

# Fabrication of polymer solar cells from organic nanoparticle dispersions by doctor blading or ink-jet printing

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## ABSTRACT

Dispersions of surfactant-free organic nanoparticles have proven a viable route to an eco-friendly lab-scale deposition of bulk-heterojunctions for polymer solar cells, matching the performance of solar cells deposited from toxic chlorinated solvents. In this work, we demonstrate the feasibility of doctor blading for the deposition of photo-active layers from organic nanoparticles comprising poly(3-hexylthiophene) and indene C<sub>60</sub>-bisadduct. The devices yielded power conversion efficiencies of 3.9% on 0.105 cm<sup>2</sup> photo-active area and 3.4% on 1.1 cm<sup>2</sup>, being close to the performance of their spin cast counterparts. Likewise, the photo-active layers can be ink-jet printed yielding overall power conversion efficiencies of 2.9%, altogether rendering surfactant-free organic nanoparticles in eco-friendly dispersion agents a valuable process route to a sustainable large-area fabrication of organic solar cells.

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## 1. Introduction

Polymer bulk-heterojunction solar cells are steadily advancing towards market-readiness. With printing and coating being widely discussed for potential low-cost fabrication [1–4], the choice of processing solvents has been identified as one of the main challenges to transfer lab-processes to an industrially viable production environment [5–8]. Although the use of chlorinated solvents in the lab yields high power conversion efficiencies (PCEs) beyond 11% [9–11], non-chlorinated and eco-friendly processing agents are mandatory prerequisites for large-area processing. Recently, dispersions of poly(3-hexylthiophene):indene-C<sub>60</sub> bisadduct (P3HT:IC [60]BA) nanoparticles in eco-friendly aqueous or alcoholic dispersion and their subsequent incorporation in polymer solar cells have attracted a lot of attention. In the literature, two approaches to the synthesis of polymer-fullerene nanoparticle dispersions are followed: On the one hand, utilizing stabilizers, Landfester et al. reported on organic nanoparticles that were synthesized using a mini-emulsion method [12]. Incorporating such nanoparticles into organic solar cells yielded maximum PCEs of 2.5% [13], most

likely due to the stabilizers that remain within the photo-active layer, hampering the efficient conversion of solar energy. Employing slot-die coating, Yamamoto et al. achieved PCEs of 2.6% utilizing a narrow-band gap light-absorbing polymer [14]. On the other hand, Gärtner et al. demonstrated 4% efficient polymer solar cells, fabricated from stabilizer-free nanoparticle dispersions that were synthesized by precipitation in alcohol [15]. Although the PCEs of these “green” solar cells fabricated from non-stabilized dispersions are very encouraging, the process transfer from lab-scale spin coating to industrially relevant and scalable deposition processes yet remains to be demonstrated.

Doctor blading, its advancement slot-die coating and ink-jet printing are common deposition techniques that are compatible with roll-to-roll processing. Using chlorinated solvents or high boiling aromatic solvents, a lot of prior work has been done in fabricating P3HT:fullerene solar cells by slot-die coating [16], doctor blading [17,18] or ink-jet printing [19,20] yielding maximum PCEs of 3%–4%.

In this work we transfer the literature-known concept of polymer solar cell fabrication from stabilizer-free organic nanoparticle dispersions to scalable deposition processes such as doctor blading and ink-jet printing and demonstrate the feasibility of the nanoparticle concept for future large-area printing.

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## 2. Experimental details

### 2.1. Nanoparticle synthesis

P3HT:IC[60]BA (P3HT: Rieke Metals,  $M_w = 57,000$  g/mol,  $D_M = 2.4$ ,  $RR = 91\%$ ; IC[60]BA: Luminescence Technology Corp., Purity >99%) nanoparticle dispersions were synthesized by precipitation in ethanol (blend ratio 1:1, 10 mg/mL) as described in our previous work [15]. The nanoparticles had diameters of 100 nm and exhibited a dispersity of 0.05 according to dynamic light scattering measurements. The dispersions were stable for at least three weeks which allowed further processing without filtering.

### 2.2. Spin coating

Polymer solar cells were fabricated according to the inverted device architectures depicted in Fig. 1a. Therefore, patterned indium tin oxide (ITO,  $R_{\square} = 13 \Omega/\square$ , 125 nm) coated glass substrates were cleaned by successive ultra-sonication in acetone and isopropanol (10 min) before exposing them to oxygen plasma to remove organic residues from the ITO surface (2 min). Then the samples were transferred to a glovebox and kept under nitrogen atmosphere for the entire fabrication and characterization process. The electron transport layer (30 nm) from zinc oxide nanoparticles (ZnO, Nanograde Ltd., N-10, 1 wt% in isopropanol) was spin cast from dispersion (4000 rpm, 30 s) and then thermally annealed (150 °C, 5 min). The nanoparticulate P3HT:IC[60]BA layer with a total thickness of 170 nm was applied by spin coating the dispersion three times in 20 s intervals on a rotating substrate (1000 rpm). This multi-pass application of the active layer is necessary to compensate the moderate nanoparticle concentration of 10 mg/mL. Henceforth, we distinguish *layers* and *sublayers*, with the sublayers resulting from one deposition step and the layers comprising several sublayers of the same material. The P3HT:IC[60]BA photo-active layer (200 nm) of the reference device was spin cast (800 rpm, 30 s) from *o*-dichlorobenzene solution (*o*-DCB, 40 mg/mL) and then solvent annealed for about 1 h. Both active layers were thermally annealed (150 °C, 10 min). The PEDOT:PSS hole transport layer (HTL Solar, Heraeus Holding GmbH, water-diluted 1:1 v/v, 35 nm) was spin cast (500 rpm, 5 s; 2000 rpm, 30 s) and then thermally annealed (120 °C, 10 min). A 100 nm silver electrode was thermally evaporated in high vacuum ( $10^{-6}$  mbar). The cross sections of the silver and the ITO electrodes defined active areas of 0.105 cm<sup>2</sup> per solar cell.

### 2.3. Doctor blading

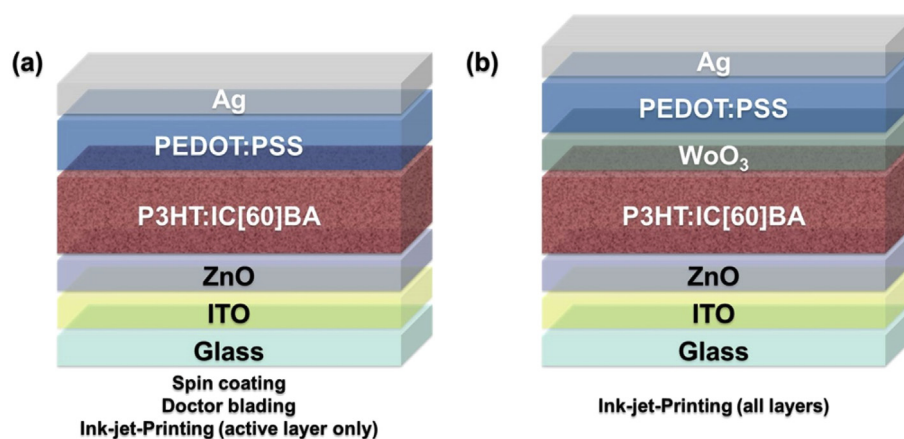
All layers between the ITO and silver electrodes were applied from dispersion on an automated film applicator (ZAA 2300, Zehntner Testing Instruments) under inert conditions, yielding the same layer thicknesses described above. Devices were fabricated with areas of 0.105 cm<sup>2</sup> or 1.1 cm<sup>2</sup>. Zinc oxide nanoparticles were doctor bladed using an Universal Applicator (ZUA 2000, Zehntner Testing Instruments, velocity 18 mm/s, gap height 150  $\mu$ m, platen temperature 35 °C, volume 30  $\mu$ L or 50  $\mu$ L) and annealed on a hotplate (80 °C, 10 min). Subsequently, two sublayers of P3HT:IC[60]BA nanoparticles were applied from ethanol dispersion using a cylindrical applicator (diameter 10 mm, velocity 90 mm/s, gap height 400  $\mu$ m, platen temperature 40 °C, volume 25  $\mu$ L or 40  $\mu$ L). Afterwards, the layer was thermally annealed (150 °C, 10 min) in order to join the nanoparticles. The PEDOT:PSS hole transport layer was applied using the same cylindrical applicator (velocity 20 mm/s, gap height 400  $\mu$ m, platen temperature 60 °C, volume 55  $\mu$ L or 90  $\mu$ L). After another thermal annealing step (120 °C, 10 min), the silver electrode was deposited as described above.

### 2.4. Ink-jet printing (active layer only)

The electron transport layer of ZnO was spin cast as described above under nitrogen atmosphere. The P3HT:IC[60]BA nanoparticle dispersion was diluted with 2-butoxyethanol (3:1 v/v) yielding an overall solid content of 7.7 mg/mL. The dispersion was printed using a 16-nozzle piezo print head (Dimatix DMC 11610) mounted on a Dimatix platform (Dimatix DMP 2831) in air. The platen temperature was set to 40 °C. 8 sublayers were printed using a drop spacing of 65  $\mu$ m and, subsequently, another 8 sublayers were printed with a drop spacing of 35  $\mu$ m. The substrate was rotated by 90° after deposition of each 4 sublayers. After transferring the samples into a nitrogen glovebox, they were annealed (150 °C, 10 min). The hole transport layer was spin cast and the top electrode was evaporated as described above.

### 2.5. Ink-jet printing (all layers)

All layers between the bottom ITO and top silver electrode were ink-jet printed in air. Two sublayers of ZnO nanoparticles were printed using a drop spacing of 50  $\mu$ m. Then the samples were annealed under nitrogen atmosphere (150 °C, 5 min). The active



**Fig. 1.** (a) Architecture of spin coated, doctor bladed and ink-jet printed (active layer only) devices. (b) When ink-jet printing all functional layers between the electrodes, an additional WO<sub>3</sub> layer is introduced to facilitate PEDOT:PSS wetting on the active layer.

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