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A novel blue emission PLED material: Synthesis, photophysical and electrochemical properties

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ABSTRACT

A novel π-conjugated polymer (PCQH) containing N-benzylcarbazole and 8-methoxyquinoline units has been successfully synthesized and characterized. The polymer possesses good thermal stability with the decomposition temperature (Td) of 295 °C and the glass transition temperature (Tg) of 250 °C. The absorption and emission spectra show that the polymer can emit blue light with higher fluorescence quantum yield (81%) in $CHCl_3$. With the increase of solvent polarity, the emission peaks of PCQH display an obvious red-shifted. The study of the interactions of PCQH with electron donor (DMA) and electron acceptor (DMTP) indicates that the polymer has excellent electron transfer property. Furthermore, the interactions of PCQH with carbon materials (C_{60} and CNTs) also have been investigated. The electrochemical properties and energy levels of PCQH were calculated by the Gaussian 09 software, which are correspond to the experiment detection with cyclic voltammetry method.

1. Introduction

Polymer light-emitting devices (PLEDs) have drawn immense attention because of their large π-conjugated backbones, delocalized electronic structures, low cost, ease of modification of properties by appropriate substitution, and of solvent processability [1–3]. Among various PLEDs, the blue emitting material is scarce and desired for the development of current PLEDs [4]. Compared with organic small molecule light-emitting materials, the most obvious advantage of the polymer light-emitting materials lies in allowing adjustment of their properties by the facile design and modification [5–7].

Carbazole is a typical hole-transfer material, and these dyes containing carbazole units have excellent photo-conductivity and relatively intense luminescence. Through introducing carbazole units to the core structure of PLED materials, the thermal stability and glass state durability of these materials can be greatly improved [8–10]. Conjugated polymers with carbazole units display strong brightness and high stability. Due to carbazole with high triplet energy level and low oxidation potential, its derivations have been widely applied as reasonable host materials in OLED and PLED [11–15]. Hydroxyquinoline and its derivations as one of the most important chelators for metal ions have been applied in a variety of investigations involving metal complexes [16]. They are also applied in manufacturing dyes, food colorants, pH indicators and other organic compounds [17–20]. In the light-emitting devices field, 8-hydroxyquinoline aluminum has been widely used as the emissive and electron transporting material. It is well known that the balance of the injection of electron and hole transport is crucial for obtaining good performance LEDs. So, it is a significative work to combine the hole-transfer group, electron-transfer group and luminescence group in the same polymer molecule [21].

Based on the above thought, herein, we designed and synthesized a novel π-conjugated polymer (PCQH) containing N-benzylcarbazole and 8-methoxy-quinoline units as a PLEDs material. In the polymer, N-benzylcarbazole is a hole-transfer unit; 8-methoxyquinoline acts as both electron-transfer and luminescence roles. The absorption and emission spectra, electrochemical properties of the polymer PCQH have been studied. And the interactions of PCQH with N, N-dimethylaniline (DMA) and dimethylterephthalate (DMTP) also been investigated by fluorescence technique. The results indicate that the designed polymer has not only excellent electronic transmission stability which comes from the 8-methoxyquinoline group, but also the hole-transfer ability that belongs to N-benzylcarbazole group. The obtained results suggest that the π-conjugated polymer (PCQH) may a desired blue-emitting material in polymer light-emitting diodes (PLEDs).

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2. Experimental

2.1. Materials and instruments

Unless otherwise stated, all chemical reagents were obtained from commercial suppliers and used without further purification. Solvents used were purified and dried by standard methods prior to use. Bis(pinacolato)diboron, Pd(dppf)Cl_2, Pd(PPh_3)$_4$, dimethyl formamide, dimethyl sulfate, C_{60}, carbon nanotubes CNTs, dimethylterephthalate, 1,4-Dicyanobenzene, 5,7-dibromo-8-hydroxyquinoline and *N*-benzyl-3,6-dibromo-carbazole were purchased from Aldrich (Steinheim, Germany). Other solvents including cyclohexane, chloroform, ethyl acetate, dichloromethane, acetone and methanol of analytical-grade were purchased from Beijing Chemical Plant (Beijing, China). ^{1}H NMR and ^{13}C NMR were measured on a Bruker ARX400 spectrometer with chemical shifts reported as ppm (TMS as an internal standard). Gel permeation chromatography (GPC) measurements were performed on a Waters-410 system against polystyrene standard with tetrahydrofuran (THF) as eluant. UV–Vis absorption and fluorescence emission spectra were taken on a JASCO V-550 spectrophotometer. Fluorescence spectra were acquired with a Varian Cary Eclipse fluorescence spectrophotometer, the excitation and emission slit widths were both 5.0 nm. High-resolution mass spectra (HRMS) were acquired on an Agilent 6510 Q-TOF LC/MS instrument (Agilent Technologies, Palo Alto, CA) equipped with an electrospray ionization (ESI) source. The glass transition temperature of polymer was determined by DSC using a DSC-Q10 instrument under a nitrogen atmosphere. The decomposition temperature corresponding to 5% weight loss was detected using a Perkin–Elmer Pyris 1 TGA thermal analyzer. Cyclic voltammetry (CV) measurement was determined on a three-electrode AUTOLAB (model PGSTAT30) workstation in a solution of Bu_4NClO_4 (0.1 M) in acetonitrile at a scan rate of 50 mV/s at room temperature.

2.2. Synthesis of monomer 1, 2 and polymer (PCQH)

2.2.1. 5,7-dibromo-8-methoxyquinoline (monomer 1)

A solution of 3.02 g 5,7-dibromo-8-hydroxyquinoline (0.01 mol) and 8.28 g anhydrous potassium carbonate (0.06 mol) in 100 mL acetone was heated and refluxed for 30 min under stirring. Then, 12.6 g fresh dimethyl sulfate (0.1 mol) generally added by dropping funnel to the mixture. The whole mixture was refluxed for 24 h. Next, the reaction mixture was cooled and filtered the resulting solid. The organic solution was concentrated to 20 mL and poured into 300 mL iced water under stirred. The product was obtained by filtering and drying under vacuum in 95.6% yield. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.99 (d, *J* = 4.0 Hz, 1H), 8.52 (d, *J* = 8.8 Hz, 1H), 8.00 (s, 1H), 7.56 (t, *J* = 4.4, 4.4 Hz, 1H), 4.18 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 62.31, 116.44, 116.52, 122.55, 128.15, 133.71, 136.28, 143.58, 150.81, 153.51; EI-MS ($C_{10}H_7Br_2NO$, 316.98, m/z) 317.9 [M + 1], 418.9 [M + 2], 414.9 [M − 2].

2.2.2. N-benzyl-3,6- di(4′,4′,5′,5′-tetramethyl-1′,3′,2′-dioxaborolan) carbazole (monomer 2)

To a solution of 4.15 g *N*-benzyl-3,6-dibromocarbazole (0.01 mol), 6.09 g bis(pinacolato)diboron (0.024 mol), 0.29 g Pd(dppf)Cl_2 (8 mol %) and 5.88 g potassium acetate (0.06 mol) in 70 mL DMF was stirred at 90 °C for 12 h under nitrogen atmosphere. The reaction mixture was cooled to room temperature, poured into the 300 mL ice water, filtrated and then purified by column chromatography on silica gel with ethyl acetate/petroleum ether (1/20) as the eluant to afford a white power in 73.1% yield. ^{1}H NMR ($CDCl_3$, 400 MHz, ppm) δ 8.67 (s, 2H), 7.88 (d, *J* = 8.4 Hz, 2H), 7.34 (d, *J* = 8.4 Hz, 2H), 7.26–7.21 (m, 3H), 7.11 (dd, *J* = 5.6, 2.0 Hz, 2H), 5.52 (s, 2H), 1.38 (s, 24H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 24.99, 46.51, 83.49, 108.42, 119.39, 123.12, 126.34, 127.49, 128.11, 128.76, 132.28, 136.77, 142.85; EI-MS ($C_{31}H_{37}B_2NO_4$, 509.25, m/z) 510 [M + 1], 509 [M], 384 [M − 125].

2.2.3. Polymer (PCQH)

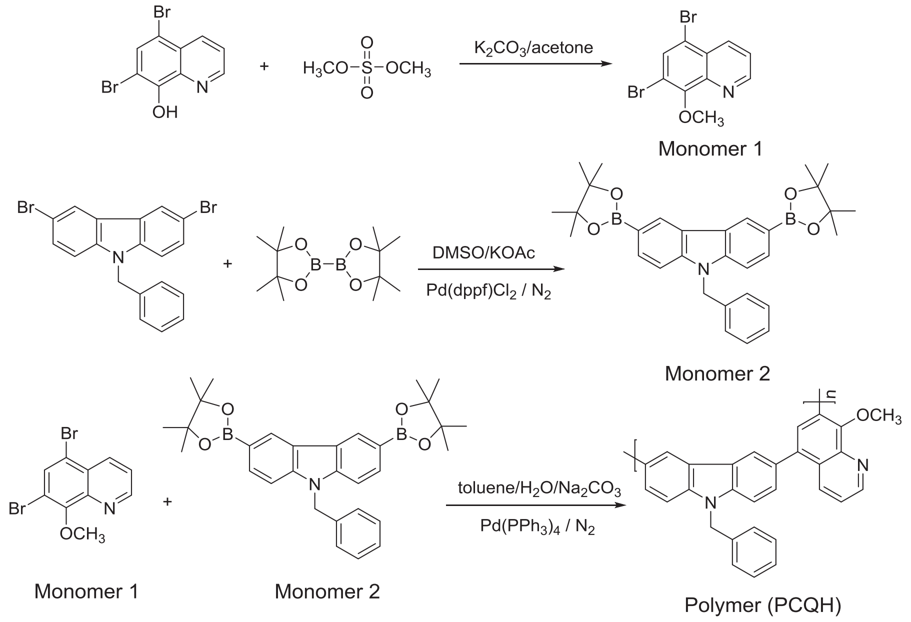
A mixture with 0.317 g monomer **1** (1.0 mmol) and 0.509 g monomer **2** (1.0 mmol) in toluene (10.0 mL), 2.0 M Na_2CO_3 (5.0 mL) was added and stirred for 30 min at room temperature under nitrogen atmosphere. Then, 50 mg Pd(PPh_3)$_4$ catalyst was quickly added to the suspension and heated to 110 °C for 48 h under nitrogen atmosphere. After the suspension was concentrated to 2–3 mL, 50 mL chloroform was added to the suspension. The obtained mixture was washed with water for three times. The combined organic layer was dried over anhydrous $MgSO_4$. After removal of a mass of chloroform, the residue was precipitated with methanol and the precipitate was collected by centrifugation. The crude polymer was purified by precipitation for twice from chloroform into methanol. The final yellow solid product was obtained through centrifugation and drying under vacuum in 39.7% yield. ^{1}H NMR (400 MHz, $CDCl_3$, ppm):^{1}H NMR (*d*-DMSO, 400 MHz, ppm) δ 8.98 (s, 1H), 8.61–7.76 (m, 2H), 7.93–7.74 (m, 2H), 7.68–7.55 (m, 5H), 7.33–7.26 (m, 5H), 5.79 (s, 2H), 4.12 (s, 3H); ^{13}C NMR (*d*-DMSO, 100 MHz) δ 46.43, 62.18, 110.18, 115.79, 122.56, 123.09, 128.95, 129.26, 130.71, 131.99, 132.46, 133.74, 136.40, 138.19, 143.29, 151.92, 152.37; M_n = 29,715, M_w = 35,692, PDI = 1.21.

3. Results and discussion

3.1. Synthesis and photophysical properties of conjugated polymer PCQH

The π-conjugated polymer (PCHQ) containing hole transport (carbazole) and luminophore (8-methoxyquinoline) units was obtained via Suzuki coupling of monomers 1 and 2 within yield of 39.7% (Scheme 1). The UV–vis absorption and fluorescence emission spectra of conjugated polymer (PCHQ, 1.0 × 10^{-6} mol/L) were determined in $CHCl_3$ and showed in Fig. 1. As can be seen from Fig. 1, the maximum absorption peak of polymer PCHQ is about 300 nm with two major absorption bands at 230–270 nm and 280–330 nm, which mainly comes from the characteristic peaks of carbazole group. And, the corresponding optical energy band gap of PCHQ is 4.13 eV by calculating from the absorption band edge of the absorption spectrum. The fluorescence spectrum of PCQH exhibits that it has a strong fluorescence emission band at 370–590 nm with an emission maximum (λ_{em}) at 435 nm, which is matched with ideal blue emitting materials. The fluorescent quantum yield (Φ) of PCQH in $CHCl_3$ was 0.81% based on quinine sulfate in 0.10 M sulfuric acid as the reference [22].

Next, the photophysical properties of PCQH in different solvents were observed by fluorescence instrument. Fig. 2 gives the photoluminescence (PL) spectra of PCQH in different organic solvents including methanol, acetone, ethyl acetate, chloroform, dichloromethane, *n*-hexane. The PL spectra vary from 420 nm to 460 nm with the increase of solvent polarity. In addition, the emission bands display good normal Gaussian shape and their half bandwidths ($\lambda_{em½}$) increases significantly with the increase of the solvent polarity. The Stokes shift for PCQH is prominent in polar solvents such as dichloromethane, acetone and ethanol. The obvious solvent effect can be explained as following: the polymer has a unique D–π–A structure, which comes from the electron donor (carbazole unit) and the electron receptor (quinoline unit). Based on the D–π–A structure, the intramolecular charge transfer (ICT) action of the polymer takes place with the increase of polar solvents and the extent of the action depends on the size of the



Scheme 1. The synthesis routes of monomers and polymer PCQH.

solvent polarity. So the emission spectra are obvious red-shifted with the increase of solvent polarity.

3.2. The interactions of PCQH with electron donor and acceptor

In order to study the mechanism of molecular interaction, the fluorescence quenching technique was adopted on many experiments. Due to DMA acting as a typical electron donor and DMTP as a typical electron acceptor, the electron transfer property of PCQH was investigated by the interactions of it with DMA and DMTP. The interactions of PCQH with DMA or DMTP were showed in Fig. S1 and Fig. S2. It can be seen from these figures that the fluorescence of PCQH can be quenched and the quenching processes follow the Stern–Volmer equation $F_0/F = 1 + K_{SV}[Q]$, where the F_o and F are the emission intensities of PCQH in the absence and presence of DMA or DMTP, [Q] is the concentration of DMA or DMTP. Accordingly, the quenching coefficients, K_{SV}, are $8.76 \times 103\ M^{-1}$ (DMA) and $7.9 \times 103\ M^{-1}$ (DMTP), respectively (Fig. 3). These results display that the conjugated polymer PCQH is an electronic storage body and its electron can transfer freely on π-conjugated system, which is desired for the PLED materials.

3.3. The interactions of PCQH with C_{60} and CNTs

Due to their remarkable electron transfer property, C_{60} and CNTs have been widely utilized as photo-electron materials in chemistry, physical and materials fields. Herein, we investigated the interactions of polymer PCQH with C_{60} and CNTs in static solution, respectively. Fig. 4 gives the interaction processes of PCQH and C_{60}. As we have seen in Fig. 4, with the gradual increasement of C_{60}, the fluorescence of PCQH was quenched efficiently and the process was also following the Stern–Volmer equation. The apparent quenching constant was $3.79 \times 10^4\ M^{-1}$, which indicates that the strong interaction between PCQH and C_{60} happens in the excited state. Similarly, the electron transfer process between PCQH and CNTs also can be observed (Fig. S3). The results can be explained by two aspects. Firstly, the large π-conjugated system of C_{60} and CNTs may

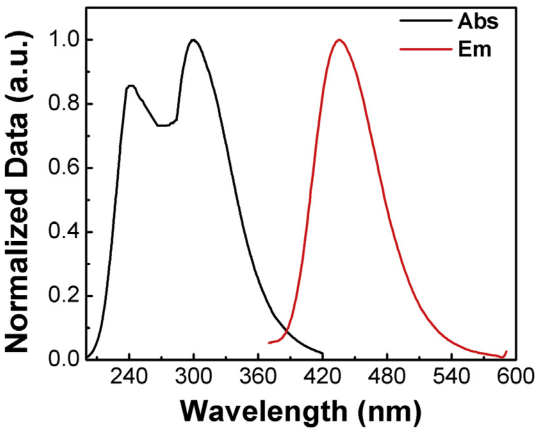


Fig. 1. The UV–vis absorption and fluorescence emission spectra of polymer PCQH (1.0×10^{-6} mol/L) in $CHCl_3$.

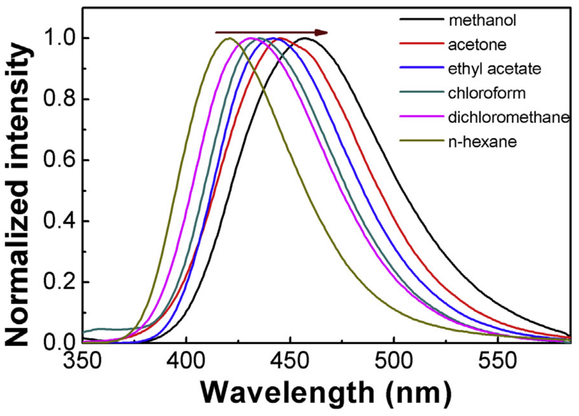


Fig. 2. Fluorescence spectra of conjugated polymer PCQH in different solvents. Determined method: the stock solution (PCQH, 1.0×10^{-3} mol/L in $CHCl_3$ was added to the corresponding solvents respectively, and the final solutions were all 1.0×10^{-6} mol/L).

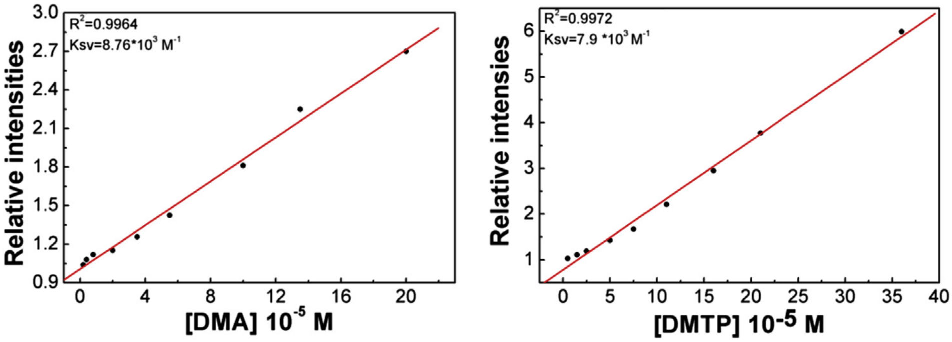


Fig. 3. The interaction fluorescence spectra of PCQH (1.0×10^{-6} mol/L) with DMA or DMTP in $CHCl_3$, respectively.

change the configuration of PCQH by π-π interaction. Secondly, due to the rapid photo-induced charge transfer from excited PCQH to C_{60}, charge transfer of conjugated system may be dramatically modified and distorted [23].

3.4. Thermal properties of conjugated polymer PCQH

Thermal property is one of the most parameters for evaluating the PLED materials. The thermal properties of PCQH were evaluated by thermal gravimetric analysis (TGA) with a heating rate of 5 °C/min and differential scanning calorimetry (DSC) under the nitrogen atmosphere. The polymer exhibits high thermal stability. The decomposition temperatures (Td, 5% weight loss) is 295 °C (Fig. 5), which might be attributed to its large molecular mass. And the glass transition temperatures (Tg) of the polymer is 250 °C. The result indicates that PCQH has good thermal stability and high glass transition temperature, which would be facilitated for the opto-electronic device applications.

3.5. Theoretical calculation

Theoretical calculation for its energy levels is an effective method for obtaining a reasonable qualitative indication of the excitation and emission properties of a conjugated polymer [24]. Based on density functional theory (DFT) and taking one repeat unit as a model, the geometries and electron density distributions of the HOMO and LUMO energy levels of PCQH were obtained in Fig. 6. As can be seen Fig. 6, the HOMO energy level of PCQH is −5.14 eV and electron density mainly distributes on the carbazole moiety. The LUMO energy level of PCQH is −1.06 eV and electron density distribution mainly distributes on the quinoline moiety. In addition, complete localization of the HOMO and LUMO is essential for efficient hole and electron transport and for the prevention of reverse energy transfer as well [25].

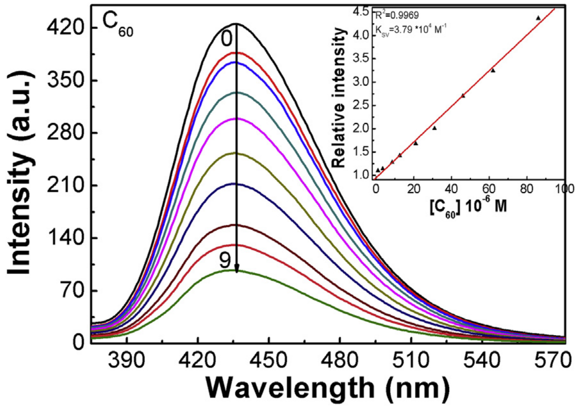


Fig. 4. The interaction fluorescence spectra of PCQH (1.0×10^{-6} mol/L) with C_{60} in $CHCl_3$. The concentrations of C_{60} are 1.0×10^{-6} mol/L, 3.5×10^{-6} mol/L, 8.5×10^{-6} mol/L, 1.25×10^{-5} mol/L, 2.1×10^{-5} mol/L, 3.1×10^{-5} mol/L, 4.6×10^{-5} mol/L, 6.2×10^{-5} mol/L, 8.6×10^{-5} mol/L, respectively.

3.6. Electrochemical properties

The electrochemical properties of PCQH were studied by CV with saturated calomel electrode as the reference electrode. Based on the supporting electrolyte (0.10 M tetrabutylammonium perchlorate) under nitrogen atmosphere, the electrochemical experiments were investigated on 1.0 mM PCQH in acetonitrile. As can been seen from Fig. 7, one peak and one reversible reduction peak were observed within the entire electrochemical window. The reversible oxidation peak is around +0.5 eV, which mainly comes from the oxidation of the carbazole groups. The reversible reduction peak is around −1.1 eV are due to electron injection into the vacant *p*-orbital of quinolone. The reduction potential (−1.1 eV) of conjugated polymer suggests that the electron distribution of LUMO lies in the quinolone moiety. The HOMO energy level is −5.25 eV, which is calculated by the empirical equation [HOMO = −(Eox + 4.5 + 0.24) eV]. The HOMO energy level suggests that the electron distribution mainly on the carbazole moiety, which indicates the PCQH can be used as a good hole-transport material. The energy gap (Eg) of PCQH is 4.13 eV, which is calculated from CV data well agreed with the UV–vis onset result. Thus, the LUMO energy level is −1.12 eV, which is calculated by the HOMO and Eg. The HOMO, LUMO and Eg are all very close to the theoretical values.

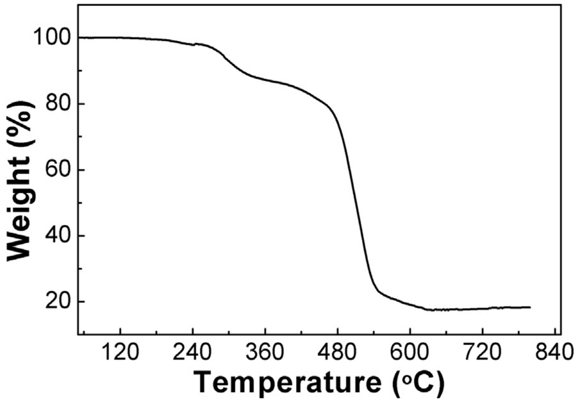


Fig. 5. TGA thermogram of conjugated polymer PCQH with a heating rate of 5 °C/min under nitrogen atmosphere.

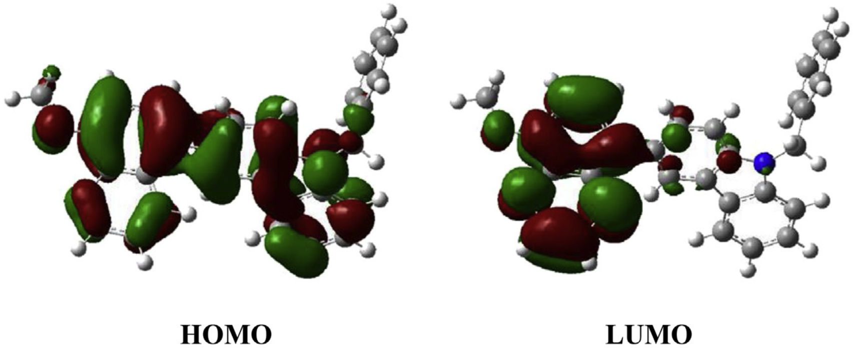


Fig. 6. Selected computed (Gaussian03) orbital plot for PCQH.

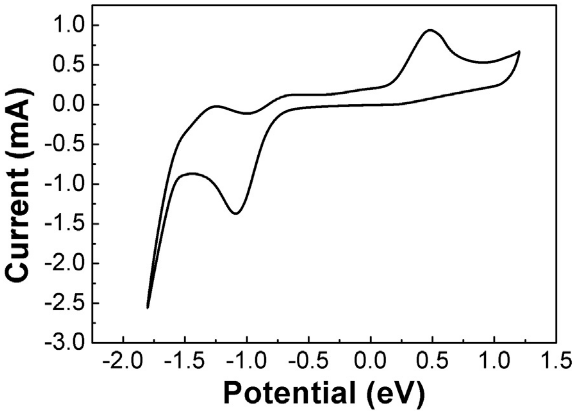


Fig. 7. Cyclic voltammogram of PCQH, the supporting electrolyte is 0.10 M Bu_4NClO_4 in acetonitrile and the scan rate is 0.10 V/s.

4. Conclusion

In summary, we successfully prepared a conjugated polymer as PLED material with carbazole and quinolone functional groups. The photophysical and electrochemical properties of PCQH indicate that it has the following advantages: (1) it is a new PLED material with good π-electron delocalization and conjugation features; (2) the PLED material contains both hole-transfer group (carbazole) and luminophore (quinolone); (3) the material has good thermal stability and high glass transition temperatures; (4) the conjugated polymer as a PLED material displays excellent optical and electrical characteristics. In view of above points, the conjugated polymer is a potential PLED with desired optical and electrical characteristics. Further photoelectric device test of PCQH is in progress and reported subsequently.

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Appendix A. Supplementary data

Supplementary data related to this article can be found at http://dx.doi.org/10.1016/j.dyepig.2015.02.016.

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