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Amir Hossein Habibi, Pierre Josse, Antoine Labrunie, Frédéric Gohier, Gregory Welch, et al.. Effect of Length of Ethylene Glycol Sigma-Linkers on Triphenylamine-Based Dimers Used as Molecular Donors for Organic Solar Cells. *ChemistrySelect*, 2024, 9 (2), 10.1002/slct.202303515 . hal-04803770

HAL Id: hal-04803770

<https://univ-angers.hal.science/hal-04803770v1>

Submitted on 25 Nov 2024

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Use of ethylene glycol σ -linkers: simple strategy to tune the morphology of push-pull based (photo) active layer

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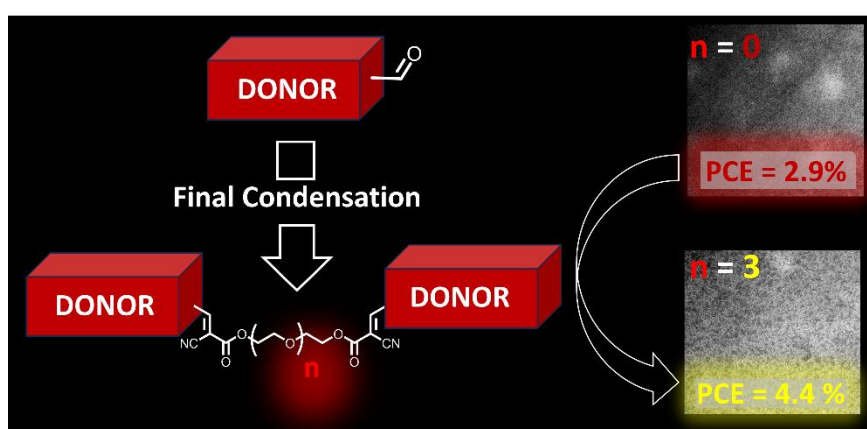
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Abstract: Morphology control of the active layer in organic solar cells is still of major concern since demonstrated to be directly correlated to both efficiency and stability. In this context, we report herein the direct comparison of two push-pull based dimers built around similar absorbing units linked by ethylene glycol σ -connectors of different lengths. Introduced in the last synthetic step, results gathered in this study with benchmark molecular (photoactive) donors emphasized the potential of such simple yet efficient strategy to control the self-assembly and subsequent photovoltaic properties.



Introduction:

Since the early 2000's, organic photovoltaics (OPVs) have attracted considerable research attention breaking efficiency records one after the other.¹⁻³ With increasing power conversion efficiencies (PCEs), almost competing with those of silicon and perovskite based solar cells,⁴ this technology keeps demonstrating its huge potential as a renewable source of energy. Usually prepared from blends containing an electron donor (D) and acceptor (A), a precise control of the morphology of the resulting active layer was demonstrated to be a key parameter to achieve both efficient and stable devices. To do so, several strategies were reported such as the use of co-solvents, thermal annealing and above all, chemical modifications through specific side chains engineering. As a general trend, records in PCEs

usually go hand in hand with a significant increase of the synthetic complexity despite a cost effectiveness usually claimed as a major advantage.^{5,6} In this context, several groups have devoted their efforts in providing simple and accessible strategies and therefore active compounds by deliberately limiting the number of synthetic steps.⁷ Particularly active in this initiative, our groups reported, since 2005, myriads of simple molecular structures successfully used as donors,⁸⁻¹¹ non-fullerene acceptors¹²⁻¹⁵ and even as single compounds.¹⁶⁻¹⁹

Hence, following this guideline and recent results on multimer structures,²⁰⁻²² we report herein the synthesis, characterization and preliminary evaluation of two dimers as molecular donor in organic solar cells (OSCs), built from our benchmark triphenylamine based push-pull (Figure 1).

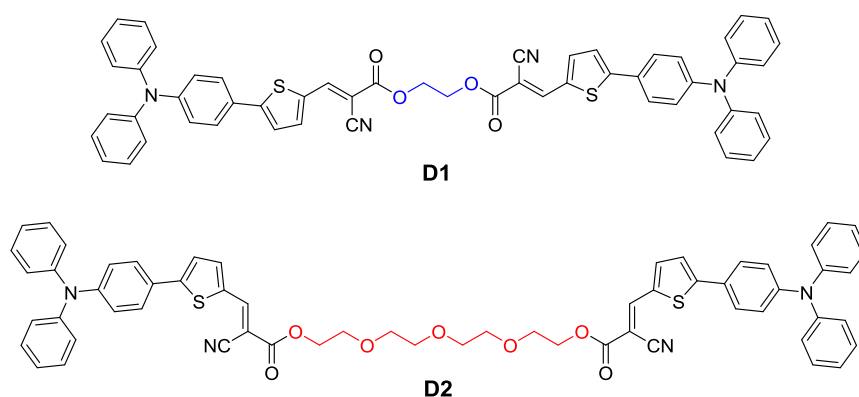


Figure 1. Structure of the two dimers studied herein, *ie*, **D1** and **D2**.

Prepared in only three steps from affordable and commercially available starting materials, this study aims at introducing during the final condensation reaction (*ie*, for a practical and versatile reasons) an ethylene glycol-based sigma (σ) linkers and assess its impact of on the solubility, self-assembly and photovoltaic properties. Stemming from the literature, the replacement of alkyl side chains on active material by oligo(ethylene glycol) (OEG) moieties, or even the simple inclusion of polyethylene glycol (PEG) within the donor/acceptor blends, were indeed found to have beneficial effects on the overall performance.²³⁻²⁵

Materials and Methods

Compounds **1**, **L1** and **L2** were prepared according to an early reported procedure.^{26,27} Reagents and chemicals from commercial sources were used without further purification. Solvents were dried and purified using standard techniques. Silica gel Chromatographies were performed on Aldrich (Saint-Louis, USA) silica gel (technical grade, pore size 60 Å, 230-400 mesh particle size) packed with analytical-grade solvents. Flexible plates ALUGRAM® Xtra SIL G UV254 from MACHEREY-NAGEL (Düren, Germany) were used for TLC. Compounds were detected by UV irradiation (Thermo Fisher Scientific, Waltham, MA, USA). NMR spectra were recorded with an AVANCE III 300 (1H, 300 MHz and 13C, 75MHz) from Bruker (Bruker, Billerica, MA, USA). Chemical shifts are given in

ppm relative to TMS and coupling constants J in Hz. UV-vis spectra were recorded on a Shimadzu UV-1800 spectrometer (Shimadzu, Kyoto, Japan). High-resolution mass spectrometry (HRMS) was performed with a JEOL JMS-700 B/E (JEOL, Peabody, USA). Photoelectron spectroscopy in air (PESA) were carried out on a Riken AC-2 photoelectron spectrometer (Riken Keiki, Tokyo, Japan). Transmission Electron Microscopy (TEM) images were recorded in bright field mode with a JEOL JEM 1400 microscope operating at 120 keV (JEOL, Peabody, USA). A 20mm objective aperture was used in to improve the contrast. Blends were spun cast on PEDOT:PSS coated glass substrates. The films were floated off in deionized water and collected on copper TEM grids. J-V curves of organic solar cells were recorded using a Keithley 236 source-measure unit (Beaverton, USA) and a home-made acquisition program. The light source is an AM1.5 Solar Constant 575 PV simulator (Steuernagel Lichttechnik, equipped with a metal halogen lamp, $100 \text{ mW}\cdot\text{cm}^{-2}$, Morfelden-Walldorf, Germany). The light intensity was measured by a broad-band power meter (13PEM001, Melles Griot, Rochester, USA).

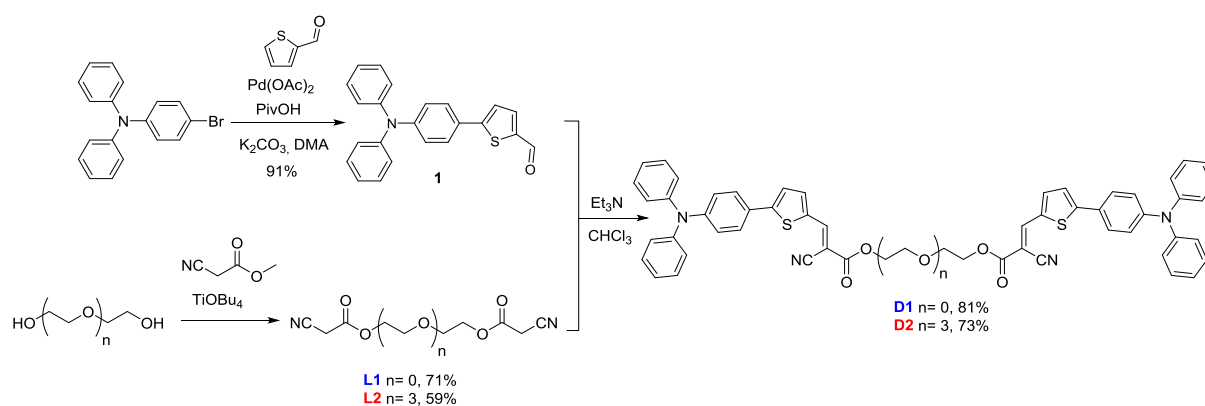
General procedure for the preparation of D1 and D2. 5-(4-(Diphenylamino)phenyl)thiophene-2-carbaldehyde (**1**) (1.20 g ; 3.36 mmol, 2.2 eq.) and **L1** or **L2** (1.53 mmol, 1eq) were poured in a 250 mL round bottom flask and dissolved into 100 mL of HPLC grade CHCl_3 . Two drops of Et_3N were then added and the reaction mixture. After a night of stirring at room temperature, the solvent was removed under vacuum and the crude was directly subjected to silica gel chromatography (eluent: $\text{CH}_2\text{Cl}_2/\text{PE}$ (8:2), CH_2Cl_2 and then $\text{CH}_2\text{Cl}_2/\text{EtOAc}$ (98:2)) to afford the desired products as red powders. **D1** (81%): ^1H NMR (300 MHz, CDCl_3) δ 8.30 (s, 1H), 7.75 (d, $J = 4.2$ Hz, 1H), 7.58 – 7.50 (m, 2H), 7.35 – 7.27 (m, 5H), 7.18 – 7.09 (m, 6H), 7.05 (m, 2H), 4.60 (s, 2H). ^{13}C NMR (76 MHz, CDCl_3) δ 163.20, 155.80, 149.58, 147.28, 147.01, 140.17, 133.99, 129.67, 127.58, 125.84, 125.45, 124.19, 123.39, 122.29, 116.15, 96.23, 63.49. HRMS (EI) calculated for $\text{C}_{54}\text{H}_{38}\text{N}_4\text{O}_4\text{S}_2$ 870.2334, found 870.2338 ($\Delta = 0.4$ ppm). **D2** (73%): ^1H NMR (300 MHz, CD_2Cl_2): δ 8.28 (s, 2H), 7.73 (d, $J = 4.1$ Hz, 2H), 7.55 (d, $J = 8.7$ Hz, 4H), 7.36 – 7.26 (m, 10H), 7.17 – 7.06 (m, 12H), 7.03 (d, $J = 8.7$ Hz, 4H), 4.45 – 4.37 (m, 4H), 3.81 – 3.75 (m, 4H), 3.71 – 3.60 (m, 8H). ^{13}C NMR (76 MHz, CD_2Cl_2): δ 163.47, 155.46, 149.85, 147.37, 146.92, 140.23, 134.34, 129.93, 127.77, 126.13, 125.79, 124.48, 123.71, 122.44, 116.55, 97.21, 71.23, 71.05, 69.28, 65.87. HRMS (FAB): Calculated for $\text{C}_{60}\text{H}_{50}\text{N}_4\text{O}_7\text{S}_2$ 1002.3121, found 1002.3115.

Solar cell fabrication: Pre-patterned indium-tin oxide coated glass slides of $24 \times 25 \times 1.1$ mm with a sheet resistance of $\text{RS} = 7 \Omega/\text{sq}$ were purchased from Visiontek Systems (Cheshire, United Kingdom). The substrates were washed by successive ultrasonic baths with diluted Deconex® 12 PA-x solution (2% in water), water, acetone and isopropanol for 15 min each. Once dried under a steam of air, a UV-ozone plasma treatment (Ossila UV/Ozone cleaner E511, Sheffield, UK) was performed for 15 min. A filtered aqueous solution of poly(3,4-ethylenedioxy-thiophene)-poly(styrenesulfonate)

(PEDOT:PSS; Ossila Al 4083) through a 0.45 μm RC membrane (Minisart® RC 15) was spun-cast onto the ITO surface at 5000 rpm for 40 s before being baked at 120 °C for 30 min. Then, blends of **D1** or **D2** and **PC₇₁BM** were dissolved in chloroform (10 mg/mL) prior to be spun-cast onto the above described PEDOT:PSS layer. Finally, devices were completed by the thermal deposition of lithium fluoride (0.5 nm) and aluminum (100 nm) at a pressure of 1.5×10^{-5} Torr through a shadow mask defining six cells of 27 mm² each (13.5 mm \times 2 mm).

Results and Discussions

The straightforward and common synthetic route to both dimers is depicted in scheme 1.



*Scheme 1. Synthesis of **D1** and **D2***

Built around the same push-pull moieties, used as benchmark since already been demonstrated as simple and accessible donor material in simple organic solar cells, the common precursor **1** was prepared by direct arylation between the 4-bromo-*N,N*-diphenylaniline and the thiophene-2-carbaldehyde as per a home-made procedure. In parallel, ethane-1,2-diol and tetraethyleneglycol were engaged in a tetrabutoxytitanium catalyzed esterification with the methyl 2-cyanoacetate to afford the functionalized linkers **L1** and **L2** that finally reacted with the above mentioned aldehyde **1**, *via* a Knoevenagel condensation, yielding the target compounds. Note that unlike most of side chain engineering studies that involved the decoration of building blocks at early stages, the sigma linker/group is introduced herein during the last step, thus allowing a higher synthetic versatility.

Once purified by conventional silica gel chromatography, optical properties of both dimers were investigated by UV-visible and fluorescence spectroscopy. The absorption and emission spectra of **D-1** and **D-2**, plotted in Figure 2, were recorded in diluted dichloromethane solutions (*ca.* 10^{-6} M) and on thin films spun cast on glass sheets.

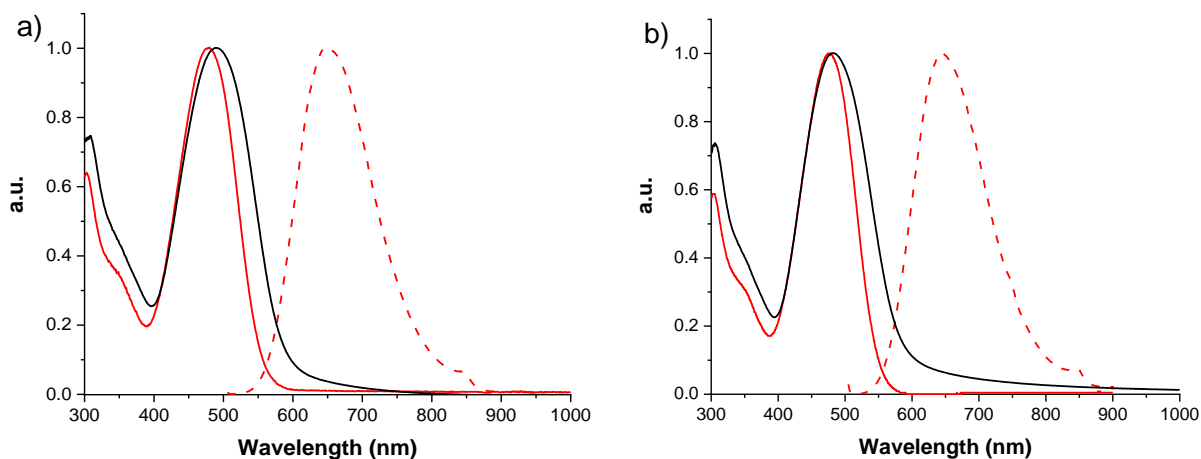


Figure 2. UV-Visible (solid line) and photoluminescence spectra (dashed line) of **D1** (a) and **D2** (b) in solution (red) and as thin-films (black).

As expected, both dimers show similar patterns, in solution, with internal charge transfer (ICT) bands peaking at *ca* 477 nm (characterized by extinction coefficients (ϵ) > 66500 L.mol⁻¹.cm⁻¹) and emission bands centered at *ca* 646 nm associated to a relatively high fluorescence quantum efficiency of \sim 22% (Table 1).

Table 1. Optical data of dimers in solution and as thin films.

Dimer	λ_{abs} (nm) ^a	ϵ_{max} (L.mol ⁻¹ .cm ⁻¹) ^a	ΔE^{opt} (eV) ^a	λ_{em} (nm) ^{a,c}	Φ_{f} (%) ^a	λ_{abs} (nm) ^b	$E_{\text{g}}^{\text{opt}}$ (eV) ^b
D1	478	66500	2.21	648	22	489	2.08
D2	476	69700	2.23	645	22	481	2.13

^a in solution, ^b as thin film, ^c $\lambda_{\text{exc}} = 500$ nm, Standard : Rhodamine B in Ethanol ($\Phi_{\text{f}} = 0.50$).

On the other hand, subtle differences were nonetheless recorded in the solid state, highlighting the effects of the σ -linker on the structural organization. The absorption spectra of **D1** indeed appears slightly more red shifted than that of **D2** (11 nm vs 5 nm) with a steepest onset at low-energies, suggesting higher degree of organization and therefore reduced energetic disorders (more prominent Urbach tail in the case of the longest linker, *ie*, **D2**).²⁸ These features results in different optical bandgaps ($E_{\text{g}}^{\text{opt}}$) values estimated to be of *ca* 2.08 eV and 2.13 eV for **D1** and **D2**, respectively (Table 1). To gain further insight into the impact of the linker on the self-assembly and therefor energetics of the materials (in solid state), thin films were subsequently analyzed by photoemission yield spectroscopy in air (PYSA). Comparison of the spectra plotted in figure 4-a revealed that the longer oligo(ethyleneglycol) based dimer, namely **D2**, exhibits a shallower highest occupied molecular orbital (HOMO) energy level than that of **D1** ($\text{HOMO}^{\text{D2}} = -5.53$ eV vs $\text{HOMO}^{\text{D1}} = -5.47$ eV). Cross

tabulated with optical data, lowest unoccupied molecular orbital energy levels were thus deduced leading to the energetic diagram illustrated in Figure 4-b.

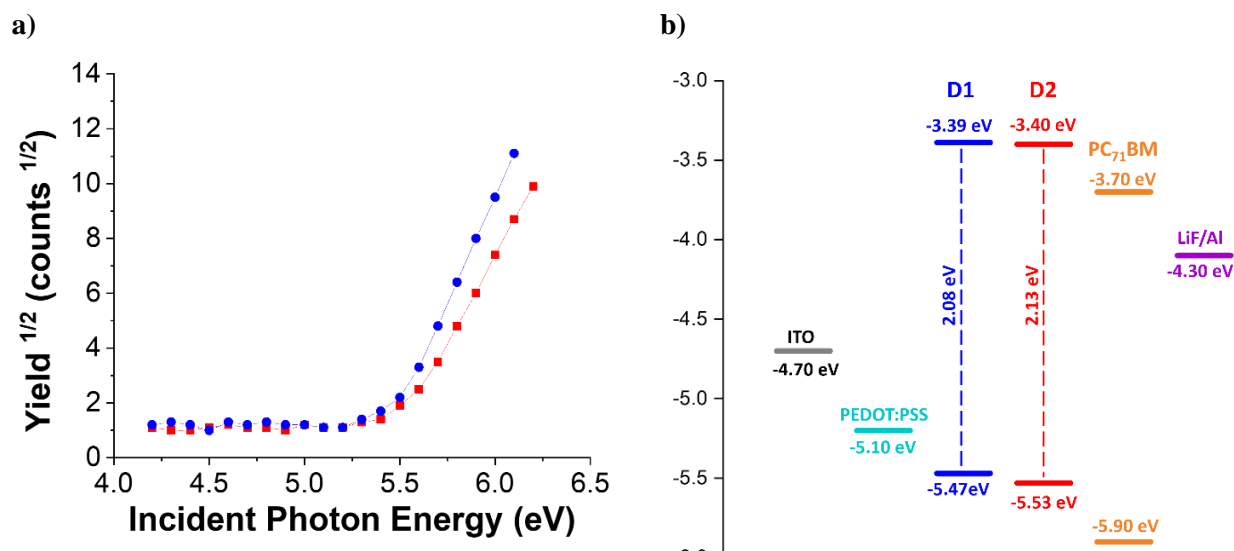


Figure 4. a) photoemission yield spectroscopy in air of **D1** and **D2** recorded on thin films spun cast on glass sheets b) deduced energy level diagrams of **D1** and **D2** compared to those of other constituting materials used for the preparation of OSCs.

With similar LUMO energy levels, the reduction of the optical band gap monitored for **D1** originates from the destabilization of its HOMO energy level induced by a different organization/packing in the solid state. Compatible with the 6,6-Phenyl C₇₁ butyric acid methyl ester (PC₇₁BM) for an energetic point of view, the impact of the linker on the photovoltaic performances was then evaluated through the preparation of air processed bulk heterojunction solar cells of architecture: ITO/PEDOT:PSS/dimer:PC₇₁BM/LiF/Al. Best power conversion efficiencies were obtained with 1:2 weight to weight Donor:Acceptor (D:A) ratio based active layers spun cast at 2000 rpm. The corresponding JV curves and related incident photon to current efficiency (IPCE) spectra are plotted in Figure 5.

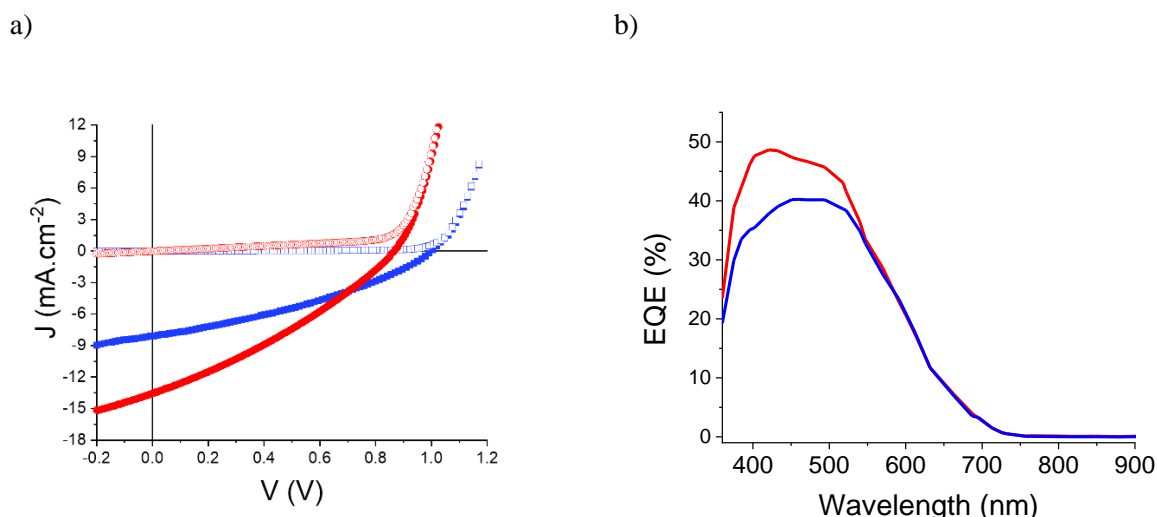


Figure 5. a) J-V curves recorded in the dark (hollow) and under AM1.5G illumination at 100.mW. cm⁻² a(full) and b) IPCE spectra recorded on the best **D1** (blue) and **D2** (red) OSCs

Interestingly, while higher open circuit voltage (V_{oc}) parameters were theoretically expected for **D2** based OSCs (due to its lower lying HOMO energy level), the opposite was actually monitored since V_{oc} values of 0.98 V and 0.86 V were achieved with **D1** and **D2**, respectively. On the other hand, the replacement of the small ethylene linker by a longer oligo(ethylene glycol) lead to a significant improvement of the short circuit current (J_{sc}) parameters with values increasing from 8.2 mA.cm⁻² for **D1** based devices to 13.6 mA.cm⁻² for **D2**. This significant difference was directly confirmed by IPCE measurements (Figure 5-b) highlighting an improved contribution of both the donor and acceptor in **D2** based active layers. With similar fill factors of 36% and 38%, suggesting rooms for optimizations, promising maximum power conversions efficiencies of 2.9 % and 4.4% were therefore calculated for **D1** and **D2** based OSCs respectively.

In an attempt to rationalize such significant difference, particularly on the J_{sc} values, devices were immersed in deionized water to dissolve the PEDOT:PSS and therefore delaminate the active layers from the substrate. Once collected on copper-grids, the latter were subsequently analyzed by transmission electron microscopy (TEM) (Figure 6).

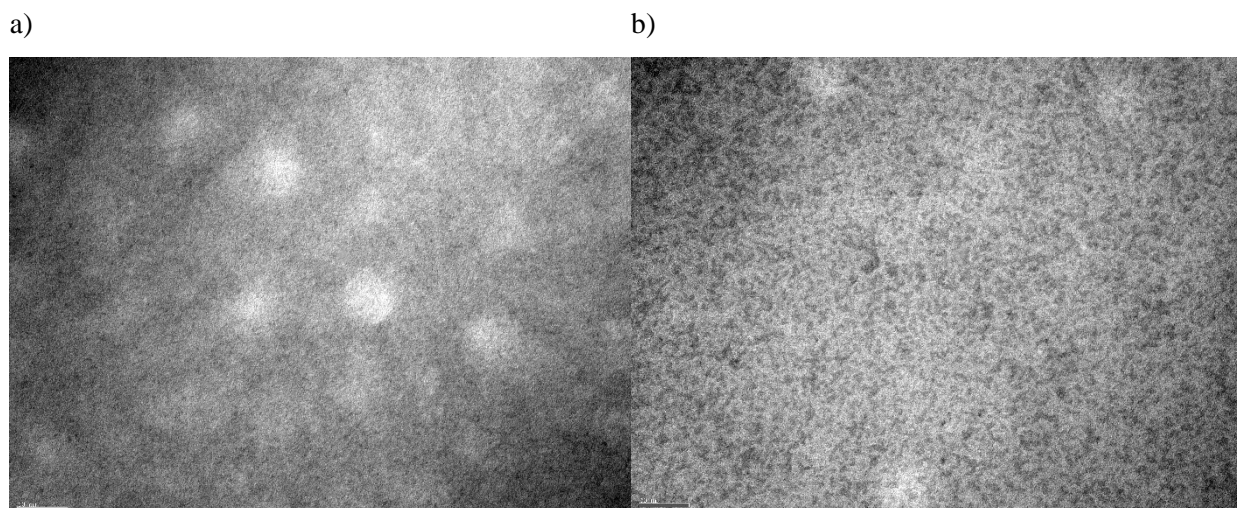


Figure 6. TEM images of **D1** (a) and **D2** (b) based active layers.

Considering that dark grey areas are attributed to fullerene-rich regions, **D2** based blend exhibits a more pronounced phase segregation than that of **D1** which seems to be, according to the photovoltaic performances, beneficial for the carrier generation and transport to the electrodes.

Conclusion

With promising power conversion efficiencies achieved in basic solution and air processed OSCs, the introduction of a sigma linker between the two benchmark absorbing units was found to be an efficient and simple way to *i*) impact the self-assembly properties, *ii*) control the morphology of the blends without the use of additives and/or thermal/solvent annealing and *iii*) improve the photovoltaic properties once embedded in simple devices. This simple yet efficient strategy hence opens doors to original design principles since it can potentially be applied to any photoactive compound whose preparation involves a Knoevenagel condensation.

Acknowledgment

The University of Isfahan is thanked for the PhD grant of A. H. The RFI LUMOMAT (Region Pays de la Loire) is thanked for the PhD grant of A.L. and the French Ministry of Research for the PhD grant of P.J.

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