Fully Solution-Processed n–i–p-Like Perovskite Solar Cells with Planar Junction: How the Charge Extracting Layer Determines the Open-Circuit Voltage

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Solution-processable hybrid perovskite semiconductors have risen to the forefront of photovoltaics research, offering the potential to combine high power conversion efficiency with low-cost fabrication and the shortest energy payback time.\(^{1,2}\) The first architectures made use of nanocrystalline TiO\(_2\) deposited on the transparent electrode, both in the form of mesoporous and compact films, as selective contact for electrons alike the so-called Grätzel solar cells.\(^{3–5}\) Such a structure is generally indicated in the field as direct architecture as electrons are collected at the front transparent electrode and holes at the metal contact (often gold). Further development has been driven by empirical optimization strategies and testing of a variety of architectures. One of these promising architectures uses fully organic selective contacts. This choice brings, first of all, the advantage of low temperature processing; additionally, a beneficial effect in terms of electrical stabilization of the device with organic electron extraction layers in a flat junction configuration, with respect to the use of TiO\(_2\), has been repeatedly observed.\(^{6–8}\) So far, architectures with fully organic selective contacts are either limited to the so-called inverted architecture, as a consequence of difficulties in the solution processing of multilayers, or they have to rely on vacuum deposition methods.\(^{7–9}\) Solution-processed, flat junctions, multilayered direct architectures, which would open the path toward large-area, low-temperature processing of high-efficiency direct cells, have been poorly explored, and their power conversion efficiencies are still limited.\(^{10–12}\) Intuitively, the development and optimization of different architectures need a full engineering of different interfaces. This is especially critical for metal halide perovskites that, unlike more widely studied inorganic semiconductors, have fluctuating ionic structures where tilting, distortions, and polarizability of the lattice strongly affect their optoelectronic properties. This makes the reliability of perovskite-based devices strongly dependent on the control of the microstructure of the active material and of its response to external stimuli, such as chemical interactions upon interface formation,\(^{13}\) electric field,\(^{14,15}\) light,\(^{16}\) and environmental agents.\(^{17,18}\)

It is also worth noting that, while perovskite solar cells have made spectacular progress in terms of solar-to-electrical current conversion efficiency in very short time, such advances are mainly based on an empirical approach, which has led to a myriad of architectures, whose advantages and limitations cannot be attributed yet to a fundamental property of the active material or to the design and processing of the device.

Herein we present the fabrication of a fully solution-processed direct perovskite solar cell with planar junction, reaching power conversion efficiencies (PCEs) close to 19\% and an open-circuit voltage (\(V_\text{oc}\)) larger than 1.1 V. Such a result is achieved by using a fullerene derivative, the [6,6]-phenyl-C\(_6\)1-butycry styryl dendron ester (PCBSD) functionalized with a dendron containing two styryl groups as thermal cross-linkers, as a selective contact for electrons. In situ cross-linking of
PCBSD by thermal treatment forms a robust, adhesive, and solvent-resistant thin electron accepting layer, on top of which a high quality perovskite film processed from solution can be grown. We show that the choice of the electron extracting layer (EEL) is of utmost importance to maximize the solar cell $V_{oc}$ as interface engineering accounts not only for the energy level alignment between the EELs and the perovskite, but also for the quality of the microstructure of the perovskite bulk film that is driven by the substrate surface. We further demonstrate that in this device architecture, at $V_{oc}$ conditions, the high carrier density is sustained not only by the charge extracting contacts but also by the semiconducting active layer. This rationalizes the optimal optoelectronic performance of the proposed devices as a beneficial effect of the engineering of the solution-processed EEL/perovskite interface in terms of microstructure and reduced carrier recombination/charge extraction improvement.

The PCBSD is prepared by optimizing the synthesis route described by Hsieh et al. in order to achieve a final yield of 81% (see the Supporting Information for details). The cross-linking of PCBSD is a thermal-induced radical polymerization between the terminal vinyl bonds of PCBSD (see Figure 1a).[20,21] In order to optimize the timing of the cross-linking process at 160 °C (see Figure S16 of the Supporting Information for the temperature optimization), its progress is monitored with in situ infrared (IR) spectroscopy for 45 min. Figure 1b presents the initial and the final spectra. The bands at 987 and 908 cm$^{-1}$, which are assigned to the CH and CH$_2$ wagging of the vinyl groups in trans conformation (see Figure S11 in the Supporting Information for assignment through DFT calculations), show an absolute reduction in intensity in time during the thermal cross-linking. Their lowering is due to the conversion of the sp$^2$ carbon atoms of the vinyl groups into sp$^3$ carbon atoms, which occurs during the PCBSD cross-linking.[22] The band at 908 cm$^{-1}$ is selected for quantitative analysis and monitoring of the cross-linking time. The peak area of the band at 908 cm$^{-1}$ is determined every 5 min up to a final reaction time of 45 min. The values, normalized over the peak area at $t=0$, show that the reaction follows a first-order exponential decay and 35% of the carbon atoms of the vinyl groups are converted after a 45 min thermal annealing (Figure 1c). To prove that the achieved cross-linking degree is sufficient enough for the cross-linked PCBSD (C-PCBSD) layer to be solvent resistant, UV–vis spectra of the substrates are measured before and after being rinsed with dimethyl formamide (DMF). As-cast PCBSD films are almost completely washed off (Figure 1d), whereas only a weak decrease in the intensity of the bands is shown after cross-linking induced with a 30 min thermal annealing at 160 °C (Figure 1e).

Before incorporating such EELs in a device structure, we tested its electronic properties. One of the main challenges to face upon cross-linking procedure of an organic semiconductor is a possible degradation of its transport properties.[23] C-PCBSD has been tested in a field-effect transistor (FET) geometry, which is quite demanding device in terms of electronic properties, as it requires in-plane charge transport along tens of micrometers. Electron percolation pathways can be evidently formed and a neat field-effect modulation of the current can be observed, already in a nonoptimized device, exhibiting an electron mobility in the order of $10^{-5}$ cm$^2$ V$^{-1}$ s$^{-1}$ for surface charge densities in the range $8 \times 10^{15}$ to $3 \times 10^{16}$ m$^{-2}$ ($1.3 \times 10^{-7}$ to $5.1 \times 10^{-7}$ C cm$^{-2}$) (Figure S1 in the Supporting Information). This result shows that our C-PCBSD has almost an order of magnitude higher mobility than what previously reported in undoped C-PCBSD-based FET devices.[10] Thus we can safely
confirm that the C-PCBSD film can sustain in-plane electron transport over several micrometers, and it is therefore likely to show good transversal electron transport properties over tens of nanometres of its thickness thus being a potential candidate as a thin EEL.

To start the solar cell fabrication, a very thin layer of C-PCBSD (less than 10–20 nm; see Figure S14 in the Supporting Information) is deposited onto the FTO/TiO2 substrates as thicker films eventually lead to an increase of the device series resistance (see Figure S15 in the Supporting Information). Its surface work function is measured in air by Kelvin probe and it results more electronegative with respect to that of TiO2 and comparable to the [6,6]-phenyl-C61-butyric acid methyl ester (60-PCBM) (see Table S1 in the Supporting Information). Subsequently a methylammonium lead iodide (CH3NH3PbI3) thin film is directly synthesised from solution on top of FTO/TiO2/C-PCBSD via a two-step procedure (see the Materials and Methods section in the Supporting Information for details). The final thickness of CH3NH3PbI3 depends on the thickness of the PbI2 layer (see the Supporting Information for the layer optimization). Figure 2a shows X-ray diffraction spectra measured before and after CH3NH3PbI3 spun cast on top of PbI2 followed by thermal annealing on top of TiO2 and TiO2/C-PCBSD substrates. In both cases, we do not observe any trace of the crystal precursors and the sharp peaks suggest the formation of large perovskite grains. Figure 2b–e shows the top-view images of the perovskite films grown on TiO2 and TiO2/C-PCBSD, respectively, at different magnifications. The perovskite films show a good coverage on both substrates; however, the film grown on TiO2/C-PCBSD presents much larger grains (>1 μm) and a lower roughness. The static contact angle of water on C-PCBSD is more than twice larger with respect to that of TiO2 and comparable to the [6,6]-phenyl-C61-butyric acid methyl ester (60-PCBM) (see Table S1 in the Supporting Information). Subsequently a methylammonium lead iodide (CH3NH3PbI3) thin film is directly synthesised from solution on top of FTO/TiO2/C-PCBSD via a two-step procedure (see the Materials and Methods section in the Supporting Information). Subsequently a methylammonium lead iodide (CH3NH3PbI3) thin film is directly synthesised from solution on top of FTO/TiO2/C-PCBSD via a two-step procedure (see the Materials and Methods section in the Supporting Information for details). The final thickness of CH3NH3PbI3 depends on the thickness of the PbI2 layer (see the Supporting Information for the layer optimization).

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It is worthy to notice that flat junction solar cells, especially those with direct architecture, hardly achieve a Voc of 1 V.[4,26,27] However, here we show a Voc that is consistently higher than 1.1 V (see statistics in Figure S3 in the Supporting Information). In this regard, these devices even outperform those made with 60-PCBM (Figure S5, Supporting Information), despite the two EELs presenting very similar energy levels, which will likely align in the same way once interfaced with the perovskite semiconductor, given the same nature of the material. Recently, it has been shown that the structural order of the 60-PCBM layer has a significant impact on the enhancement in Voc due to a concomitant reduction of the energy disorder, as deduced from the significant decrease in the DOS of the organic semiconductor.[28] However, here, while the 60-PCBM clearly shows a sharp X-ray diffraction peak at 20.5° as a signature.
of order and crystallinity, there is hardly any diffraction peak in C-PCBSD (see Figure S6 in the Supporting Information), thus excluding any direct role of the electron extracting layer morphology. Note that, for a fair comparison, we grew the perovskite film by thermally evaporating PbI$_2$ followed by MAI deposition by spin coating from 2-propanol, a procedure that some of us have recently developed\[8\] to preserve the soluble 60-PCBM layer and we noticed, also in this case, a tendency of forming larger crystallites over the C-PCBSD (see Figure S7 in the Supporting Information). Overall, beyond the interfacial energetics, the recombination dynamics will contribute to the definition of $V_{oc}$ of solar cells. Since the perovskite film is grown on top of the EEL, the latter's choice will determine its microstructure, which may influence the degree of defect states and the carrier recombination losses within the perovskite film.\[29\] In the following we will try to disentangle all these effects by combining excitation density dependent steady state photocurrent measurements and photoinduced transient techniques.

To understand the effect of the fabrication route and the substrate on the quality of the thin perovskite film, we performed photocurrent measurements within a photoconductor device configuration.\[30\] Briefly, planar symmetric gold contacts separated by 1 mm are evaporated on top of the film and continuous wavelength (cw)-laser-induced carriers are extracted by applying a bias voltage of 10 V across them (Figure 4a). Note that the electric fields within this configuration are smaller by a factor of 100 in comparison to the usual electric fields within the solar cell architecture. In Figure 4b, we show the photocurrent plotted as a function of the photoexcitation density. All measured films are $\approx$300 nm thick and grown on a thin layer of TiO$_2$, TiO$_2$/60-PCBM, and TiO$_2$/C-PCBSD on glass; this guarantees to have a thin film microstructure comparable to the one present in the device while observing mainly bulk processes upon top layer illumination. Since perovskite cannot be solution-processed on top of TiO$_2$/60-PCBM we also compare two thin films grown on C-PCBSD, one solution-processed and the other grown through thermal evaporation.

While all the films show a monotonic growth of photocurrent with the excitation density, they exhibit distinct intensity trends. As a guide for the eyes, we draw hypothetical linear ($\approx$I) and sublinear ($\overline{\sqrt{I}}$ intensity dependent photocurrent in dotted lines. The photocurrent trends in all the films follow sublinear behavior at low excitation densities and reach a linear trend at higher densities. Such a behavior can be explained within a trap-limited Shockley–Read–Hall (SRH)-like recombination scenario, where at low-excitation densities, electrons (holes) are most likely trapped within defects and thus do not contribute to the photocurrent. In such case, the photocurrent has a sole contribution from hole (electron) population, which can be shown to scale as $\overline{\sqrt{I}}$ under steady state conditions,\[31\] subsequently giving rise to the sublinear behavior of the photocurrent.

### Table 1. Summary of the best device performance for perovskite solar cells with TiO$_2$ and TiO$_2$/C-PCBSD as EELs with the structure of FTO/EEL/CH$_3$NH$_3$PbI$_3$/spiro-OMeTAD/Au.

| Device          | Scan direction | Scan rate [V s$^{-1}$] | $V_{oc}$ [V] | $J_{sc}$ [mA cm$^{-2}$] | FF [%] | PCE [%] |
|-----------------|----------------|------------------------|--------------|------------------------|--------|---------|
| TiO$_2$         | Reverse scan   | 0.2                    | 0.80         | 19.0                   | 74.5   | 11.4    |
|                 |                | 0.023                  | 18.7         | 73.1                   | 11.6   |         |
|                 | Forward scan   | 0.2                    | 0.73         | 19.1                   | 64.4   | 9.0     |
|                 |                | 0.023                  | 18.9         | 64.7                   | 8.9    |         |
| TiO$_2$/C-PCBSD | Reverse scan   | 0.2                    | 1.12         | 21.1                   | 79.0   | 18.7    |
|                 |                | 0.023                  | 21.1         | 77.8                   | 18.3   |         |
|                 | Forward scan   | 0.2                    | 1.10         | 21.2                   | 69.4   | 16.2    |
|                 |                | 0.023                  | 21.1         | 76.1                   | 17.8   |         |
is a substantial contribution from the untrapped electron (hole) current. Nevertheless, given that the effective defect density is around \(10^{16} \text{ cm}^{-3}\), the carrier densities at the investigated densities still lie within the trap-limited regime,\(^{32,33}\) where the electron (hole) population can be shown to scale linearly with intensity within the SRH formalism.

Since the photocurrent is proportional to the number of free charge carriers and therefore composed of electron and hole current density, \(j = j_e + j_p\), we expect that the general light intensity dependence in the simplest approximation can be described with a linear and square root contribution. The ratio between this sublinear and linear component is therefore an indication for the degree of trap-limited behavior of the perovskite films, with greater linear component suggesting lower degree of carrier trapping. The photocurrent extracted from films grown on C-PCBSD in Figure 4b follows a behavior closer to the linear limit at excitation densities of \(10^3 - 10^5 \text{ cm}^{-3}\) relevant for the PV operation, in comparison to the films grown directly on \(\text{TiO}_2\), \(\text{TiO}_2/60\)-PCBM, and \(\text{TiO}_2/\text{C-PCBSD}\) photovoltaic devices.

Figure 4. a) Sketch of the sample geometry for photocurrent measurement. b) Photocurrent as a function of excitation density of four perovskite layers deposited via evaporation (eMAPbI\(_3\)) or spincoating (sMAPbI\(_3\)) on different electron extracting layers. The scattered points represent the measured data, the lines are fits according to \(\gamma = c(\alpha x + b\sqrt{x})\). The gray lines show the two extreme cases of the fit model (\(b = 0\) and \(d = 0\), respectively) as a guide to the eyes.

We now turn to the measurement of carrier recombination kinetics in working conditions (i.e., under 1 sun light bias), comparing the different device architectures presented in this work. First, we carried out the measurement of the device capacitances using photoinduced differential charging after subtraction of the geometrical capacitance (Figure 5a) as reported before.\(^{34}\)

Keeping the layers thickness comparable, these measurements allow us to compare the accumulated charge densities in solar cells at different bias. We report such densities in Figure 5b, showing that the solar cell with the C-PCBSD fullerene selective contact is capable to accumulate higher amount of charges. It is worthy to mention that the capacitance increases super linearly for voltages above 0.8 V and it is independent on the thickness of the C-PCBSD layer (see Figures S12 and S13 in the Supporting Information). This profile has been also observed in other thin film solar cells alike polymer\(^{35}\) and small molecule organic solar cells\(^{36,37}\). While the linear increase of capacitance with the light bias is correlated to the accumulation of carriers at the device contacts,\(^{38}\) the super linear behavior indicates that the contacts are not able to sustain the entire density of charges which accumulate in the bulk of the device as well. This is further supported by the fact that charge density, as well as the charges lifetimes, scale as the thickness of the perovskite active layer (see Figures S16–S18 of the Supporting Information). Importantly, this leads to a greater split of the quasi-Fermi levels for holes and electrons leading to a larger \(V_{oc}\)—as long as strong recombination losses do not kick in which then points to the importance of the electronic quality of bulk semiconductor layer.

Figure 5. Capacitance after subtraction of a) the geometrical capacitance and b) the charge density of \(\text{TiO}_2\), \(\text{TiO}_2/60\)-PCBM, and \(\text{TiO}_2/\text{C-PCBSD}\) photovoltaic devices.

We now can compare the carrier recombination kinetics for the solar cells at equal carrier densities (\(n\)). As mentioned above, the \(V_{oc}\) of solar cells will be influenced by the materials energetics (band alignment) and the carrier recombination kinetics. Figure 6 illustrates the measurements carried out using the same devices as in Figure 5 in identical light irradiation conditions. As can be seen, the solar cells using C-PCBSD as electron selective contact show longer carrier lifetimes and lower recombination losses. For instance at illumination intensities equivalent at 1 sun, the 60-PCBM-based devices display a total charge density of 20 nC cm\(^{-2}\) alike the \(\text{TiO}_2\)-based ones and carrier lifetimes of 0.6 and 2 µs, respectively (as shown with a vertical line in Figure 6). Nonetheless, considering the same charge density for C-PCBSD perovskite solar cells the carrier lifetime is hundred times slower (\(\tau > 200\) µs). In fact, illuminating these solar cells at 1 sun the total charge was of hundreds of nC cm\(^{-2}\) and still the carrier recombination lifetime was the slowest
between the three devices with a value close to $\tau = 10 \mu$s. For a fair comparison with the decays recorded for the organic-based perovskite solar cells, we have used in Figure 6 the fastest component of the biexponential decay.

In conclusion, by employing a cross-linking strategy we demonstrate a fully solution-processed planar junction solar cell in direct architecture. The device delivers a PCE close to 19% and on 1 sun illumination, the high carrier density achieved in open-circuit condition is not sustained by the extracting contact only, but accumulates within the active layer as well. Thus to enhance the $V_{oc}$, where greater separation of the quasi-Fermi levels is desired, the carrier recombination losses via carrier trapping in the bulk perovskite films has to be minimized. Here, we show that the choice of the EEL layer not only determines the interface energetic but also the electronic quality of the active layer and, with c-PCBSD we achieve superior characteristics in both these aspects. These results, beyond the technological validity provided by the demonstration of a high efficiency, fully solution-processed, flat junction solar cell, which is electrically stable, define an important direction toward an engineered design of highly performing perovskite solar cells.

Supporting Information

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

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