A Dopant-Free Hole Transporting Layer for Efficient and Stable Planar Perovskite Solar Cells

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Herein, a new dopant-free organic material, PV2000, as a stable hole transporting layer (HTL) for the fabrication of stable and efficient perovskite solar cells (PSCs) is introduced. For this purpose, planar PSCs using a triple-A cation perovskite composition are fabricated and the commonly used 2,2',7,7'-tetrakis[N,N-di(4-methoxyphenyl)amino]-9,9'-spirobifluorene (spiro-OMeTAD) HTL is replaced by dopant-free PV2000 polymer. The characterization results disclose that the PV2000 has a great thermal stability, good hole mobility, and suitable band alignment that matches well with the valence band of triple-A cation perovskite. After proper optimization of PV2000 film thickness, a planar PSC with maximum power conversion efficiency (PCE) of 18.93% is achieved, which is comparable with the spiro-based device (19.62%). Moreover, the PCE of the PV2000-based device is further improved up to 20.5% using a band alignment engineering by deposition of thin layer of polyvinylpyrrolidone (PVP) at perovskite/HTL interface. More importantly, it is found that the thermal, moisture, and operational stabilities of the PSCs with PV2000 HTL are improved drastically compared with the spiro-based devices, where the PSC with PV2000 retains ≈88% of its initial PCE value under continuous illumination for 250 h compared with the spiro-based one (39%).

Organohalide lead perovskite solar cells (PSCs) make a revolution in the field of thin-film photovoltaics (PVs) due to their impressive record efficiency of 25.2%, low-cost and low-temperature processing, bandgap tunability, and ease of fabrication.[1–10] However, their long-term stability is still remained unsolved and needs to be addressed precisely to move a step toward commercialization.[11–15] The stability issue in a PSC can be originated from the perovskite absorber, electron transporting layer (ETL), hole transporting layer (HTL), and interfaces.[16–18] Over the past few years, many researchers focused on the perovskite layer and its interfaces by considering compositional engineering, additive engineering, interface engineering, and passivation techniques to improve the device stability.[19–28] In addition, a proper material selection for both ETL and HTL is an another effective strategy to improve the overall stability.[29] In case of ETL, there are several inorganic materials such as TiO2, ZnO, Zn2SnO4, and SnO2 exhibiting good thermal stability.[30–33] However, for the HTL case, there are not many stable materials and thus the development of new HTLs is required to effectively improve the stability of the PSCs.[34]

The 2,2',7,7'-tetrakis[N,N-di(4-methoxyphenyl)amino]-9,9'-spirobifluorene (spiro-OMeTAD) is an organic HTL which is commonly used in the normal architecture of the PSCs.[35] As reported in the literature, spiro HTL is a great choice to obtain high efficiency up to 24%[36] however, the stability of the device is still a big challenge mainly due to the presence of unstable dopants in the HTL and its organic nature.[37] To improve the stability of the spiro-based PSCs, additive engineering and double-layer HTL could be effective solutions.[38,39] For example, Sanehira et al.[40] improved the stability of the spiro-based PSCs by applying a thin layer of MoO3 at the interface of HTL/electrode. Previously, we also improved the stability of the PSCs by addition of adamantylammonium iodide to the spiro HTL solution, which leads to pinhole-free HTL morphology and better HTL/perovskite interface.[39] These approaches can slightly improve the stability of the PSCs but may not be an ideal approach to solve this issue. Therefore, the development of new HTL materials could be a better strategy to obtain stable devices.
especially at elevated temperatures. There are many reports in the literature on the use of organic and inorganic HTLs suitable for the fabrication of efficient PSCs. Some of them are expensive such as poly(bis(4-phenyl)(2,4,6-trimethylphenyl)amine) (PTAA), and few of them require high-temperature annealing such as nickel oxide (NiO) or have poor hole mobility, stability, and efficiency.

In this work, we report a new dopant-free HTL for the PSCs, i.e., PV2000, exhibiting great thermal stability and hole mobility. Our ultraviolet photoelectron spectroscopy (UPS) results reveal that the highest occupied molecular orbital (HOMO) level of PV2000 HTL is perfectly aligned with the valence band (VB) of the triple-A cation perovskite film and there is a strong quenching effect at the interface of PV2000/perovskite, better than that of the control spiro-based device. We achieve an efficient PSC using PV2000 HTL with power conversion efficiency (PCE) of 18.93% after optimization, fairly comparable with the efficiency of the spiro-based PSC. Using band alignment engineering with a thin layer of polyvinylpyrrolidone (PVP) at the HTL/perovskite interface, we improve this PCE up to 20.5%, higher than the reference PSC. Apart from the high efficiency, the devices with PV2000 show excellent moisture, thermal, and operational stabilities compared with the spiro-based devices. The PV2000-based PSC depicts only 12% PCE loss after 250 h under continuous illumination, much better than that of its spiro counterpart (61%).

PV2000 (Raynergy Tek) is a great alternative dopant-free HTL for the fabrication of efficient PSCs, due to its interesting optoelectronic properties. Figure 1a shows the thermogravimetric analysis (TGA) of PV2000 polymer. As seen, PV2000 has excellent thermal stability, where it starts to decompose over 400 °C and has a degradation temperature of 434 °C with 5% weight loss ($T_{10\%}$). Figure 1b shows the UV–vis and photoluminescence (PL) spectra of this polymer, indicating a bandgap of 1.67 eV. To study the electrical property of PV2000, we measured its hole mobility using space-charge-limited currents (SCLC) method by preparing a only hole-conducting device. Figure S1, Supporting Information, shows the current density–voltage ($J$–$V$) curve of the corresponding device, which is made of PV2000. We extracted hole mobility of $2.8 \times 10^{-4}$ cm$^2$ (V s)$^{-1}$ for PV2000, which is better than undoped spiro ($8.1 \times 10^{-5}$ cm$^2$ (V s)$^{-1}$) and state-of-the-art polymers reported in the literature. Therefore, PV2000 can act as an efficient HTL without any doping. To study the charge transfer property of PV2000 in the PSCs, we used triple A-cation perovskite film (cesium [Cs]/formamidinium [FA]/methylammonium [MA]) and deposited PV2000 HTL on the perovskite layer. Figure S2, Supporting Information, shows the UV–vis spectrum of the perovskite film, indicating a bandgap of 1.61 eV. To explore the charge transfer at perovskite/HTL interface, we measured the PL spectra of the bare perovskite, perovskite/PV2000, and perovskite/spiro films (Figure 1c). The PL results suggest a strong quenching effect in the perovskite/PV2000 sample compared with the pure perovskite, which is slightly stronger than the perovskite/spiro sample. We also confirm this result using time-resolved PL (TRPL) measurement, as shown in Figure 1d. The TRPL curves were fitted by a biexponential equation and the fitting parameters are shown in Table S1, Supporting Information. TRPL results follow the same trend as PL spectra, where the perovskite/PV2000 sample shows shorter lifetime than its counterparts, suggesting better hole transfer property in this sample.

Figure 1. a) TGA curve and b) UV–vis and PL spectra of PV2000 film. c) PL spectra and d) TRPL curves of the bare perovskite, perovskite/spiro, and perovskite/PV2000 films.
To evaluate the role of PV2000 as an HTL in solar cells, planar PSCs with normal structure were fabricated. Figure 2a,b shows the cross-sectional scanning electron microscopy (SEM) image of the PV2000-based device and its schematic, respectively, with indium-doped tin oxide (ITO)/SnO$_2$/perovskite/PV2000/Au architecture (n–i–p). For comparison of PV performance, we consider PSCs with spiro HTL as reference device. Figure 2c shows the J–V curves of the best performing PSCs with both PV2000 and spiro HTLs under reverse bias measured under AM1.5G standard condition. Table 1 shows the PV parameters of these PSCs. A PCE of 19.62% (open-circuit voltage [$V_{OC}$]: 1.13 V, short-circuit current density [$J_{SC}$]: 22.85 mA cm$^{-2}$, and fill factor [FF]: 76%) is achieved for the reference device with spiro HTL. In contrast, the device with PV2000 HTL shows a PCE of 18.93% ($V_{OC}$: 1.11 V, $J_{SC}$: 22.75 mA cm$^{-2}$, and FF: 75%), which is comparable with the reference device. The inset graph in Figure 2c shows the maximum power point (MPP) tracking of the PV2000 and spiro PSCs over 60 s, indicating stabilized PCEs of 18.74% and 19.48%, respectively. In fact, the main reasons behind lower PCE in PV2000 PSCs are the lower values of $V_{OC}$ and FF.

Figure S3, Supporting Information, shows the statistics of the PV parameters for the corresponding devices, indicating the same trend as best performing devices. We also measure the hysteresis indices (HIs) of these devices by measuring the J–V curves in both reverse and forward scan directions and using the following formula

$$HI(\%) = \left(\frac{PCE_{Reverse} - PCE_{Forward}}{PCE_{Reverse}}\right) \times 100$$  \hspace{1cm} (1)

Figure S4, Supporting Information, shows the statistic of HI for 12 devices in both cases and the devices with PV2000 show an average HI of 1.93%, lower than that of (2.52%) spiro PSCs, due to better hole transfer at the perovskite/HTL interface.

To verify that the $J_{SC}$ is almost the same in both devices, we measure the external quantum efficiency (EQE) and extract the $J_{SC}$ for both devices by integration of solar spectrum influenced by EQE over the entire wavelength (Figure 2d). As shown in Table 1, the integrated $J_{SC}$ is 21.97 mA cm$^{-2}$ for reference device and 21.86 mA cm$^{-2}$ for PV2000 PSC, which are almost the same and well-matched with the measured J–V results. Notably, the aforementioned PV results for PV2000-based PSCs are obtained after optimization of PV2000 film thickness. We found that 20 nm-thick PV2000 film is the optimum condition for the fabrication of efficient PSCs (Figure S5, Supporting Information).

To further improve the efficiency of the PV2000 device, we study the band diagram of both HTLs using the UPS. Figure 3a shows the UPS results of the PV2000 and spiro films deposited on ITO glass. We extracted the HOMO levels of both PV2000 and spiro to be $-5.55$ and $-5.27$ eV, respectively. Figure 3b shows the band diagram of the PSCs based on these values and literature.$^{[23]}$ As seen, the band offset between

Table 1. J–V parameters of the best performing devices under reverse scan.

| Device       | $V_{OC}$ [V] | $J_{SC}$ [mA cm$^{-2}$] | FF [%] | PCE [%] | $J_{SC}$ from EQE |
|--------------|--------------|--------------------------|--------|---------|-------------------|
| Spiro        | 1.13         | 22.85                    | 76     | 19.62   | 21.97             |
| PV2000       | 1.11         | 22.75                    | 75     | 18.93   | 21.86             |
| PVP/spiro    | 1.15         | 22.71                    | 77     | 20.1    | 21.81             |
| PVP/PV2000   | 1.16         | 22.65                    | 78     | 20.5    | 21.72             |
perovskite and PV2000 (150 meV) is much lower than that of (430 meV) perovskite with respect to the spiro, suggesting the great potential of PV2000 in terms of band alignment. When the HOMO level of the HTL is much higher than the VB of the perovskite layer, interface recombination becomes prominent because of the reduction of the activation energy for the carrier recombination, resulting in low \( V_{OC} \). When the driving force for the carrier is high, after transfer, they can easily recombine with the holes leaking from the VB. In contrast, if the HOMO level of the HTL is too low (deeper [lower] than the VB of the perovskite), the barrier for the photogenerated carrier flow is enhanced. Consequently, there is an optimum band offset for the HTL and the absorber, and it requires to be in the range of 0.0–0.3 eV.\[^{52}\] However, the HOMO level is not the only parameter and the bandgap of the HTL is also an effective factor for the interface recombination.\[^{25}\] Because PV2000 has a bandgap of 1.67 eV, much lower than that of spiro, the PV2000 devices show lower \( V_{OC} \). In fact, the photogenerated electrons in the perovskite film can potentially reach to the holes located at the VB of the PV2000 interfaces due to the lower energy barrier at the interface of perovskite/PV2000 and thus recombine with the holes.\[^{25,53}\] Consequently, in the next step, we modify the interface using a thin layer of PVP.\[^{54}\] PVP layer can effectively address this issue due to its large bandgap. As reported in the literature, PVP can also passivate the surface of perovskite film and reduce the non-radiative recombination, resulting in higher \( V_{OC} \) and FF in the device.\[^{55}\]

Using this approach, we fabricate PSCs with spike structure,\[^{53}\] which can drastically improve the charge transfer and reduce the interface recombination, as schematically shown in Figure 3b. Therefore, we fabricate PSCs using new architecture for both PV2000 and spiro HTLs, and measure their \( J-V \) curves. Figure 3c shows the \( J-V \) curves of the best performing devices with PVP/PV2000 and PVP/spiro layers. Using this modification, the \( V_{OC} \) of the PSC with PVP/PV2000 increases up to 1.16 V, which is higher than that of PVP/spiro device (1.15 V). More interestingly, both devices show high FF up to 78%, indicating the beneficial role of interface engineering in PSCs. Based on this approach, a PCE of 20.5% was achieved using PVP/PV2000, which is better than the PVP/spiro-based device (20.1%). The MPP results shown in inset graph of Figure 3c are well-matched with the \( J-V \) results, where the stabilized PCEs for the PVP/PV2000 and PVP/spiro devices are 20.39% and 20.07%, respectively. The statistics of PV results shown in Figure S3, Supporting Information, are in good agreement with the best performing PSCs. As shown in Figure 3c, the \( J_{SC} \) remains almost the same in the modified devices, as further proved by EQE measurement (Figure 3d). The extracted \( J_{SC} \) for PVP/PV2000 and PVP/Spiro PSCs are 21.72 and 21.81 mA cm\(^{-2}\), respectively, confirming the \( J-V \) results. Similarly, the average of HI for both devices remains the same by adding PVP (Figure S4, Supporting Information).

To investigate the \( V_{OC} \) enhancement in the PVP/PV2000 device, we analyze the dark \( J-V \) curves of these devices (Figure S6, Supporting Information). Our results indicate that the PVP/PV2000 PSC has lower dark current compared with that of PVP/spiro device, which can be ascribed to the lower recombination and better charge transfer in PVP/PV2000 PSCs. In addition, we measured the electrochemical impedance...
spectroscopy (EIS) for both devices to further study the perovskite/HTL interface. Figure S7, Supporting Information, shows the Nyquist plots of both PSCs with PVP/PV2000 and PVP/spiro. The inset image shows the equivalent circuit for fitting purpose. In this circuit, $R_s$, $R_{\text{rec}}$, and $C$ are series resistance, recombination resistance, and capacitance, respectively. We find that the device with PVP/PV2000 has a $R_s$ of 97 $\Omega$, which is smaller than that of PVP/spiro device (132 $\Omega$). This can explain the slightly higher FF in the device with PVP/PV2000 HTL. Moreover, the recombination resistance (corresponded to the radius of semicircle) of PSC with PVP/PV2000 (1126 $\Omega$) is larger than the device with PVP/spiro (432 $\Omega$), indicating the lower interface recombination in PVP/PV2000 device, which is a good explanation for the observed higher $V_{\text{OC}}$ in this device.

One of the main advantages of dopant-free HTL in PSCs is the improvement in device stability. To make PSCs ready for commercialization, the devices need to work efficiently over time upon exposure to humid environment, heat, and light. To study the stability of our PSCs, shelf-life, operational, and thermal stabilities of both PVP/PV2000 and PVP/spiro PSCs are measured. To see the nature of spiro and PV2000 films under humid environment, contact angle (CA) of water droplets placed on these films are estimated, as shown in Figure 4a,b, respectively. The CA is 56° for spiro and 114° for PV2000 films, indicating superhydrophobic nature of PV2000 film. Due to this effect, we find that the PV2000-based device is more stable in ambient condition with 50–60% relative humidity (RH), as shown in Figure 4c, where it retains 71% of its initial PCE value after 21 days, much better than the spiro-based PSC (39%). We also test the operational stability of these PSCs over 250 h under continuous illumination and nitrogen flow (Figure 4d). Our results prove better stability of PV2000 PSC under light, mainly due to lack of any dopant in this device and its high thermal stability (Figure 1a). This device shows only 12% PCE loss under illumination, which is more stable than spiro device with 51% PCE loss. To evaluate the effect of PVP, we performed light stability for PSCs with only PV2000 and spiro HTLs, as shown in Figure S9, Supporting Information. Our results show that the PCE under light is degraded faster for both devices without PVP. As reported in the literature,\textsuperscript{199} spiro HTL usually has many voids, which can facilitate the ion migration through the HTL, especially under illumination. In case of PV2000, we have not seen such voids and the PV2000 uniformly covers the perovskite film, as shown in Figure S8, Supporting Information. Because PV2000 has a $T_{\text{DEG}}$ of 434 °C, we measure the thermal stability of this device at 65 °C under illumination and nitrogen flow. Interestingly, this device maintains 80% of its primary PCE after 100 h, which is greatly better than the spiro PSC (24%). These results highlight the role of dopant-free and stable HTL in the PSCs, which is a crucial step for their commercialization.

In summary, we propose a dopant-free organic HTL, PV2000, for the fabrication of efficient and stable PSCs. Our characterization results indicate that PV2000 has a great hole mobility and thermal stability to work as HTL in PSCs. TRPL and UPS results prove that PV2000 film is perfectly aligned with the VB of triple-A cation perovskite film and has strong quenching effect at the interface. Using PV2000, a planar PSC with a comparable PCE of 18.93% is achieved. We further improve the PCE of the device using interface engineering approach by deposition of a thin layer of PVP at the perovskite/HTL interface. Furthermore, EIS and dark $J-V$ measurements confirm the lower interface recombination and series resistance in PVP/PV2000 PSCs. Based on this configuration, we achieve a PSC with a maximum PCE of 20.5% with negligible hysteresis, which is superior than the performance of device with spiro HTL (20.1%). More importantly, our PV2000 device depicts great stability under light, heat, and humid environment due to the water-repellent nature of PV2000, its high thermal stability, and lack of dopants. Our PV2000 PSC retains 88% of its original PCE after 250 h under illumination and nitrogen flow, which is much better than spiro-based device.

**Experimental Section**

**Device Fabrication:** Prepatterned ITO (TFD) glasses were first cleaned in Triton X-100 (1 vol% in deionized [DI] water), acetone, and isopropanol baths together with sonication for 20 min in each batch. Then, they were further treated by oxygen plasma for 15 min. Then, SnO$_2$ nanocrystal (Alfa Aesar, 15% in water, further diluted three times in water, ≈5 nm particle size) was spin-coated on ITO glass at 4000 rpm for 40 s, followed by annealing at 150 °C for 30 min.

**Colloidal Dispersion:** For perovskite film, triple-A cation perovskite solution was prepared by dissolving PbI$_2$ (1.1 M, TCI), formamidinium iodide
The solution was heated on a hotplate at 75 °C overnight (6 h) inside a nitrogen-filled glovebox. Before deposition, the SnO2-coated ITO glasses were treated by oxygen plasma for 15 min to make a hydrophilic surface. Then, the perovskite solution was deposited on the substrates in two steps using a spin-coater: 1000 rpm for 10 s and 6000 rpm for 30 s. In the second step, right 10 s prior to the end of spinning, 85 μL of chlorobenzene was poured on top of the perovskite film as antisolvent. Afterward, the films were annealed inside a glovebox at 105 °C for 40 min.

To deposit HTL, solutions of spiro-OMeTAD and PV2000 (RayenerTek) were prepared by dissolving 80 and 5 mg of these polymers in 1 mL chlorobenzene, respectively. In case of spiro, 19 μL of lithium bis(trifluoromethanesulfonyl)imide (Li-TFSI) solution (520 mg dissolved in 1 mL acetonitrile) and 32 μL of 4-tert-butyl pyridine (TBP) were added into the initial solution. These solutions were deposited by spin-coating on perovskite films at 4000 rpm for 20 s with a ramp rate of 2000 rpm s⁻¹. To complete the device architecture, 100 nm-thick gold was coated on top of the devices using thermal evaporation through a shadow mask. In this work, all devices with PV2000 were without doping and all spiro-based devices were with doping.

Film Characterization: Film morphology and cross-section were studied by focused ion beam (FIB)-equipped SEM (Helios). Optical properties were analyzed by UV–vis (Varian Cary 5) and PL measurement (Horiba Jobin Yvon Ltd.). A picosecond pulsed diode laser with excitation of 405 nm and pulse width of 49 ps (EPL-405) was used to record TRPL data. The PL lifetime was estimated using a biexponential fitting \( t(t) = a_1 \exp(-t/\tau_1) + a_2 \exp(-t/\tau_2) \), where \( a_1 \) and \( a_2 \) are the amplitude and the lifetime, respectively. For CA measurement, DFA100 (KRUSSS) was used. To perform UPS data, AXIS NOVA (Kratos Analytical Ltd., UK) was used with a He I line (21.2 eV) source. TGA was measured by UNIAX/TGA7 (PerkinElmer) with both heating and cooling rates of 10 °C min⁻¹.

Hole mobility of the PV2000 film was characterized by the fabrication of hole-conducting only device as reported in the literature. In this regard, poly(3,4-ethylenedioxythiophene):polystyrene sulfonate (PEDOT: PSS) was first deposited on ITO glass at 2500 rpm for 45 s, followed by annealing at 150 for 10 min. Afterward, the PV2000 solution (5 mg mL⁻¹) was spin-coated at 4000 rpm for 20 s (2000 rpm ramp rate) on the substrates, followed by deposition of 100 nm-thick gold using thermal evaporation. Then, \( J-V \) curve of the hole-conducting only device was recorded using a parameter analyzer, and SCLC approach was used to estimate the mobility of the PV2000 using the following equation

\[
J = \frac{9}{8} \varepsilon_i \varepsilon_0 \mu \frac{V^2}{d^3}
\]

where \( d \), \( \mu \), \( V \), and \( J \) are film thickness, hole mobility, applied bias, and current density, respectively. In the calculation, \( \varepsilon_i \) (the vacuum permittivity) and \( \varepsilon_0 \) (the dielectric constant) of the PV2000 were assumed to be 8.85 × 10⁻¹² F m⁻¹ and 3, respectively, which is common for organic semiconductors.

Device Measurement: For \( J-V \) measurement, a sun simulator (xenon lamp [450 W], Oriel, USA) equipped with a Keithley model 2400 (USA) was used. The light was calibrated using a standard silicon solar cell (Newport). The active area of devices was 0.054 cm². During the \( J-V \) scan, the light intensity was 100 mW cm⁻² and the scan rate was 10 mV s⁻¹. The EQE spectra were recorded by a commercial setup (Arkeo-Ariadne, Cicc Research s.r.l.) with xenon lamp (300 W). The device stability was measured in ambient condition (50–60% RH) and also in a nitrogen-filled glovebox using a white LED lamp (AM1.5G condition). The RH% in our lab is normally 50–60%. In this text, the MPP of the devices was monitored over time. For thermal stability test, the devices were placed on a hotplate inside a glovebox at 65 °C temperature. The EIS measurement (Solartron Analytical) was performed at zero bias with frequency ranged from 0.1 Hz to 1 MHz under dark condition.

Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

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Conflict of Interest

The authors declare no conflict of interest.

Keywords

device stability, hole transporting layer, perovskite solar cells, power conversion efficiency, PV2000 polymer

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