



Polymer solar cells spray coated with non-halogenated solvents



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ABSTRACT

Using spray-coating technique, we successfully fabricated conventional ITO-based and inverted ITO-free polymer solar cells (PSCs) based on a conjugated polymer poly[2,3-bis-(3-octyloxyphenyl) quinoxaline-5,8-diyl-alt-thiophene-2,5-diyl] (TQ1) as the donor and [6,6]-phenyl-C₆₁-butyric acid methyl ester (PC₆₁BM) or [6,6]-phenyl-C₇₁-butyric acid methyl ester (PC₇₁BM) as the acceptor. Environment-friendly non-halogenated solvents were used to process the active layers. The influence of substrate temperatures and processing solvents on the photovoltaic performance of the ITO-based TQ1:PC₆₁BM PSCs was systemically investigated. A higher substrate temperature can accelerate the solvent evaporating rate and afford a micro-textured rougher surface, which efficiently reduced light reflectance and enhanced absorption. Furthermore, finer phase separation was observed when using this high substrate temperature, which led to enhanced photocurrent due to the reduced bimolecular recombination. The device performance of spray-processed PSCs using the non-halogenated solvent mixtures was comparable to that of spray-processed PSCs using the halogenated *o*-dichlorobenzene (*o*DCB), which demonstrates that the non-halogenated solvents are very promising in spray-processed PSCs. This work sheds new light on developing efficient roll-to-roll compatible spray-coated PSCs with environment-friendly solvents.

1. Introduction

In the past decade, polymer solar cells (PSCs) have attracted considerable attention due to their unique advantages of low cost, light weight, environmental benignity and flexibility through roll-to-roll (R2R) manufacturing [1–4]. Up to now, the power conversion efficiency (PCE) of above 11% has been achieved both for single junction [5,6] and tandem PSCs [7]. Such impressive performance promotes bulk heterojunction (BHJ) PSCs to be one of the most feasible and renewable energy techniques in future. As one of the widely used solution-processing technique, spin-coating is not suitable for the productive R2R fabrication of large-scale devices [2,8]. Towards large-scale module production, several new solution-processing techniques such as inkjet-coating [9], doctor blading [10] and screen printing [11], have gained growing interests now. Among these techniques, spray-coating [12,13] has recently been considered as a promising method with particular advantages such as less material

loss, high production speed and various substrate compatibility, which has been successfully used to fabricate large-area PSCs [14,15].

Fabrication of large-scale PSCs involves the use of halogenated organic solvents, which has been banned worldwide due to the environmental and health hazards. Moreover, the use of halogenated solvents largely increases the overall production costs due to the specific requirement of personal protection and disposal of hazardous waste. Therefore, it is necessary to replace the toxic halogenated solvents with non-halogenated alternatives. However, halogenated solvents such as chloroform (CF), chlorobenzene (CB) and *o*-dichlorobenzene (*o*DCB), usually afford higher device performance in PSCs. Recent study reveals that some of non-halogenated solvents such as toluene (TL), xylene and trimethylbenzene can attain comparable device performance. The key limiting factor is the poor solubility of fullerene derivatives in non-halogenated solvents. This leads to large fullerene aggregates and large-scale phase segregation in the blend films, which significantly limits the device performance [16–18].

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Therefore, it is essential to improve the solubility of fullerene in non-halogenated solvents. One facile method is using binary or ternary solvent mixtures to modulate the solubility of fullerene and polymer to achieve favorable donor/acceptor morphology [19,20]. For example, Park *et al.* used acetophenone and mesitylene mixtures instead of *o*DCB to fabricate spin-coated PSCs [21]. Chen *et al.* reported high-performance spin-coated PSCs using TL as the processing solvent and 2% 1-methylnaphthalene as the additive [20] or with various xylene mixtures as the processing solvents [22].

Using non-halogenated solvents, great progress has been achieved in spin-coating processed PSCs. However, the correlation between processing methods and device performance is not clear for spray-coating PSCs using non-halogenated solvents. Especially, the drying kinetics and charge decay dynamic of spray-processed blend films have not been systematically studied. In this work, we used an easily synthesized polymer poly[2,3-bis-(3-octyloxyphenyl)quinoxaline-5,8-diyl-*alt*-thiophene-2,5-diyl] (TQ1) as the donor, which afforded good efficiency of 6–7% in spin-coated PSCs with [6,6]-phenyl-C₇₁-butyric acid methyl ester (PC₇₁BM) as the acceptor [23,24]. Although spin-coated TQ1:[6,6]-phenyl-C₆₁-butyric acid methyl ester (PC₆₁BM) devices show low PCEs of 4–5% due to the weak PC₆₁BM absorption-coefficient in visible region, the blends solution with PC₆₁BM shows the more controllable surface tension and viscosity than with PC₇₁BM [10,24], which are very important to inhibit the nozzle orifice blocking in production of large-area PSCs. Both spray-coating and spin-coating PSCs were fabricated using halogenated and non-halogenated solvents for comparison. The correlations between the drying kinetics and the photovoltaic parameters were investigated using different temperatures and processing solvents. It was noted that the TQ1:PC₆₁BM PSCs spray-coated from the non-halogenated solvent mixture of TL: indane (ID) afforded analogous device performance compared to those using the halogenated *o*DCB (3.3% vs. 3.6%), which was also close to the PSCs using the spin-coating method (3.7%). The role of ID additive in TL is that it has a high boiling point and low vapor pressure with a good solubility of acceptors in conjunction with the non-halogenated property. It indicates that the non-halogenated solvent mixtures can replace the toxic *o*DCB in high-throughput spray-coating processed PSCs. In addition, since the production cost is another issue for commercialization of PSCs, we also study the spray-coated ITO-free inverted solar cells (IFISCs) using the solution-processed PEDOT:PSS as the anode and Al/TiO_x as the cathode, which is considered to be more useful for the R2R processing. The spray-coated and spin-coated IFISCs show comparable device performance. Using a higher substrate temperature in the spray-coated IFISCs, superior short-circuit current density (J_{sc}) of 7.30 mA/cm² and 8.27 mA/cm² was obtained in the TQ1:PC₆₁BM and TQ1:PC₇₁BM devices, respectively. Our approach presents a promising method to develop low-cost, large-scale environment-friendly PSCs using the spray-coating technique.

2. Experimental details

2.1. Materials

The molecular structures of TQ1, PC₆₁BM and PC₇₁BM are shown in Fig. 1a. PC₆₁BM and PC₇₁BM were purchased from Solenne. The TQ1 material was synthesized in our lab with the number-average molecular weight (M_n) of 71.0 kDa and a polydispersity index (PDI) of 3.7 [24]. All reagents and metals were purchased from Alfa, Dupont, GCRF, and used without further purification. The weight ratio of TQ1:PC₆₁BM and TQ1:PC₇₁BM used in this work is 1:2.5 (w/w). The non-halogenated TL:ID solvent mixtures and the halogenated *o*DCB are used as the processing solvent with the concentrations of 20 mg/mL and 40 mg/mL, respectively. For the non-halogenated TL:ID solvent mixtures, the active solutions were firstly prepared in the pure TL solvent and pure ID solvent respectively, and then the pure solutions based on the volume ratios were mixed. The boiling points

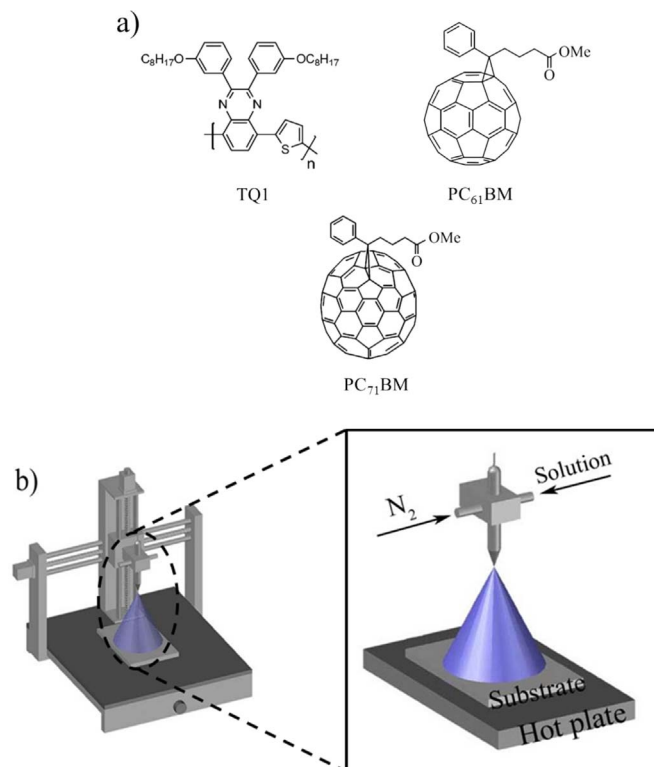


Fig. 1. (a) The molecular structures of TQ1, PC₆₁BM and PC₇₁BM. (b) Setup of spray coating system with a zoom illustration.

of solvents were measured by Thiele tube. The vapor pressures of pure and mixed solvents were calculated by Antoine equation and Raoult's law (see details in [Supplementary information \(SI\)](#)). The thicknesses of the spray-coated films are controlled by changing the fabrication parameters, including the solution concentration and the spray-coating cycles [12,13,25].

2.2. Film characterization

Contact angles using water and formamide as the testing liquids were determined on solid active films prepared from different processing conditions using a drop shape analyzer DSA100 instrument (KRÜSS GmbH). These contact angles were converted into surface energy values based on the Owens method (see details in SI). To monitor the transient spray-coated wet film drying process, the film specular reflection excited by a blue LED was recorded by a CMOS camera (IDS uEye). The transient drying process of spin-coated wet films was achieved by synchronizing the camera with a near-infrared sensor, which was connected to a spin-coater. The thickness of final dry film was measured by a surface profilometer (XP-2). The film topography was investigated using atomic force microscope (AFM) (CSPM 5500), transmission electron microscopy (TEM) (JEM-2100F) as well as scanning electron microscopy (SEM) (ULTRA55, Zeiss). UV–vis absorption spectra were recorded on a Shimadzu UV-2550 UV–vis spectrophotometer.

2.3. Spraying process

The working principle of spray coating apparatus is described in Fig. 1b, which consists of four parts: The first part is the injection unit, which is composed of the 0.3 mm diameter nozzle orifice (MA-S), active solution and high-pressure nitrogen gas (N₂) for atomizing the solution. The second part is the heating unit to control the morphology of the spray-processed film. The third part is the moving unit, which

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