Investigation of various commercial PEDOT:PSS (poly(3,4-ethylenedioxythiophene)polystyrene sulfonate) as a hole transport layer in lead iodide based inverted planar perovskite solar cells

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Abstract

Inverted-type perovskite solar cells have drawn remarkable attention due to solution-processable, straightforward configuration, low-cost processing, and manufacturing at very high throughput, even on top of flexible materials. The hole transport material (HTM) plays a vital role to achieve high performance in inverted type of perovskite solar cells. Herein, we report on the effect of different commercial PEDOT: PSS such as PH 1000, PH 500, P VP AI, and P T2, on the performance of CH\textsubscript{3}NH\textsubscript{3}PbI\textsubscript{3} based planar perovskite solar cells.

Keywords: PEDOT: PSS, hole transport layer, CH\textsubscript{3}NH\textsubscript{3}PbI\textsubscript{3}, inverted perovskite solar cells

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1. Introduction

As a result of expanding population and industrial growth, worldwide energy utilization has escalated, then demands for renewable and viable energy sources became inevitable. Alternative resources should keep a balancing act between efficiency, expenditure, technology issues, and environmental effects. Using photovoltaic devices for gathering energy from sunlight offers a fresh perspective for producing energy globally. Due to high cost, high technology requirements such as high vacuum and temperature for the fabrication, and additionally the existence of toxic components, the use of first and second generation of solar cells is limited. Hence, the new third-generation of PVs with low-cost, high flexibility, low molecular weight, and high efficiency are demanded. For a decade perovskite-based solar cells have attracted a great deal of attention because of its superb characteristics such as low-cost, long carrier diffusion length, high absorption coefficient, low-temperature processing, low recombination rate[1–3], high electron and hole mobility, tunable bandgap[4], and a rapid increase in the efficiency from 3.9%[5] to more than 25%[6] which is not common for a photovoltaic device to show extremely rapid development in such a short period. Perovskite solar cell fabrication can be classified into two; evaporation based methods[7,8], and solution-based methods. Solution-based methods have three different approaches including the one-step deposition method[9,10], spray coating[11], two-step deposition method[12]. Evaporation based techniques guarantee a pin-hole free perovskite growth with a moderate uniformity of grains, but these processes require high vacuum technology. One-step deposition includes two forms, one with solvent annealing[13,14], and one with antisolvent washing treatment[15]. The latter method is usually carried out, where during the substrate is spinning a selected antisolvent such as toluene or chlorobenzene is dropped on the perovskite precursor layer and end with
thermal annealing. This method produces a conformal and dense perovskite layer, but the grain size is relatively small (around 200 nm). On the other hand, in terms of device architecture, perovskite solar cells can be classified as mesoporous and planar types. In the mesoporous type, the device requires high temperature (more than 450 °C) through manufacturing progress which leads to increasing fabrication cost[16], while in planar structure the processing temperature is low. Due to the analogous efficiency of planar to mesoporous, employing planar device structure in PSCs is feasible. Different perovskite solar device architectures are shown in Fig. 1[17]. In n-i-p (normal) structure, TiO₂ layer is used as an electron transport layer (ETL) and spiro-OMeTAD (2,2,7,7-tetrakis(N,N-di-p-methoxyphenylamine)-9,9-spirobiﬂuorene) as HTL, while in the p-i-n (inverted) structure commonly PEDOT:PSS is using as a hole transport layer (HTL) and [6,6]-Phenyl-C61-Butyric Acid Methyl Ester (PCBM) as ETL which need a buffer layer for reduction of interfacial charge recombination such as BCP[18–21], LiF[22,23]. Applying high temperature for the sintering TiO₂ layer is inevitable that increases the
manufacturing costs and trapped charges at the interface between the perovskite and TiO$_2$ layer[16]. Moreover, spiro-OMeTAD has low stability and high cost[24]. Alternatively, by using PEDOT:PSS in p-i-n device structure these problems can be eliminated. Both organic and inorganic materials can be employed as ETL and HTL by evolving from sensitized meso-structure to planar structure[25].

Generally, PEDOT: PSS is considered as an HTL in inverted-type perovskite solar cells due to high conductivity and transparency, low-temperature solution-processable HTL, and suitable for the fabrication of flexible devices[26,27]. Despite these advantages, PEDOT: PSS has some disadvantages such as hygroscopic and acidic nature, mismatching between work function energy level of PEDOT: PSS and HOMO energy level of perovskite which lead to less $V_{oc}$[28–30], consequently, unfavourable effects on the performance of PSCs. Various approaches applied for modifying these properties such as solvent additive methods[31,32], UV treatment[33,34], doped-PEDOT: PSS[35,36], interfacial engineering[37][38].

![Fig. 2](image_url) (a) schematic configuration and (b) energy diagram of device

In this work the performance of inverted perovskite solar cells, with different commercial PEDOT:PSS precursors was studied in a configuration of
ITO/PEDOT:PSS/CH₃NH₃PbI₃/PCBM/BCP/Ag, where all the experiments were performed in ambient air under humidity 40-50% (Fig. 2).

2. Experimental

PCBM ([6, 6]-phenyl-C61-butyric acid methyl ester, Solenne, 99%), lead iodide (PbI₂, Sigma-Aldrich 99.0%), BCP (2,9-dimethyl-4,7-diphenyl-1,10-phenanthroline, 96%, Sigma), different commercial PEDOT:PSS precursors (Poly(3,4-ethylenedioxythiophene)-poly(styrenesulfonate) {PH 1000 (Clevios), PH 500 (Clevios), P T2 (Clevios), P VP AI (Clevios)}) were purchased and used without any further purification. Methylammonium iodide (CH₃NH₃I (MAI)) was synthesized according to reported method [39]. ITO-coated glass substrates with a surface resistivity of 35-45 Ω/sq, were purchased from Labkon. Perovskite precursor solution was prepared by co-dissolution of PbI₂ and MAI with 1.4:1.4 molar ratio in γ-butyrolactone (GBL). This solution was stirred at 60 °C for at least 12 hours and double filtered with 0.45 μm PVDF and 0.22 PTFE μm filters prior to the deposition. PCBM solution was prepared by dissolving 20 mg of the PCBM powder in 1 mL chlorobenzene and stirred at room temperature for a few hours. BCP precursor was used prepared by solving 0.5 mg BCP powder in absolute ethanol. All the commercial PEDOT:PSS precursors; including PH 1000, PH 500, P T2, P VP AI were filtered with a 0.45 μm PTFE filter before coating.

The fabrication of the device with the structure ITO/PEDOT:PSS/CH₃NH₃PbI₃/PCBM/BCP/Ag was as follow: Indium-tin-oxide (ITO) coated glasses were cut into small slides of 1.5 × 1.5 cm². The etching process was used to pattern the ITO-coated glasses by applying a mixture of HCl:HNO₃:H₂O (4.6:0.4:5) for 45 min. The etched substrates were cleaned in an ultrasonic bath, before fabricating PEDOT:PSS layers. The patterned ITO substrates were wiped by toluene, ethanol, acetone, and thereafter, the substrates were
sonicated separately in acetone (for 20 minutes) and isopropanol (for 45 minutes at 60 °C). Finally, the substrates were dried by N₂ gas. Firstly, all types of used PEDOT:PSS was spin-coated on the ITO coated glass substrate at 2000 rpm for 50 seconds and then thermally annealed on a hot-plate at 140 °C for 10 minutes. Perovskite layer was formed on PEDOT:PSS layer by spin coating the perovskite solution at 2000 rpm for 10 seconds and 4000 rpm for 20 seconds. In order to get high and fast crystallinity, toluene (60 μl) was dropped onto spinning substrates for anti-solvent washing when the rotation speed reached 4000 rpm. Perovskite growth was completed in ambient air with heat treatment on a hot-plate at 100 °C for 20 minutes. Subsequently, PCBM film was formed by coating it at 1500 rpm for 30 seconds and dried at 90 °C for 2 minutes, afterward BCP layer was spin-coated at 4000 rpm for 40 seconds. Ag is used as the top electrode and deposited by the thermal evaporation with a thickness of 110 nm (Fig. 3).

3. Characterization

UV - vis spectroscopy was used to investigate the bandgap of the perovskite layer with a PG Instruments T80 spectrophotometer. Atomic force microscope (AFM) was performed using Hitachi 5100N model device for interpreting surface morphologies of the PEDOT:PSS layers. Scanning electron microscopy (SEM) was used to investigate the surface morphologies of perovskite layers onto different PEDOT:PSS layers with a FEI Versa 3D field emission/low vacuum scanning electron microscope. Current density-voltage (J-V) curves of solar cell devices were obtained by a Keithley 2400 source meter with a scan rate of 0.25 V/s and 0.03 s delay time, under the illumination of 100 mW/cm² to simulate the condition of AM 1.5G which was calibrated using an ABET solar simulator.
Incident photon to current efficiency (IPCE) measurements were conducted with New Port measurement system, including an optical system with a xenon lamp, a filter wheel, a mechanical chopper, and a monochromator (Acton SP150) and recorded by using a lock-in amplifier. The open-circuit voltage ($V_{OC}$), short-circuit current density ($J_{SC}$), fill factor (FF) were determined from J-V curves. Fill factor is given by the following formula:

$$ FF = \frac{V_{mpp} \times J_{mpp}}{V_{oc} \times J_{sc}} $$

(1)

And the power conversion efficiency (PCE) of device is determined by the ratio of power-out to power-in as following formula:

$$ PCE(\%) = \left(\frac{P_{out}}{P_{in}}\right) \times 100 = \left(\frac{FF \times V_{oc} \times J_{sc}}{P_{in}}\right) \times 100 \) 

(2)

Theoretically the maximum short-circuit current density was calculated by using IPCE as following[40]:

$$ J_{SC} = \int qF(\lambda)IPCE(\lambda)d\lambda $$

(3)
Where “λ” is wavelength of incident light, “q” is electron charge, and “F(λ)” presents incident photon flux density.

4. Results and discussion

Fig. 4 depicts the AFM images for PH 1000, PH 500, P VP AI, and P T2 based PEDOT:PSS films deposited on to ITO coated glass substrates in two different area of 2 × 2 µm² and 10 × 10 µm².

Fig. 4 AFM images of PH 1000, PH 500, P T2 and P VP AI, PEDOT:PSS layers deposited on to ITO coated glass substrates
The mean roughness ($S_a$) values of PH 1000, PH 500, P T2, and P VP AI were obtained as 1.122 nm, 1.101 nm, 1.170 nm, and 1.407 nm, respectively for $2 \times 2 \mu$m$^2$. While, for $10 \times 10 \mu$m$^2$ area the mean roughness ($S_a$) values of PH 1000, PH 500, P T2, and P VP AI were obtained as 2.504 nm, 2.300 nm, 2.644 nm, and 1.122 nm, respectively. This shows P VP AI layer is more uniform and homogeneous at large area. Although the difference between the roughness of different PEDOT:PSS layers are not noticeably significant, the surface of P VP AI film is smoother compared to others. Since a more uniform and smoother film is favourable for charge transport enhancement, P VP AI employed devices expected to lead better photovoltaic performance.

Furthermore, the effect of the surface morphology of PEDOT:PSS on the perovskite growth quality was investigated by scanning electron microscopy (Fig. 5). SEM images reveal that perovskite layer growth on the P VP AI was successfully achieved with larger grain sizes and homogeneous grain distribution without any pin-holes. Increasing grain size and reducing grain boundaries in the perovskite film is known to be advantageous for high short-circuit current and fill factor values. On the other hand, in the case of PH 500 the perovskite film suffers from small grain size which consequently lead to a reduction in the performance of the device. The perovskite layer on PH 1000 presents a uniform and homogeneous film as P T2 layer. However, the grain sizes of the formed perovskite film obviously smaller compared to P VP AI employed samples which is directly linked to the lower $J_{sc}$ values of the PH 1000 employed PSCs. Insulating PSS is used as a dopant in conducting PEDOT for balancing counter ion[41]. Due to different electrical properties of PEDOT and PSS (Table 1.), different commercial PEDOT:PSS electrically differ from each other which leads to a variation in conductivity and wettability of the PEDOT:PSS films.
Table 1. Properties of PEDOT:PSS precursors [42–45]

| PEDOT:PSS       | PH 1000 | PH 500 | P T2      | P VP AI  |
|-----------------|---------|--------|-----------|----------|
| Density (g/cc)  | 1       | 1      | -         | 1        |
| Boiling point (°C) | 100    | 100    | 100       | 100      |
| Viscosity (cP)  | 15-50   | 8-25   | 100-200   | 5-12     |
| pH              | 1.5-2.5 | 1.5-2.5| 1.5-2.5   | 1.2-2.2  |
| Particle size (µm) | > 0.030 | > 0.025 | > 0.040   | > 0.080  |
| Electrical resistivity (Ω·cm) | 0.00120 | 0.00330 | 0.00330-0.0100 | 500-5000 |
| Work function (eV) | 4.8-5.0 | -      | -         | 5.0-5.2  |
| PEDOT:PSS ratio (by weight) | 1:2.5   | 1:2.5  | 1:2.5     | 1:6      |

Fig. 5 SEM images of perovskite layers on top of PH 1000, PH 500, P T2, and P VP AI PEDOT:PSS layers
Fig. 6 (a) indicates the UV-Vis transmittance spectra (between 300-1100 nm) of different type of PEDOT: PSS which coated on ITO coated glasses as a substrate. Transmittance spectrum shows how many percentage of the incident light can pass through the sample without absorbing, reflecting, or scattering at different wavelengths.

Fig. 6 (a) UV-vis transmittance spectra for the PEDOT:PSS films onto ITO coated glass substrates (b) I-V curves of ITO/PEDOT:PSS/Ag (c) J-V curves of ITO/PEDOT:PSS/CH$_3$NH$_3$PbI$_3$/PCBM/BCP/Ag structures
As can be seen the transmittance of PH 1000 is lower while the PH 500, P T2 and P VP AI are similar. The higher transmittance, the more photons reaching to the active layer, which can increase the probability of charge generation. There is a clear transmittance increase around 400 nm for the PH 500 and P T2 and a slight rise with a different pattern, for the P VP AI. Additionally, transmittances slightly increased after 800 nm for P VP AI.

A surface with lower wettability minimizes the number of sites for perovskite nucleation which spans to the larger crystal growth[46]. The resistivity of the PEDOT:PSS precursors are known to be different, and also following the PEDOT:PSS film formation by spinning and annealing, the resulted layers may exhibit different resistances. To determine the electrical conductivity of different types of PEDOT:PSS films, current-voltage (I-V) was characterized across the ITO/PEDOT:PSS/Ag structure (Fig. 6 (b)). The current-voltage characteristics for the PEDOT:PSS layers display different slopes for each indicating various conductivities. The conductivity of the films was achieved by using the following formula[47],

$$\sigma = \frac{d}{AR}$$  \hspace{1cm} (4)

where \(\sigma\) is conductivity, \(d\) is the thickness of the film, \(A\) is active area, and \(R\) is resistance that can be extracted from current-voltage (I-V) curves. The conductivities of the different PEDOT:PSS layers were calculated to be \(13.6\times10^{-5} \text{ S.cm}^{-1}\), \(19.2\times10^{-5} \text{ S.cm}^{-1}\), \(20\times10^{-5} \text{ S.cm}^{-1}\), and \(24\times10^{-5} \text{ S.cm}^{-1}\), for P VP AI, P T2, PH 500, and PH 1000, respectively which is consistent with the literature[48]. Even though the high electrical conductivity of HTL is favourable for charge transport, higher \(J_{SC}\) in P VP AI employed PSCs can be attributed to the increased perovskite grain sizes and enhanced interface between perovskite layer and HTL which will provide an improvement in both FF and \(J_{SC}\) of the devices[49–51]. On the other hand the lower \(J_{SC}\) values of PH 500 and P T2 employed PSCs might originate from the pin-hole dominated
perovskite growth. Fig. 6 (c) shows current density-voltage (J-V) curves of devices consisting of different PEDOT:PSS layers. The device employed P VP Al PEDOT:PSS as a HTL demonstrated the best performance, while the device that PH 500 employed device exhibited the lowest performance. The photovoltaic parameters are summarized in Table 2.

Table 2. Photovoltaic parameters of fabricated devices

|        | Voc  (mV) | Jsc (mA cm⁻²) | FF  | PCE (%) | Rs (Ω cm²) | Rs (Ω cm²) |
|--------|----------|----------------|-----|---------|------------|------------|
| PH 1000 | 900      | 14             | 0.70| 8.8     | 5.05       | 7.39×10²   |
| P T2   | 875      | 16             | 0.55| 7.7     | 5.5        | 3.84×10²   |
| PH 500 | 900      | 13             | 0.44| 5.1     | 25.0       | 3.13×10²   |
| P VP Al| 925      | 19             | 0.64| 11.2    | 4.97       | 5.30×10²   |

The P VP Al PEDOT:PSS based PSC have an open-circuit voltage $V_{oc}$ of 925 mV, a short circuit current density $J_{sc}$ of 19 mA.cm⁻², and a fill factor FF of 0.64, resulting in a PCE of 11.2%. The highest $V_{oc}$ and FF values of P VP Al employed PSC can be explained with higher work function of P VP Al and enhanced interface properties concluded from SEM and AFM measurements. For PH 500 based PSC, $J_{sc}$ value is decreased to 13 mA.cm⁻² and open voltage is found to be $V_{oc}$ of 900 mV while the fill factor was obtained as 0.44. This degradation can be linked to SEM and AFM measurements, resulted in a PCE of 5.1%. In the case of P T2 based PEDOT:PSS employed PSC, $J_{sc}$ exhibits a slight decrease to the 16 mA.cm⁻², and similarly, the fill factor decreased to 0.55. This result also can be explained by SEM and conductivity measurements, compared to the P VP Al and PH 500 PEDOT:PSS employed PSCs. PH 1000 based PEDOT:PSS employed PSC exhibited a PCE of 8.8% with the highest fill factor of 0.70, while $J_{sc}$ decreases to 14 mA.cm⁻² which is lower than P VP Al and P T2 PEDOT:PSS employed devices. For PH 1000 PEDOT:PSS employed PSC, $V_{oc}$ is found to be similar to the PH 500 PEDOT:PSS employed devices as of 900 mV. Additionally, the series resistance and shunt resistance values are calculated and given in Table 2.
Since the series resistance is associated to charge carrier transport and contact resistance, the minimum $R_{\text{series}}$ value for P VP AI employed device is understandable. On the other hand, $R_{\text{series}}$ increased to 25.0 ohm cm$^2$ for PH 500 employed device and it has the highest value in comparison with others which leads to low photovoltaic performance, by decreased $J_{\text{SC}}$.

$J_{\text{SC}}$ values of the PH 500 based PEDOT:PSS HTL employed devices is related to the low shunt resistance with higher $I_0$ current values compared to the P VP AI employed PSC, rather than the resistive nature of the PEDOT:PSS layers. Dark J-V curves of the PSCs as seen in Fig. 7
also indicate the low Fig. 8 describes the box charts graphics for the photovoltaic parameters of different types of PEDOT:PSS layer employed solar cells over 15 devices, including $J_{sc}$, $V_{oc}$, FF, and PCE, which pointed out the photoelectric properties of the devices remained steady, and the devices indicate a good reproducibility.

Fig. 8 Histograms of photovoltaic parameters for solar cell devices with different type of PEDOT: PSS layers

Fig. 9 (a) shows the incident photon-to-current conversion efficiency (IPCE) spectra and integrated current density. The photocurrent onset at 800 nm was in a good agreement with the perovskite (CH$_3$NH$_3$PbI$_3$) bandgap. The devices employed P VP Al-based PEDOT:PSS layer as a HTL presented higher quantum yield compared to the others. The integrated photocurrent for P VP Al-based PSC was 18.5 mA.cm$^{-2}$. While the values for PH 500, PH 1000, and P T2 were observed as 12.7 mA.cm$^{-2}$, 13.8 mA.cm$^{-2}$, and 15.8 mA.cm$^{-2}$, respectively. The results acquired from IPCE spectra were slightly lower than the JSC values achieved from current
density-voltage measurements, which may have been affected by the test environment and equipment.

Fig. 9 (a) IPCE spectra and integrated photocurrent obtained from the corresponding IPCE spectra (b) normalized PCE of different type of PEDOT:PSS layer employed solar cell devices under ambient condition

Fig. 9 (b) presents the normalized power conversion energy (PCE) of different types of PEDOT:PSS employed un-encapsulated devices, under ambient air (humidity 40-50%, temperature ~25°C). It can be seen that devices employing P VP Al and PH 1000 showed better stability over a period of time as compared with others.

5. Conclusions
We investigated the photovoltaic performance of the methylammonium lead iodide based inverted perovskite solar cells based on different types of PEDOT:PSS HTLs including PH 1000, PH 500, P T2 and P VP AI precursors, which are commercially available. The device with P VP AI PEDOT:PSS HTL has exhibited the best photovoltaic performance of all, while the device using PH 500 presented the lowest photovoltaic performance as compared to other devices. The main reason refers to large grains dominated pin-hole free growth of the perovskite layer on the top of P VP AI layer due to smoother surface PVP AI layer. Also, it was observed that the device employing P VP AI and PH 1000 shows more resistance against degradation in the ambient environment over a short period of time.

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