3.2 Green-emitting OLEDs based on TT3

A novel green-emitting material of (1,3,5-triazine-2,4,6-triyl)-tris(ethene-2,1-diyl)-4,4',4''-tris(N,N-diphenylamine) (TT3) was synthesized, as shown in Fig. 9, and TT3 acted as hole-transporting and emitting layer in the device of ITO/TT3/2,9-dimethyl-4,7-diphenyl-1,10-phenanthroline (BCP)/Mg:Ag [18]. The device gave pure and bright green-emission peaking at 513 nm and the FWHM of the emission was narrowed by 13.2% compared with control green EL device of ITO/TPD:Poly(N-vinylcarbazole) (PVK)/Alq₃/Mg:Ag. Moreover, the maximum luminance was higher than that of the control device at the same voltage.

3.3 Red-emitting OLEDs based on TC3, PQP, PQM and PQTN

As shown in Fig. 9, we changed triphenylamine groups into carbazoles, and synthesized a branched molecule, 2,4,6-tris(2-(9-ethyl-9H-carbazol-3-yl)vinyl)-1,3,5-triazine (TC3). TC3 can be used as red-emitting material in OLEDs. Furthermore, we successfully fabricated white EL devices using TC3 as red-orange emitter [20], and the detailed elucidation is in following.

The EL properties of a series of novel polymers poly(3,7-N-octyl phenothiazinyl cyanoterephthalylidene) (PQP), poly(3,7-N-octyl phenothiazinyl cyanoisophthalylidene) (PQM), poly(3,7-divinylene-N-octyl-phenothiazine 2-octyl-benzotriazole) (PQTN) and poly(3,7-N-octyl phenothiazinyl terephthalylidene) (POPTP) containing phenothiazine were studied in detail [94–96] and their chemical structures are shown in Fig. 10.

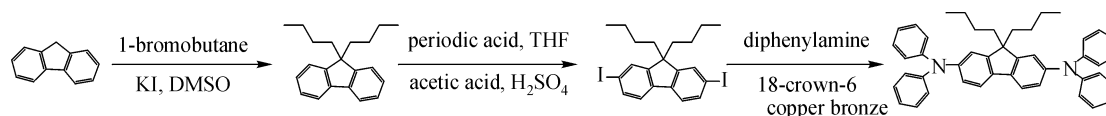


Fig. 7 Synthetic route for DTFD

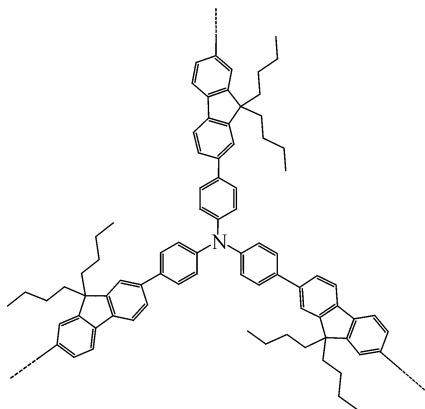


Fig. 8 Dendritic structure of PDFA

Experimental data demonstrated that the EL spectra from devices based on PQP peaked at the wavelength of 664 nm, and covered red and infrared regions [94]. As to PQM, orange-emission peaking at 608 nm was obtained, and the maximal luminance of device ITO/PQM:TPD (52 nm)/BCP (5 nm)/AlQ₃ (20 nm)/Mg:Ag was 150 cd/m² at the applied voltage of 14 V. Furthermore, the measurement of CIE coordinates indicated that the chrominance of the EL devices based on PQP and PQM was stable on bias voltage, which was beneficial to their application on display.

A nondoped orange-red emission device, ITO/poly(3,4-ethylenedioxythiophene):poly(styrenesulfonate) (PEDOT:PSS)/PQTN/Mg:Ag, was obtained, and its luminous efficiency as well as chromaticity after thermal treatment was stable [95]. The EL emission peaked at 616 nm, and the maximum luminance reached 2432 cd/m² at the bias voltage of 11 V.

3.4 Yellow-emitting OLEDs based on POPTP

Undoped and single-layer yellow-emitting device, ITO/POPTP (60 nm)/Mg:Ag (150 nm), reached the maximal luminance of 1129 cd/m² at the bias voltage of 7.2 V, and the peak wavelength, FWHM and CIE coordinates for the devices almost do not change at various bias voltage [96]. Moreover, the dominant wavelength λ_D of around 575 nm and the color purity of approximately 100% indicated that POPTP was a candidate material for pure yellow-emitting and undoped OLEDs.

3.5 White OLEDs

White OLEDs have applications in low-cost back lighting in liquid-crystal displays, full-color displays combined with color filters and various lightings. Thus, many scientists are very interested in the research of white OLEDs. However, the chrominance of white OLEDs is

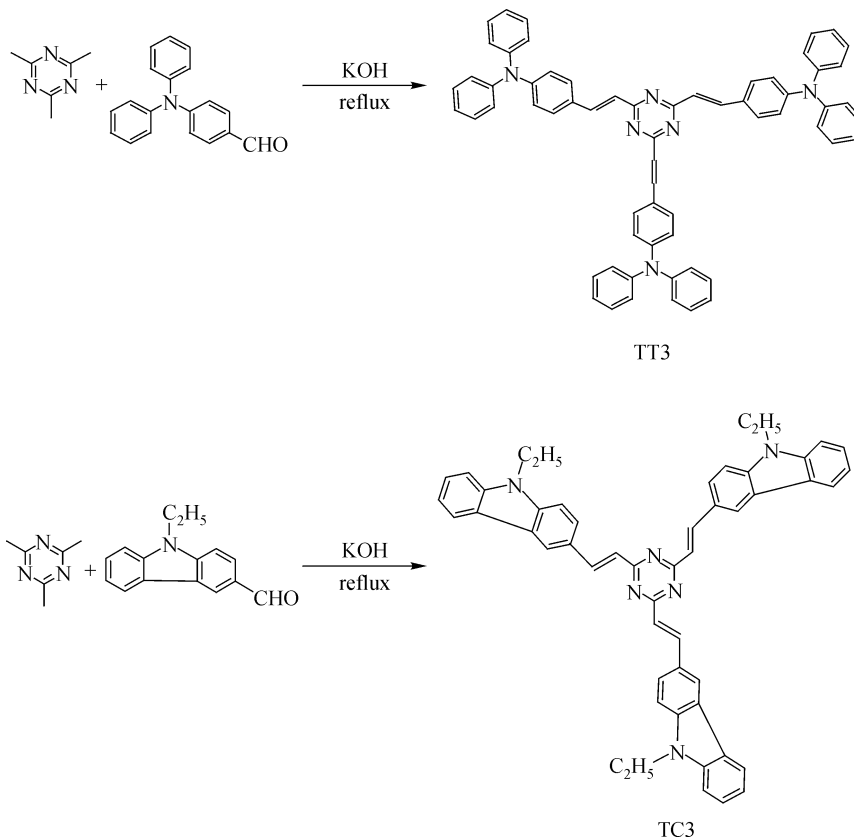


Fig. 9 Chemical structures of TT3 and TC3

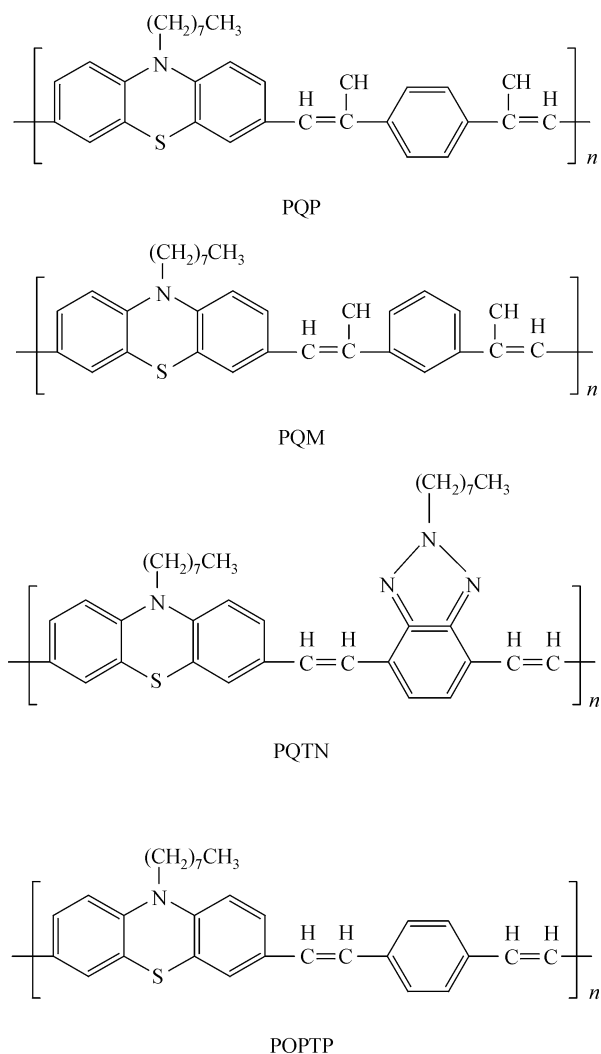


Fig. 10 Chemical structures of polymers

usually changed with varying bias voltage, which restricts the practical application of white devices.

Recently, our group has successfully fabricated white-emitting devices, and their CIE coordinates were stable on bias voltage [20]. In the device of ITO/PVK:TC3 (56 nm)/TPD (5 nm)/8-hydroxyquinolinolato lithium (LiQ) (30 nm)/Mg:Ag, TC3 acted as red-orange emitter, LiQ functioned as blue-green emitter, and TPD functioned as the adjustor for charge carrier mobility to improve the stability of white emission color on bias voltage. The performance of the device showed good quality white emission, and the CIE coordinates stabilized at (0.34, 0.39) while bias voltage varied. The maximum brightness of 1001 cd/m² was measured at the driving voltage of 17 V. We doped 4-dicyanomethylene-2-methyl-6-(p-dimethylaminostyryl)-4H-pyran (DCM) into PDFA, and fabricated white OLEDs based on PDFA as blue-green emitter and DCM as red emitter [97]. With the structure of ITO/PDFA:DCM/BCP/Mg:Ag, a true white emission covering all

visible region from 400 to 700 nm was obtained at DCM doping concentration of 0.06% in weight.

4 Efficiency enhanced by using microstructure

An inexpensive and easily controlled method was introduced, which implied a potential application in electro-optic devices [98]. Silver films with thickness varying from 60 to 360 nm were evaporated on disordered porous AAO substrates, and 75 nm Alq₃ films were deposited on Ag films. The highest quantum yield of photoluminescence of Alq₃/Ag/AAO was increased by 2.3 times compared with the samples based on flat Ag films, because the local electromagnetic field was enhanced due to the plasmon resonance of nanotextured silver films, as well as optical absorption was increased.

5 Conclusion

The materials used for OLEDs, optimizing device structure to increase luminous efficiency and our works based on conjugated materials are briefly reviewed. OLEDs have superior characteristics, such as high luminance, low driving voltage, self-light-emission, flexible panel, super-thin width and low weight. Compared with other displays, OLED is a promising candidate and eventually it will lead to a reform in display industry.

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