

行政院國家科學委員會專題研究計畫 成果報告

電激光高分子的合成與研究

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計畫主持人：許慶豐

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一、中文摘要

Part A: 我們合成含有 bis(2,2-diphenylvinyl)fluorene 的聚芴共聚高分子材料，在芴的九號碳位置以垂直交錯的方式相連接。此高分子具有著高玻璃轉移溫度及熱穩定性。由光學性質的實驗結果指出，藉由光激發的方式在聚芴的主鏈所形成的激子，可經由能量轉移的方式轉移至低能的側鏈發光。從元件的結果顯示，當操作電壓為 11 V 的情況下，以 PF4-DPVF 為發光層可得穩定的藍光放射 (0.15, 0.17)；當操作電壓為 9 V，可得到最大亮度 3137 cd/m^2 及最大外部量子效率 1.06%。除此之外，我們將 PF4-DPVF 摻混加入了 0.5% 的 MEH-PPV 來的到白光元件 CIE 為 (0.29, 0.34)，當電流密度為 119 mA/cm^2 ，可得到最大亮度 3258 cd/m^2 。我們進一步的證實了白光產生的機制是經由 Förster 能量轉移與電荷捕捉所產生。

Part B: 利用 Gilch 的合成方法，我們成功的合成了一新穎的 PPV 延伸物 poly(p-phenylenevinylene) (PPV-PP)，在側鏈具有兩個 pentaphenylene 的楔形物，並對此高分子進行結構與性質研究。我們加入了側鏈這兩個 pentaphenylene 的楔形物，除了用來防止高分子鏈間的堆疊，避免在固態下所形成激發複合體的缺陷，同時這兩個具有烷氧鏈結構的楔形物，也改善了溶解度的問題。從光物理實驗結果，我們發現了側鏈的能量可以有效的轉移至 PPV 的主鏈，並且固態薄膜的最大放射峰座落於 510 nm 和量子效率 46% 與溶液態大至相同。固態薄膜在 150 °C 加熱 20 小時，光學性質仍然穩定並無激發複合體的缺陷產生。元件的製作下，以 ITO/PEDOT/PPV-PP/Mg:Ag/Ag 為元件結

構，最大放射峰座落於 513 nm 的綠光元件 CIE (0.30, 0.62)，其最大亮度為 1562 cd/m^2 及最大發光效率 1.93 cd/A 。

關鍵詞：電激光二極體；聚芴高分子；聚對苯乙烯。

Abstract

Part A: We have synthesized polyfluorene copolymers containing bis(2,2-diphenylvinyl)fluorene pendent groups attached orthogonally to the C-9 positions of fluorene units. These polymers possess high glass transition temperatures and good thermal stability. The results from PL studies indicate that most excitons formed in the polyfluorene backbone by direct photoexcitation are likely to migrate to lower-energy pendent groups, from which emission occurs. An organic light-emitting device using the copolymer PF4-DPVF as the emitting layer exhibits a voltage-independent and stable blue emission with color coordinates (0.15, 0.17) at 11 V, with a maximum brightness of 3137 cd/m^2 at 9 V (262 mA/cm^2) and a maximum external quantum efficiency of 1.06%. In addition, we blended PF4-DPVF as the host material with 0.5 wt% of MEH-PPV to realize a white electroluminescence having CIE coordinates of (0.29, 0.34) and a maximum brightness of 3258 cd/m^2 (119 mA/cm^2). We demonstrate that both Förster energy transfer and direct charge trapping/recombination on the MEH-PPV guest are responsible for the observed EL. (Su, H. -J.; Wu, F.-I.; Shu, C. -F. "Tuning Wavelength: Synthesis and Characterization of Spiro-DPVF-Containing Polyfluorenes and Applications in Organic Light-Emitting Diodes" *Macromolecules* 2004, 37, 7197.)

Part B: Using the Gilch method, we have synthesized, a novel poly(*p*-phenylenevinylene) derivative (PPV-PP) containing two pendent pentaphenylene dendritic wedges, and have characterized its structure and properties. The incorporated side chain pentaphenylene dendrons serve as solubilizing groups, prevent π -stacking interactions from occurring between the polymer main chains, and suppress the formation of excimers in the solid state. Photoluminescence studies indicate that efficient intramolecular energy transfer occurred from the photoexcited pentaphenylene groups to the poly(*p*-phenylenevinylene) backbone. The polymer film exhibits a maximum emission at 510 nm and had a photoluminescence efficiency of 46%, which is similar to that measured in dilute solution. The photoluminescence spectra remained almost unchanged after thermal annealing at 150 °C for 20 h, and displayed inhibited excimer formation. Polymer light-emitting diodes that we fabricated in the configuration ITO/PEDOT/PPV-PP/Mg:Ag/Ag exhibited a maximum emission peak at 513 nm, corresponding to the green region [$x = 0.30$ and $y = 0.62$ in the Commission Internationale de L'Eclairage (CIE) chromaticity coordinates]. The maximum brightness and maximum luminance efficiency were 1562 cd/m² and 1.93 cd/A, respectively. (Tseng, Y.-H.; Wu, F.-I.; Shih, P.-I.; Shu, C.-F. "Poly(*p*-phenylenevinylene) Presenting Pendent Pentaphenylene Dendron Groups for Light-Emitting Diodes" *J. Polym. Sci. Part A: Polym. Chem.* 2005, 43, 5147.)

Keywords: OLED; polyfluorene; poly(*p*-phenylenevinylene).

二、Introduction

Part A: Since the discovery of poly(phenylene vinylene)-based LEDs in 1990,¹ considerable efforts have been made toward the development of new conjugated

polymers and in the performance of their related LEDs.² Organic luminescent polymers are attractive because of the flexibility available for fine-tuning their luminescence properties through the manipulation of their chemical structures and because of the feasibility of combining spin-coating and printing processes for preparing large-area flat-panel displays. Blue-emitting polymers are of special interest because they can be used either as a blue light source in full-color displays or as the host material for generating other colors through energy transfer to lower-energy fluorophores.³ Polyfluorenes (PFs) are among the most promising candidates for blue-emitting polymers because of their high photoluminescence and electroluminescence efficiencies.⁴ In addition, the facile process of functionalizing the C-9 position of the fluorene unit provides the opportunity to improve both the solubility and processability of the resulting polymers, while also offering the ability to tune the optoelectronic properties of the PFs through macromolecular engineering.⁵⁻⁸

The application of polyfluorenes in PLEDs has been hampered, however, because the formation of an undesired long-wavelength emission band occurs during device operation and results in both color instability and reduced efficiency.^{9,10} Moreover, the emissions of typical dialkylpolyfluorenes are located in the deep-blue region ($\lambda_{\text{max}} = \text{ca. } 420 \text{ nm}$) where the human eye is not very sensitive.¹¹ In this paper, we report the synthesis and characterization of fluorene-based copolymers containing bis(2,2-diphenylvinyl)fluorene (DPVF) pendent groups attached orthogonally to the C-9 positions of fluorene units. This polymer design has the advantage of permitting the incorporation of a high concentration of DPV dye without affecting the electronic properties of the polyfluorene backbone.¹² We chose DPVF dye to be the side chain because its distyrylarylene analogue, 4,4'-bis(2,2-diphenylvinyl)-1,1'-

biphenyl (DPVBi), exhibits intense fluorescence in the blue region ($\lambda_{\text{max}} = \text{ca. } 460 \text{ nm}$) and been utilized as an emitting material to achieve efficient organic blue- and white-light-emitting diodes.^{13,14} By attaching DPVF to the polyfluorene chain, energy transfer from the higher-energy polyfluorene backbone to the lower-energy DPVF pendant groups may occur, leading to emission solely or predominantly from the latter. As a result, the photoluminescence (PL) of the copolymer is fine-tuned to a blue region, which is closer to the maximum of a relative photopic luminous efficiency function.¹⁵ Another special feature of DPVBi is its nonplanar molecular structure, i.e., the phenyl rings at both ends of the molecule are forced to twist as a result of steric hindrance.¹⁶ The presence of the rigid spiro-DPVF side chain may also reduce interchain interactions and suppress the formation of long-wavelength aggregates/excimers,⁹ resulting in a stable and efficient blue electroluminescence emission bands.

Part B: Ever since poly(phenylenevinylene) (PPV) was first employed in a polymer-based light-emitting diode (PLED) in 1990,¹ organic light-emitting polymers have been subject to an intense amount of academic and industrial research because of their potential applications in flat-panel displays.²⁻⁵ Organic luminescent polymers are attractive because of (a) the ability to fine-tune the luminescence properties of polymers by manipulating their chemical structure and (b) the feasibility of utilizing spin-coating and ink-jet printing processes for preparing large-area display devices. PPV and its derivatives are among the leading candidates for light-emitting materials⁶⁻¹⁰ because they offer several advantages associated with their good mechanical properties, solution processability, thermal stability, and structural diversity; they have already been utilized in PLED applications. An ideal LED polymer, however, must possess a high

photoluminescence (PL) quantum efficiency and the relatively low quantum efficiencies of PPVs in the solid state remains an issue that needs to be resolved.¹¹ The major cause of the PPVs' low PL quantum efficiencies is mutual cofacial stacking of their conjugated backbones through favorable interchain π - π interactions, especially in the solid state, which leads to self-quenching arising from the formation of excimers.^{12,13} To overcome this problem, various bulky substituents, such as alkoxy, alkylsilyl, phenyl (aryl), and fluorenyl groups, have been attached to the PPV backbone to prevent its close packing and suppress the intermolecular interactions that lead to the formation of excimers.¹⁴⁻²⁷

Dendronized polymers, which consist of a linear, polymeric core appended with dendrons or dendrimers,²⁸⁻³⁰ are another class of macromolecules that presently is receiving a considerable degree of attention.³¹⁻³³ This dendronized polymer approach has been applied to polymers having conjugated backbones, such as poly(*p*-phenylene),^{34,35} poly(*p*-phenylenevinylene),³⁶⁻³⁸ poly(*p*-phenyleneethynylene),³⁹ poly(thiophene),⁴⁰ and poly(fluorene).⁴¹⁻⁴³ Recently, Müllen et al. reported polyfluorenes appended with pentaphenylene dendrons, anticipating that the incorporation of the three-dimensional dendritic side chain to the light-emitting polymer would improve its luminescence activity.⁴¹ They demonstrated that the shielding effect provided by the dendritic side chains on the conjugated polyfluorene backbone prevents π -stacking and suppresses the formation of aggregates/excimers. In this paper, we report the synthesis of a new PPV derivative, PPV-PP, possessing pentaphenylene dendrons incorporated as pendent units at the 2- and 5-positions of the PPV backbone. The introduction of sterically hindered pentaphenylene groups in PPV not only enhances the thermal stability but also minimizes interchain interactions and, thus, high PL quantum yields are achieved. In addition, the side substituents present two

flexible 2-ethylhexyloxy chains that improve the solubility of the polymer. We prepared PPV-PP through the macromonomer route, using the Gilch method to polymerize dendronized 1,4-bis(bromomethyl)benzene, and also investigated its thermal, photophysical, and electroluminescence (EL) properties.

三、Results and Discussion

Part A: We have developed DPVF-containing polyfluorenes having DPVF moieties as pendent groups attached orthogonally to the 9-positions of some of the fluorene units. Our PL studies indicate that color tuning can be achieved through Förster energy transfer from the higher-energy polyfluorene backbone to the lower-energy DPVF pendants, from which the emission occurs. This moderate red shift (ca 25 nm) leads to a higher luminance efficiency. An EL device based on PF4-DPVF exhibits a voltage-independent and stable blue emission having color coordinates of (0.15,0.17) at 11 V, a low turn-on voltage at 4.6 V, and a maximum brightness of 3137 cd/m² at 9 V. Moreover, we realized a white electroluminescent device having CIE coordinates of (0.29,0.34) and a maximum brightness of 3258 cd/m² using a blend of PF4-DPVF with 0.5 wt% of MEH-PPV as the emitting material.

Part B: Using the macromonomer approach, we have synthesized a dendronized polymer, PPV-PP, that consists of a conjugated poly(*p*-phenylenevinylene) backbone upon which are appended pentaphenylene dendritic wedges. The dendritic monomer **6** was prepared through Diels–Alder cycloaddition of 1,4-diethynyl-2,5-dimethyl-benzene with tetraphenylcyclopentadienone derivative **4**; it was polymerized using the Gilch method to afford PPV-PP. As a result of incorporating the dendritic pentaphenylene pendent groups, which reduce interchain interactions and suppress the formation of

excimers, this PPV-PP is soluble in common organic solvents and it exhibits a high photoluminescence efficiency in the solid state. Even after being annealed at 150 °C for 20 h, the photoluminescence spectra of the polymer film remained almost unchanged with no excimer being formed. Using this dendritic polymer, we fabricated a double-layer LED device having the configuration ITO/PEDOT/PPV-PP/Mg:Ag/Ag. The EL of the device exhibited a green light having CIE color coordinates of (0.30, 0.62). The maximum luminance efficiency was 1.93 cd/A at 824 cd/m² with a current density 42.6 mA/cm².

四、References

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