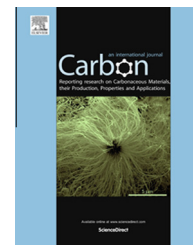


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An easy approach of preparing strongly luminescent carbon dots and their polymer based composites for enhancing solar cell efficiency



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ARTICLE INFO

Article history:

Received 8 October 2013

Accepted 31 December 2013

Available online 8 January 2014

ABSTRACT

We report a one-step approach for synthesis of carbon dots (CDs) under mild conditions. By using (N-(2-aminoethyl)-3-aminopropyl)tris-(2-ethoxy) silane (KH791) as catalyzer, stabilizing and passivation agent, the resultant CDs are allowed to be self-assembled in solution and exhibit excitation wavelength independent photoluminescence (PL) performance. When the solvent is removed, luminescent CDs filled polysiloxane composite is readily yielded through hydrolytic condensation between KH791 and CDs. According to this habit, CDs can be conveniently utilized by coating the hybrid solution on proper substrates, etc. On the basis of the luminescent down-shifting behavior of the composite with CDs, power conversion efficiency of polymer-fullerene-based bulk heterojunction solar cell is increased by about 12% due to effective light conversion of near ultraviolet and blue-violet portions of sunlight. The simple preparation, easy processing and unique PL properties of CDs and their polymer composite provide the latter with broad applicability.

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1. Introduction

Luminescent carbon dots (CDs) have attracted increasing interests owing to their excellent optical and conductive properties. Compared with conventional semiconductor nanodots and organic dyes [1], CDs are characterized by low cytotoxicity, chemical inertness, low photobleaching, cost effectiveness, and excellent biocompatibility. As a result, they have broad application prospects in many technologies, including bioimaging [2–4], sensing [5–8], photocatalysis [9–11], fluorescent ink [12–14], light emitting diode and solar cells [15–18].

So far, a variety of methods have been proposed to prepare CDs since the first report of CDs in 2004 [19], such as laser ablation [2,3], pyrolysis [20,21], combustion [22], hydrothermal process [6,23], template [24,25], electrochemistry [11,26,27], ultrasonic and microwave synthesis [13,28,29]. Relatively complicated procedures and/or strict conditions are required, which hinders large-scale production. With respect to practical application of CDs in solid-state devices, furthermore, there is an open challenge. That is, severe fluorescence quenching used to occur in dried CDs due to aggregation of these tiny particles [13].

In this work, we put forward a one-step low temperature solution approach to settle the aforesaid issue. L-ascorbic acid

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<http://dx.doi.org/10.1016/j.carbon.2013.12.092>

(L-AA) acts as the carbon source as it is more reactive and acidic than other organic acids, sugar, and amino acids [30]. (N-(2-aminoethyl)-3-aminopropyl)tris-(2-ethoxy) silane (KH791) plays the role of stabilizing and passivation agent, which leads to self-assembling of CDs and excitation wavelength-independent photoluminescence (PL) in solution. What is more, the hybrid comprising silane KH791 and CDs can be further converted into solidified composite through hydrolytic condensation in the course of solvent removal, so that fluorescence quenching is prevented. By taking advantage of the luminescent down-shifting (LDS) ability of the resultant polysiloxane based composite with CDs, power conversion efficiency (PCE) of inverted polymer-fullerene-based bulk heterojunction (BHJ) solar cell made from a blend of poly(3-hexylthiophene):[6,6]-phenyl-C61-butyric acid methyl ester (P3HT:PCBM) is obviously improved because of the effective light conversion of near ultraviolet and blue-violet portions of sunlight.

2. Experimental

2.1. Reagents and materials

KH791 was purchased from Beijing Shenda Fine Chemical Co., Ltd., China. L-AA (99.99%) was supplied by Aldrich. 2-Ketogulonic acid (99%) was supplied by Alfa Aesar. P3HT and PCBM were purchased from Echo Chemical Co., Ltd., Taiwan, China. Ag was purchased from China New Metal Materials Technology Co., Ltd., China. ZnO was prepared according to the literature [31]. MoO₃ was purchased from Beijing Aglaia Technology Development Co., Ltd., China. Other chemicals were purchased from Guangzhou Chemical Reagent Factory, China. Ultra-purified water was used throughout all experiments.

2.2. Preparation of CDs

Typically, 2.64 g L-AA was dissolved in 300 ml mixture solution of ethanol and water (1:1, v/v). Then, 7.92 g KH791 was added and the reaction proceeded at 92 °C for 12 h. The resultant light yellow solution was filtered through 0.2 μm polytetrafluoroethylene membrane to remove large particles or other impurities. The as-obtained product was directly used for making LDS layer of solar cell (see the next section), while the product for various characterizations was neutralized through dialysis (membrane molecular weight cutoff ~1000) against a large volume of ultra-purified water. The reference sample was prepared under the same condition but KH791 was excluded. 0.1 mol/L NaOH and 0.1 mol/L HCl were used to adjust pH value of the solution containing CDs.

2.3. Preparation of P3HT:PCBM based solar cell

The inverted BHJ solar cell is based on a blend of P3HT and PCBM. The glass substrate covered by transparent electrode of indium tin oxide (ITO) with sheet resistance of 50 Ω/square was ultrasonically cleaned in acetone, alcohol, and ultra-purified water for 10 min, respectively, and then treated by O₂ plasma for 10 min. Afterwards, zinc oxide (ZnO, ~30 nm

thick) was spin coated on the substrate. Having been dried at 150 °C for 10 min, a film (~50 nm thick) of the blend of P3HT:PCBM with a weight ratio of 1:1 in chlorobenzene (10 mg/mL) was further spin-coated on ZnO. This active layer was covered by evaporated MoO₃ (~10 nm thick) and Ag electrode (~100 nm thick) in 6 × 10⁻⁴ Pa vacuum. For examining the effect of the composite with CDs, the solution containing CDs without purification was spin-coated on the other side of the glass (~20 μm thick), and dried at 70 °C in atmosphere overnight.

2.4. Characterization

Transmission electron microscopy (TEM) and High resolution transmission electron microscopy (HRTEM) images were taken by FEI Tecnai G2 F30. Atomic force microscopy (AFM) images were recorded on Bruker Multimode-8. Ultraviolet-visible (UV-vis) absorption spectra were collected using PE-Lambda 750 UV-vis-NIR spectrophotometer. PL were obtained by a FLS920 combined time resolved & steady state fluorescence spectrometer (Edinburgh Instruments) using the 350 nm line of an Xe lamp as the excitation source and R1527 photomultiplier tube as the detector. For the fluorescence lifetime measurements, a pico-second pulsed diode laser was used to excite the samples. The pulse width, wavelength, and repetition rate were chosen as 100 ps, 405 nm, and 5 MHz, respectively. X-ray photoelectron spectroscopy (XPS) measurements were performed on an ESCALAB-MKII 250 photoelectron spectrometer (VG Co.) with Al K_α X-ray radiation as the X-ray source for excitation. Infrared spectra were collected on a VERTEX Fourier transform infrared (FTIR) spectrometer (Bruker). Nuclear magnetic resonance (NMR) was done on AVANCEIII400 (Bruker). Current density-voltage (*J*-*V*) measurements were performed on a Keithley 236 source meter under 100 mW/cm² simulated AM1.5G illumination. External quantum efficiency (EQE) measurements of the solar cells were performed using Solar Cell Scan100 (Beijing Zolix Instruments Co., Ltd.). The same device was also used for the solar cell with the LDS layer to ensure for direct comparison. Quantum yield measurements were conducted according to [14]. A quinine sulphate in 0.1 M H₂SO₄ solution (literature quantum yield of 54% at 360 nm) was used as a standard. The calculation of quantum yield followed the equation: $Y = Y_{st}(I/I_{st})(F_{st}/F)(n/n_{st})^2$, where *Y* is the quantum yield, *I* is the integrated area under the emission spectrum, *F* is the absorbance at the excitation wavelength, and *n* is the refractive index of the solvent. The subscript “st” refers to standard with known quantum yield. To minimize the reabsorption effect, absorption in the 10 mm fluorescence cuvette was kept below 0.10 at the excitation wavelength.

3. Results and discussion

The synthesis of CDs is rather simple here. KH791 was added to the solution of L-AA, ethanol and water, and then the reaction proceeded at 92 °C for 12 h, giving birth to silane modified CDs. Having been dried at 70 °C, KH791 was polymerized and the composite consisting of CDs and polysiloxane was yielded [21].

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