



Full paper

Preparation of sub-square-meter-sized organic semiconductor films for photovoltaics applications

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ABSTRACT

Large scale organic semiconductor films with precisely-controlled thickness are highly demanded in photovoltaic and other applications. Yet it is still challenging to prepare such kinds of organic films with conventional solution-based techniques. Here we report a simple interfacial-assembling method to fabricate various films of organic semiconductors. Sub-square-meter sized organic films of Poly (3-hexylthiophene) (P3HT) and [6, 6]-phenyl-C₆₁-butyric acid methyl ester (PC₆₁BM) with controllable thicknesses from ~ 12 nm to ~ 140 nm were obtained. Under the guidance of an ultra-fast precursor film at the water-oil-air interface, the solution of P3HT and PC₆₁BM quickly spread over the water surface in several seconds. After the evaporation of the solvent, a uniform thin film is formed with 100% utilization of the materials. This utilization indicates a power-per-weight of active materials fed as high as 152.09 W g⁻¹, which is about ten times of P3HT:PC₆₁BM-based solar cells prepared by conventional methods. In addition, the resultant films floating on the water surface are easily transferred to various water-insoluble substrates. The so-produced paper-based flexible photodetectors show good performances. This interfacial assembling strategy with general validity toward organic films is promising in future applications.

1. Introduction

Organic semiconductors have shown great potentials as feasible candidate materials for electronic [1–4], sensing [5,6], and energy conversion applications [7–10]. Many techniques such as thermal evaporating [11,12], spin coating [13–16], dip coating [17] and solution shearing [1,18,19] have been developed to deposit organic thin films. However, there are many challenges in the solution-based techniques. Firstly, it is still difficult to prepare large area films with uniform thickness. Secondly, pinning often happens at the liquid-solid-air interface, leading to the aggregation of the solutes or dispersants with un-uniformity and sometimes the formation of “coffee ring” [20–22]. Thirdly, the severe waste of precious organic semiconductor materials is unavoidable in spin coating, dip coating, and solution shearing processes. Fourthly, a suitable wettability and surface property of substrates toward the precursor solutions is essential for the formation of large area films with reasonable roughness. For instance, the fabrication

of paper-based devices [23], which show great potentials as flexible, low-cost, portable, and environment-friendly alternatives, is limited by the unsuitable wettability and high surface roughness of the paper substrates. Without surface modification, it is difficult to directly deposit high quality thin films on paper [24].

The formation of thin organic films on the surface of the liquid has been known since the 18th century when Franklin observed oil filled the surface of a pool [25]. If we can utilize similar strategies of interfacial assembly to assemble organic semiconductors, some of the above challenges may be readily solved. Compared to the solid substrates, water acts more like a “free substrate” with good heat conductivity and no pinning effect. These properties are beneficial for the preparation of large-area films. In addition, the materials can be fully utilized in this process and it should be also very convenient to transfer the resultant thin films onto various substrates.

Here we showed the feasibility of such a strategy with several kinds of materials including Poly (3-hexylthiophene): [6, 6]-phenyl-C₆₁-

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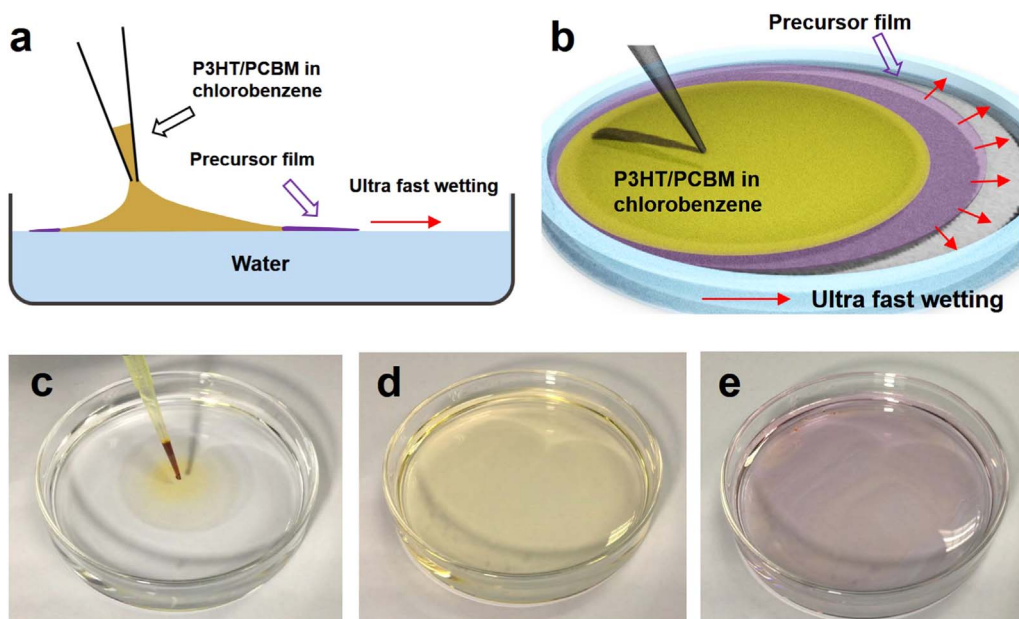


Fig. 1. Assembly process of organic P3HT:PC₆₁BM film on water. (a, b) Schematic illustration of dropping, spreading of the precursor solution and the self-supported P3HT:PC₆₁BM film on water; (c-e) corresponding photo images (diameter of the dish is 10 cm).

butyric acid methyl ester (P3HT:PC₆₁BM) film, which has been widely used to build bulk heterojunction (BHJ) organic solar cells (OSCs) [26,27]. P3HT:PC₆₁BM films of sub-square-meter scale were fabricated with precise control over the thickness and readily transferred to various kinds of substrates. The full utilization of materials results in an extremely high power-per-weight efficiency which is nearly one order of magnitude higher than previous reports. With these advantages, our method shows great potentials in industrial applications of organic semiconductors in solar cells and other fields.

2. Experimental section

2.1. Materials

P3HT (Poly (3-hexylthiophene)), PCBM ([6, 6]-phenyl-C61-butyric acid methyl ester) and chlorobenzene were purchased from Aladdin Company (Shanghai, China). The precursor solution was obtained by dissolving P3HT:PC₆₁BM into chlorobenzene with different concentration ranging from 20 mg/ml to 5 mg/ml, respectively. J61, *m*-ITIC, PBDB-T, PCE-10 and PC₇₁BM were purchased from Solarmer Materials (Beijing) Inc. The concentration of chlorobenzene precursor solution of J61:*m*-ITIC, PBDB-T:PC₇₁BM and PCE-10:PC₇₁BM was 20 mg/ml.

2.2. Fabrication of organic solar cells

The precursor of ZnO was obtained by dissolving 1 g zinc acetate dihydrate (Zn(CH₃COO)₂·2H₂O, Aladdin Company) and 0.28 g ethanolamine (H₂NCH₂CH₂OH, Aladdin Company) into 10 ml 2-methoxyethanol (CH₃OCH₂CH₂OH, Aladdin Company) under stirring for 12 h for the hydrolysis reaction according to the procedures reported previously [28]. The organic solar cells were fabricated with a structure of ITO/ZnO/active layer/MoO₃/Al. The ZnO layer was obtained by spin-coating the ZnO precursor solution onto the ITO-glass substrate and annealing at 200 °C for 2 h in the air. The thickness of ZnO film is approximately 30 nm. The precursor solutions were dropped on the surface of the water and resulted in floating photoactive layer. Then the photoactive layer was transferred to ZnO coated substrate and heated on the hot plate under 50 °C for 30 min with a thickness of 140 nm. Then the substrates were annealed (150 °C 2 min for PC₆₁BM:P3HT and J61:*m*-ITIC, 100 °C 2 min for PBDB-T:PC₇₁BM and PCE-10:PC₇₁BM). 5 nm of MoO₃ was vacuum-evaporated onto the photoactive layers and then 80 nm of Al was evaporated onto the MoO₃ as the anode. The

working area of the organic solar cells is 0.047 cm².

2.3. Preparation of the paper based photodetectors

A graphite-pencil (6B) was guided by a ruler to draw interdigitated comb-shaped electrodes with the space of ~ 2 mm on filter paper. The PC₆₁BM/P3HT organic film was directly scooped up by the pre-drawn filter paper from the surface of water. Then a thin PDMS film was coated on the PCBM/P3HT immediately. The substrate was then baked on a hot plate at 80 °C for 20 min.

2.4. Characterization

A Dimension Icon SPM (Bruker, Santa Barbara, CA, USA) was used to perform the AFM topographic measurements of the P3HT:PC₆₁BM films. A cold field emission scanning microscope (Hitachi S4800, operated at 2.0 kV, 10 μA) was employed to observe the morphologies of pencil-trace. The photoelectric efficiency of all polymer solar cells was measured on a computer-controlled Keithley 2450 Source-Measure Unit. Oriel Sol3A Class AAA Solar Simulator (model, Newport 94023 A) with a 450 W xenon lamp and an air mass (AM) 1.5 filter was used as the light source. The light intensity was calibrated to 100 mW cm⁻² by a Newport Oriel 91150 V reference cell. The photo response performance of the devices was studied at ambient condition using a tungsten lamp with the power density ranging from 0 to 10 mW cm⁻² and a sourcemeter (2400, Keithley) was used for electrical characterization during the measurements.

3. Results and discussions

3.1. Preparation and characterization

Fig. 1 illustrates the process to prepare P3HT:PC₆₁BM films. The chlorobenzene solution of P3HT and PC₆₁BM was dropped onto the water surface. When contacting the water surface, the solution spread and immediately filled the whole surface of water within 1–2 s. Interestingly, during the spreading of the solution, we observed the thin liquid precursor film near the contact line of the water-oil-air interface (Fig. 1c). This ultra-fast precursor film guided the spreading of solution and formed a thin liquid film of the chlorobenzene solution of P3HT and PC₆₁BM on the surface of water (Fig. 1d). The formation of precursor liquid films on solid substrates was discovered about a century

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