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The open circuit voltage of encapsulated plastic photovoltaic cells

Y. Berredjem [a,b], N. Karst [a], L. Cattin [a], A. Lakhdar-Toumi [c], A. Godoy [d], G. Soto [e], F. Diaz [e], M.A. Del Valle [e], M. Morsli [a], A. Drici [b], A. Boulmokh [b], A.H. Gheid [b], A. Khelil [c], J.C. Bernède [a,*]

[a] *Université de Nantes, Nantes Atlantique Universités, LAMP, EA 3825, Faculté des Sciences et des Techniques, 2 rue de la Houssinière, BP 92208, Nantes F-44000, France*
[b] *Université de Badji-Ammar Annaba, Faculté des Sciences, Algeria*
[c] *Université d'Oran Es-Senia, LPCM2E, Algeria*
[d] *Docente Facultad Ciencias de la Salud, Universidad Diego Portales, Santiago, Chile*
[e] *Department of Organic Chemistry, Faculty of Chemistry, PUCC, Av Vicuña Mackenna 4860, Cas. 6177, Santiago, Chile*

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Abstract

The variation of the open circuit voltage of organic photovoltaic cells based on a donor/acceptor heterojunction was studied as a function of the couple donor/acceptor and of the encapsulation process used for the cells. The donors used were zinc and copper phthalocyanine and the acceptors were fullerene, perylene-3,4,9,10-tetracarboxylic dianhydride, 1,4-diaminoanthraquinone and *N*,*N*′-diheptyl-3,4,9,10-tetracarboxylic-diimide. The open circuit voltage value increased, as expected, in-line with the difference between the HOMO of the donor and the LOMO of the acceptor. Moreover, for all donor/acceptor couples used, the open circuit voltage value was higher when the cells were in contact with air before encapsulation. This effect is attributed to the formation of a thin Al_2O_3 layer at the organic acceptor/aluminium interface, which increases the shunt resistance of the device and prevents chemical reaction between aluminium and the organic material.

Keywords: Organic solar cell; Interface; Alumina; Heterojunction; CuPc; C_{60}

1. Introduction

Photovoltaic cells (PV cells) are more and more attractive as clean renewable energy source. Organic based PV cells have shown promising potential as an alternative cheap energy source. They provide properties such as mechanical flexibility and easy processability.

Two organic PV cell families have given promising results. The first one is based on the "bulk-heterojunction" concept. Effectively, the power conversion efficiency of organic PV cells has been strongly improved since the introduction of this concept of bulk heterojunction, which consists in bi-continuous interpenetrating network of electron acceptor and donor materials [1,2]. Power efficiency of 6% has been reported for such polymer cells [3]. The second organic PV cell family consists in multi-heterojunction devices based on superposed thin films achieved by sublimation of small organic molecules in vacuum. With these PV cells, power efficiency of 5.8% has been reported [4]. Such PV cells are based on electron donor/electron acceptor junction. Often used bi-layer thin film heterojunction in such cells is copper phthalocyanine/fullerene ($CuPc/C_{60}$). Due to the small absorption efficiency of C_{60} in the visible region, the spectral absorption range of such bi-layer is limited. Therefore, even if C_{60} is very efficient as electron acceptor, it is interesting to probe other electron acceptors. In devices based on electron donor/acceptor organic heterojunction, the theoretically obtainable Voc is given by the difference between the highest occupied molecular orbital of the electron donor ($HOMO_D$) and the lowest unoccupied molecular orbital of the electron acceptor ($LUMO_A$) that is to say $\Delta(HOMO_D - LUMO_A)$ [5]. The

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* Corresponding author. Tel.: +33 2 51 12 55 30; fax: +33 2 51 12 55 28.
E-mail address: jean-christian.bernede@univ-nantes.fr (J.C. Bernède).

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nature of further limitations of the open circuit voltage Voc is still under discussion. For example, in the case of bulk heterojunctions, some Fermi level pinning between organic materials and metals via surface charges has been proposed [6].

Therefore, in the present work, we have probed electron acceptors with different absorption range and we have systematically studied the effect of the encapsulation process of the cells on the open circuit voltage of the PV cells. The small molecular photovoltaic cells with structure "glass/transparent conductive oxide/electron donor/electron acceptor/aluminium/protective layer", with or without interface layer between the electrodes and the organic materials, have been probed. The transparent conductive oxide used was indium tin oxide (ITO), the donor was either copper phthalocyanine (CuPc) or zinc phthalocyanine (ZnPc), the acceptor was fullerene (C_{60}), perylene-3,4,9,10-tetracarboxylic dianhydride (PTCDA), 1,4-diaminoanthraquinone (1,4-DAAQ) or *N*,*N*′-diheptyl-3,4,9,10-tetracarboxylicdiimide (PTCDI-C7). The protective coating layer (P) has been called P_I when cells were immediately coated after the aluminium deposition and P_A when the vacuum was broken 5 min before its deposition in vacuum. This coating layer is an amorphous selenium layer.

It is shown that Voc depends not only on $\Delta(\mathrm{HOMO_D} - \mathrm{LUMO_A})$ but also on the encapsulation process.

2. Experimental

The present PV cells, glass/transparent conductive oxide/donor/acceptor/aluminium/protective layer, were deposited onto pre-cleaned glass substrates coated with transparent conductive oxide (TCO). The TCO electrode was a layer of indium tin oxide (ITO) on a 30 mm by 30 mm glass substrate. ITO substrates were supplied from SOLEMS. The whole surface glass substrates were covered, therefore, some ITO must be removed to obtain the electrode. After masking a broad band of ITO (30 mm × 20 mm), the bare ITO was etched by using Zn + HCl as etchant [7].

Then the substrates were cleaned by using the H_2O_2 treatment following a process described by Osada et al. [8], which corresponds to the first solution (SC1) of the RCA process first described by Kern and Puotinen [9]. The substrates were treated with a 80 °C $H_2O/H_2O_2(30\%)/NH_4OH$ (25%) solution (5:1:1 vol. parts) for 20 min, followed by rinsing with boiling distilled H_2O for 5 min. The use of boiling water was proven to be helpful to obtain impurity free surfaces. The substrates were dried with an argon flow and then loaded into a vacuum chamber (10^{-4} Pa).

Since the goal of this work was to study the effect of the LUMO value of the acceptor and of the encapsulation process on the open circuit voltage (Voc) value, we have used simple structures. After cleaning the ITO coated glass, it has been immediately introduced in the vacuum deposition apparatus without any PEDOT:PSS buffer layer at the interface ITO/electron donor [10].

The organic materials have been provided by Aldrich, except PTCDI-C7, which has been synthesized in the "Laboratorio de Polimeros" of the "Pontificia Universidad Catolica de Chile". They were used without any purification. However, it has been shown, when using the same charge in the evaporation crucible, there is an "autopurification" of the products after around five thin film depositions [11].

Therefore, the results presented here correspond to the cells obtained after, at least, five evaporation cycles of the organic products. CuPc, ZnPc, C_{60}, PTCDA, PTCDI-C7 and 1,4-DAAQ have been deposited in vacuum by sublimation.

The chemical structures of the different organic molecules used are shown in Fig. 1.

The thin film deposition rates and thickness were estimated in situ with a quartz monitor.

The deposition rate was 0.05 nm/s for all the organic molecules and 10 nm/s for aluminium.

Aluminium was deposited in the same run without breaking the vacuum, through a mask with 1.5 mm × 6 mm active area. Then, without breaking the vacuum, or after breaking for 5 min the vacuum, an encapsulating layer of amorphous selenium, of about 100 nm thickness, was thermally evaporated, in vacuum, at a rate of 0.1 nm/s. This protective coating layer (P) has been called P_I when the former process was used and P_A when the latter was used. Often, between the acceptor and aluminium, a thin LiF film has also been evaporated.

The thickness of the organic films has been checked by AFM measurements and/or by scanning electron microscopic visualization. (Measurements have been carried out at the IMN-Nantes.)

AFM images on different sites of the films were taken ex situ at atmospheric pressure and room temperature. All measurements have been performed in non-contact mode (Nanoscope III Digital Instruments, Inc.). Classical Si3N4 cantilevers were used (Ultrasharp, AXESSTECH, France). The average force constant and resonance were approximately 14 N/m and 320 kHz, respectively. The cantilever was excited at its resonance frequency.

The surface topography and the cross-section of the films were observed with a field emission scanning electron microscope (SEM, JEOL F-6400). (Measurements have been carried out at the "Centre de Microcaractérisation", Université de Nantes.) Using simple software, the thickness of the films was measured from the cross-section visualization.

The optical measurements were carried out at room temperature using a Carry spectrometer. (Measurements have been carried out at the IMN-Nantes.) The optical density was measured at wavelengths of 2–0.25 μm.

Electrical characterizations were preformed with an automated *I*–*V* tester, in the dark and under 1 sun global AM 1.5 simulated solar illumination.

3. Results and discussion

First of all, the absorbance spectra of the different organic thin films are presented in Fig. 2. It can be seen that all the film spectra cover, at least partly, the solar spectrum. The absorbance spectra of the electron acceptors appear blue shifted relatively to the MPc (M = Cu or Zn) spectra. As a result, the association of MPc with one of these electron acceptors

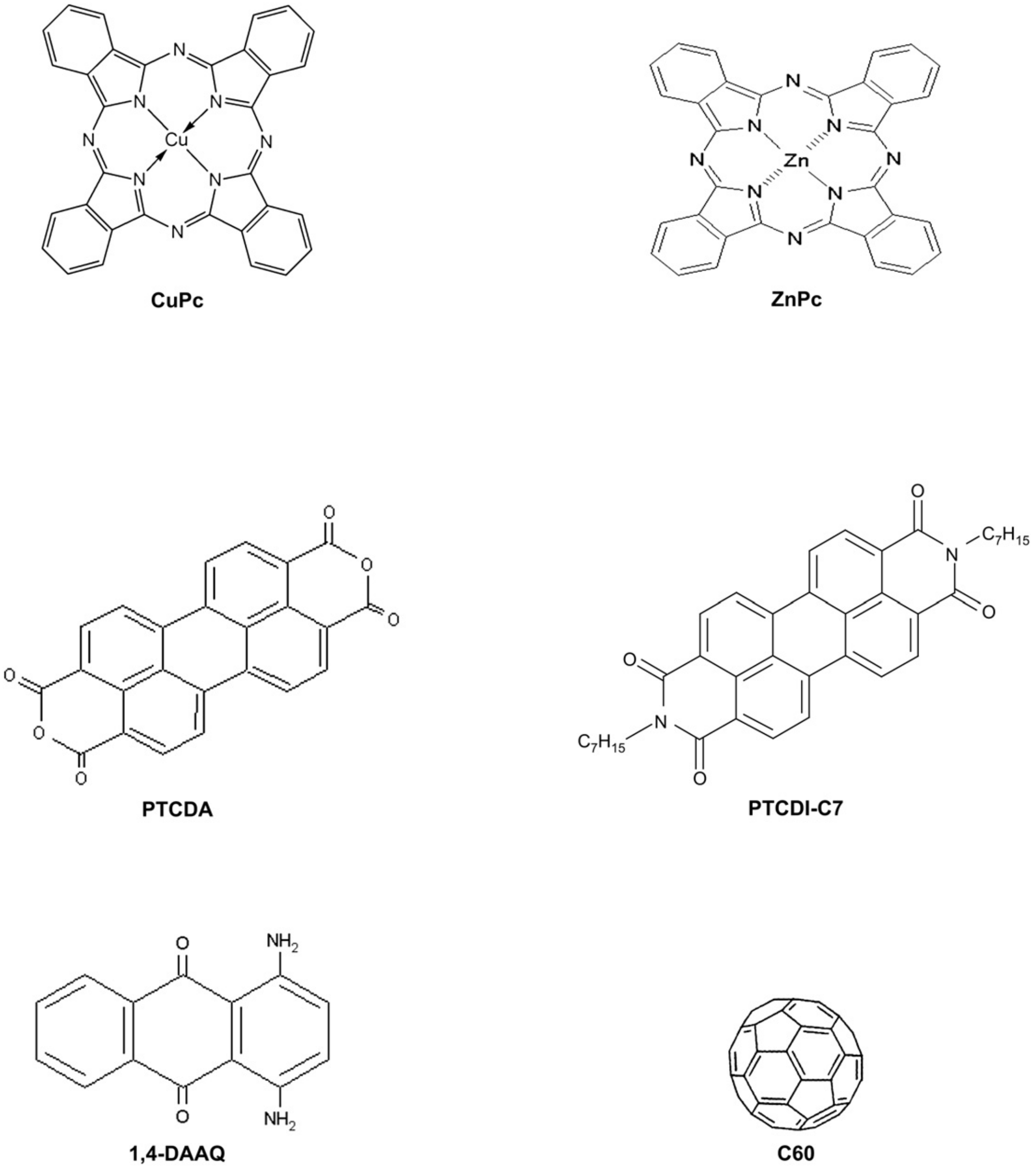

Fig. 1. Molecular structure of the organic materials used in the study.

covers a large part of the visible spectrum, which is of interest for solar cells.

Different authors have postulated the presence of a thin aluminium oxide layer below the aluminium electrode when the cells were submitted to oxygen contamination [12,13]. In order to prevent this effect we have proceeded to PV cell encapsulation. We have earlier studied the effect of an amorphous selenium coating layer on the oxygen contamination of the coated layers [14]. We have shown that, if, at the end of the deposition process of the PV cell and before breaking the vacuum, an amorphous selenium layer was deposited as encapsulating layer, there was no oxygen in the semiconductor and the aluminium oxidation was limited to that occurred during thermal deposition [15]. Nevertheless there was no more oxygen diffusion through the aluminium film, neither aluminium oxide formation at the aluminium/semiconductor interface nor oxygen contamination of the semiconductor. In this article, such encapsulation process has been called P_I.

The efficiency of selenium, as encapsulating layer, has been checked by following the evolution with time of the short circuit current of cells encapsulated (P_I) and not encapsulated. The cells used were typically glass/ITO/CuPc/C_{60}/Al.

The cells with and without selenium capping layer have been achieved in the same run. The short circuit current density, Jsc, variations with time are presented in Table 1. It can be seen that non-encapsulated devices were dead after one day in air, while after 1 h the Jsc was only 70% of the initial short circuit current. The devices coated with 100 nm amorphous selenium layer showed, even if not perfect, significant improvement in stability. After 24 h of room atmosphere exposure of the encapsulated device, there was no decrease of Jsc,

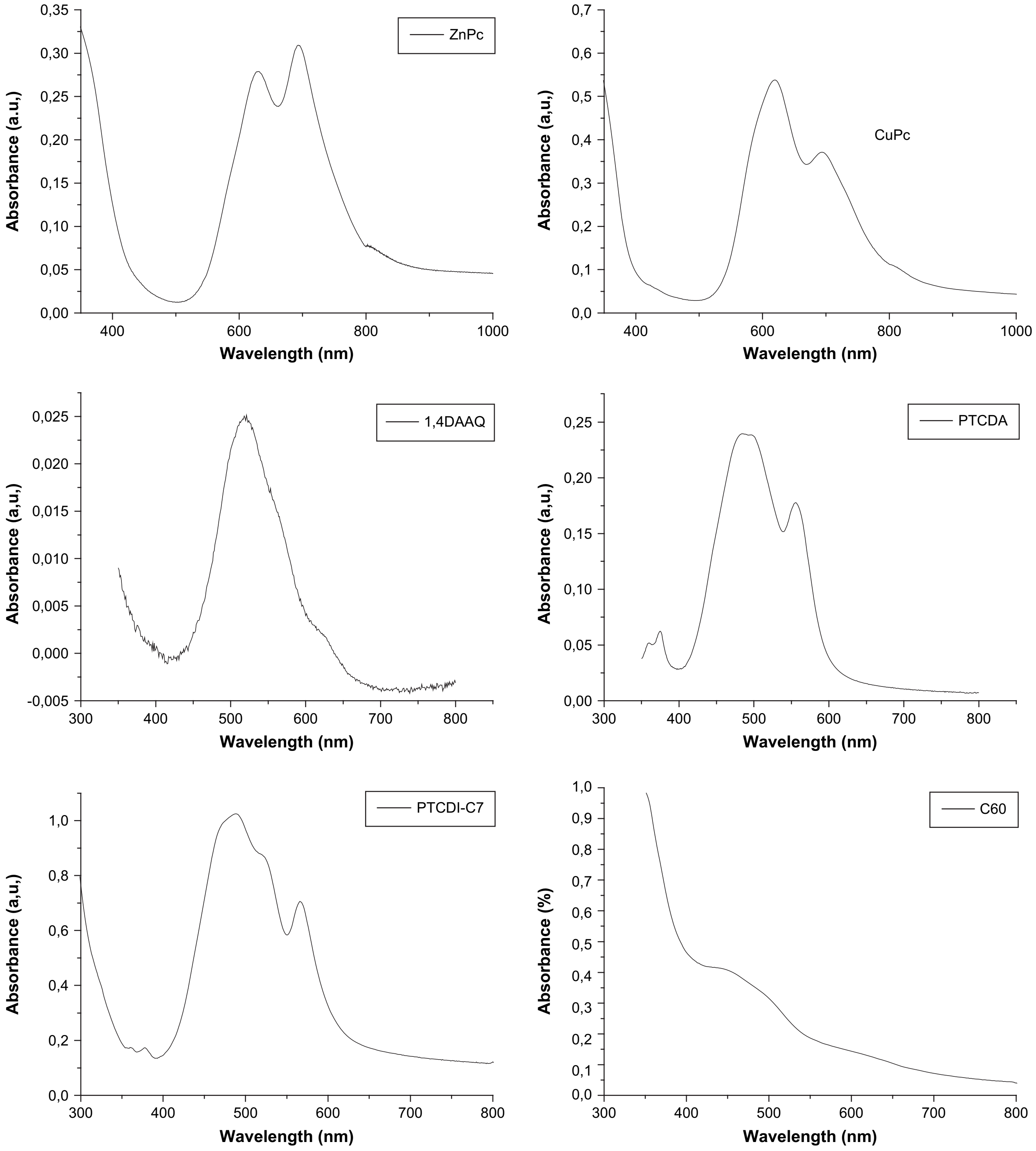

Fig. 2. Absorption spectra of the organic materials used in the study.

after 48 h the Jsc decrease was only 2% and 6.5%, after 72 h. After 144 h Jsc has decreased of about 10%.

At the interface with the other electrode, a thin LiF film (1 nm) was sandwiched between the acceptor and Al layers. Previous works have shown that the presence of an extremely thin film of LiF can boost the performance of an organic electronic component [16].

Therefore, the first PV cells, studied in the present work, had the following sequence glass/ITO/MPc (M = Cu or Zn)/acceptor/LiF/Al/P_I.

The first donor/acceptor couple probed was CuPc/PTCDA. Firstly, the CuPc film thickness has been tuned from 20 nm to 65 nm with PTCDA thickness fixed at 30 nm and the best results have been obtained with 45 nm. Then the effect of

Table 1
Variation with time of the short circuit current density Jsc of glass/ITO/CuPc/C_{60}/Al solar cells coated, or not, by an amorphous selenium layer

Room air exposure time	0	1 h	2 h	1 day	2 days	3 days	6 days
Isc (mA) variation without Se	4.43	3.06	1.91	0.05	0	0	0
Isc (mA) variation with Se	4.81	4.80	4.80	4.80	4.71	4.48	4.15

varying the acceptor film thickness from 25 nm to 65 nm was also studied with a donor thickness fixed at 45 nm. For PTCDA the best result has been obtained with a film thickness of 40 nm. With PTCDI-C7 the optimum thickness was 45 nm. In the case of 1,4-DAAQ the cell performances decreased quickly when the thickness overpass 10 nm, which will be discussed later.

The most striking result obtained for all these PV cell families was their small Voc. Indeed, it can be seen in Table 2, where we have reported the $\Delta(\mathrm{HOMO_D} - \mathrm{LUMO_A})$ and the Voc measured for these cells encapsulated following the P_I process, that these Voc values are smaller than those often previously reported in the literature [17–19]. Moreover, it can be seen that, globally, the Voc value increases with the $\Delta(\mathrm{HOMO_D} - \mathrm{LUMO_A})$ value.

It is known that the donor or acceptor character of each material in an organic couple depends on their relative HOMO and LUMO values, for example, CuPc, which is, as in the present work, usually an electron donor, has been used as electron acceptor with a triphenylamine derivative as donor [20]. Therefore, in a first attempt to improve the efficiency of the cells a thin 1,4-DAAQ film has been introduced between CuPc and PTCDA. Indeed, from the measured HOMO and LUMO values of 1,4-DAAQ estimated by cyclic voltammetry [21] it can be seen in Fig. 3 that 1,4-DAAQ can be seen as an acceptor for CuPc and a donor for PTCDA.

Since the maximum absorbance efficiency of 1,4-DAAQ is situated between those of CuPc and PTCDA (Fig. 3), the superposition of these three layers could improve the solar spectrum covering, which could increase the short circuit current Isc, moreover, 1,4-DAAQ could also improve the Voc value. The optimum thickness of these glass/ITO/CuPc (45 nm)/1,4-DAAQ (×nm)/PTCDA (40 nm)/LiF/Al/P_I has been experimentally determined. It can be seen in Fig. 4 that this optimum thickness is only 8 nm. The absorbance spectra corresponding to the different films with their optimum thickness and that of the superposed films are reported in Fig. 5a and b, respectively. We can see that the absorbance of the 1,4-DAAQ film of 8 nm thickness is quite small, which justifies the small Jsc increase (Table 3). In order to understand such thickness limitation we have visualized a thin 1,4-DAAQ film by AFM microscopy, and in order to compare the result with more classical organic thin film, we show also an image of a typical CuPc organic film. In Fig. 6 we can see that, while the surface of the CuPc is smooth, the 1,4-DAAQ films are highly inhomogeneous. Fibrils are clearly visible, the length of these fibrils increases with the film thickness, which explains the fast degradation of cell performance with increase in thickness. However, it can be seen that this thin layer allows to improve significantly the Voc, which can be attributed to this disordered 1,4-DAAQ film, since it can increase the shunt resistance of the PV cells.

Table 2
$\Delta(\mathrm{HOMO_D} - \mathrm{LUMO_A})$ of different electron donor/electron acceptor couples and open circuit voltage of ITO/PEDOT:PSS/MPc (M = Cu or Zn)/Acceptor/LiF/Al/P_I cells

	Molecule					
	Donor D		Acceptor A			
	ZnPc	CuPc	PTCDA	PTCDI-C7	C_{60}	DAAQ
LUMO (eV)	3.8	3.5	4.8	4.3	4.4	3.9
HOMO (eV)	5.2	5.2	6.9	6.3	6	5.7
Band gap	1.4	1.7	2.1	2	1.6	1.7
$\Delta(\mathrm{HOMO_D} - \mathrm{LUMO_A})$, (eV)			0.4	0.9	0.8	1.3
Voc (V)						
ZnPc/A			0.2	0.25–0.32		0.6
CuPc/A			0.125	0.17–0.19	0.25–0.4	0.5

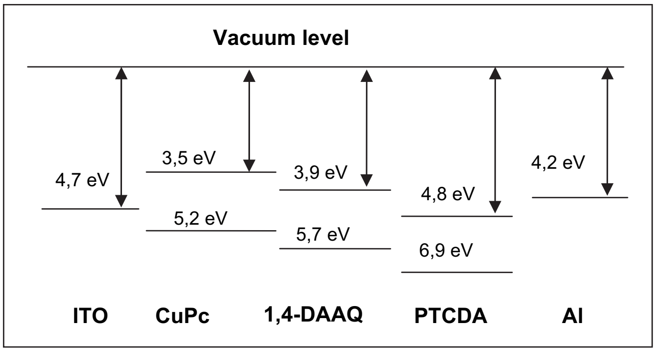

Fig. 3. Band scheme of ITO/CuPc/1,4-DAAQ/PTCDA/Al.

Therefore, a possibility to improve the Voc could be the use of a thin non-conducting layer. Recently it has been proposed that the presence of a thin Al_2O_3 layer at the interface between the organic semiconductor and the aluminium electrode

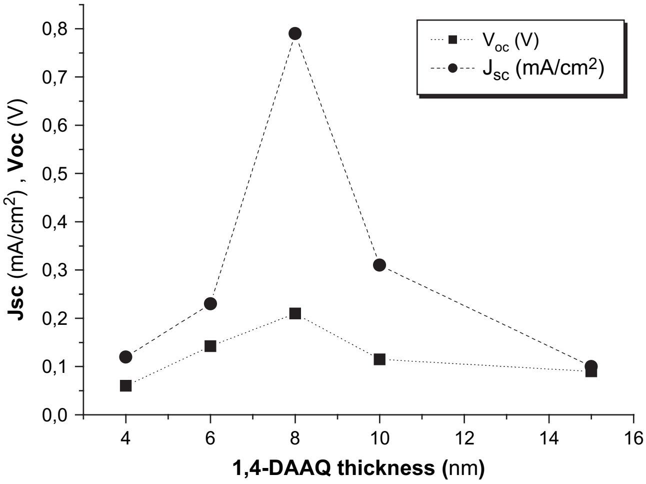

Fig. 4. Optimisation of 1,4-DAAQ thickness.

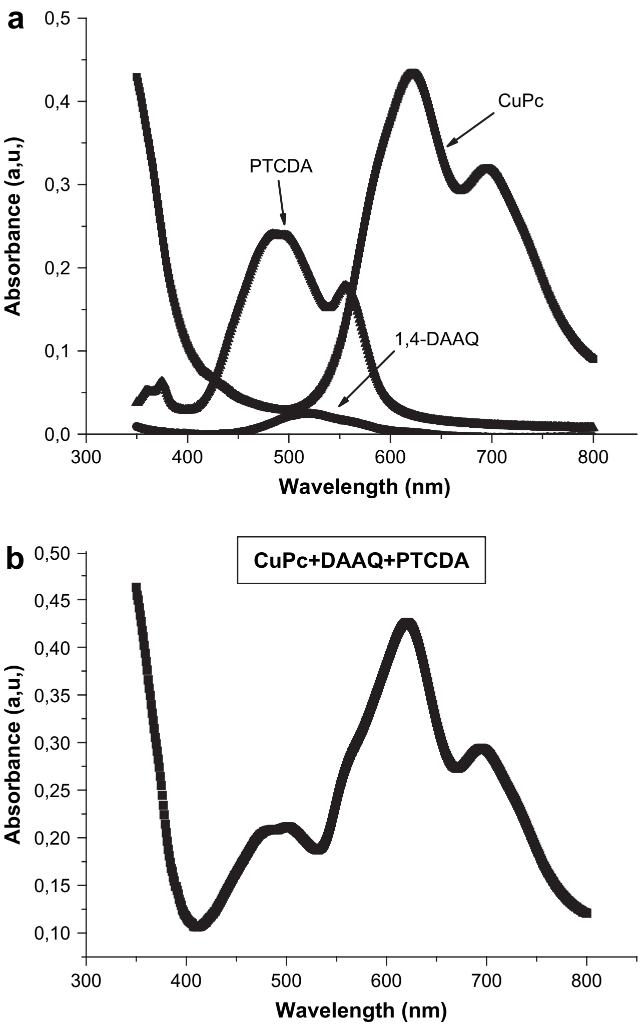

Fig. 5. Absorption spectra of the films used in the cells ITO/PEDOT:PSS/ZnPc/1,4-DAAQ/PTCDA/LiF/Al. (a) Different thin films of the organic constituents. (b) The three layers superposed.

induces higher photovoltage [12,22]. Moreover, we have shown recently in a rapid research letter that in the case of ITO/PEDOT:PSS/CuPc/acceptor/Al/P PV cells, Voc is higher when they are not encapsulated [23].

In order to probe the effect of a thin Al_2O_3 layer at the acceptor/Al interface on the Voc value, we have used another encapsulation process. After the deposition of all the PV cell thin films, we have broken the vacuum for 5 min before a new vacuum cycle for selenium deposition. This encapsulating process has been called P_A. We have earlier studied the effect of an amorphous selenium protective layer on the oxygen contamination of the coated layers [13]. We have shown that after 5 min of air exposure of a Al/semiconductor structure, there was oxygen diffusion along the grain boundaries with a complete oxidation of the first aluminium layers at the Al/semiconductor interface.

Table 3
Photovoltaic parameters of ITO/PEDOT:PSS/ZnPc/PTCDA/LiF/Al/P_I and ITO/PEDOT:PSS/ZnPc/1,4-DAAQ/PTCDA/LiF/Al/P_I cells under illumination of AM 1.5

Device	CuPc/PTCDA	CuPc/1,4-DAAQ/PTCDA
Voc (V)	0.125	0.21
Jsc (mA/cm^2)	0.61	0.79
η (%)	0.085	0.1

The main information can be seen in Table 4. When, before encapsulation of the PV cells, the vacuum was broken (P_A process), the Voc values obtained were systematically higher than the values measured in the case of devices coated without breaking the vacuum (P_I process). The best results have been obtained with C_{60}. The effect of varying the C_{60} thickness is shown in Fig. 7. The highest energy conversion efficiency has been obtained with a thickness of around 40 nm.

It can be seen also in Tables 2 and 4 that, whatever the process used to encapsulate the cells, the variation tendency of the measured Voc value follows the variation of $\Delta(\mathrm{HOMO_D} - \mathrm{LUMO_A})$, as proposed by Brabec et al. [6].

It should be noted that in the present work, the presence of LiF increases mainly the current, while the Voc was only slightly increased. Moreover, with or without LiF, the same variation in the Voc value was encountered when the encapsulation processes change (P_I or P_A). Therefore, the difference in Voc value should be attributed to the presence of a thin Al_2O_3 layer at the organic acceptor/aluminium interface, since as discussed above the P_I process prevents any oxidation of the interface, while the P_A process allows Al_2O_3 formation.

It is well known that another consequence of the admission of oxygen is that it can be responsible for p-type doping of metal-phthalocyanines [28–31]. Therefore, one can think that it is doubtful to attribute the open circuit voltage increase and the efficiency improvement to the presence of the thin alumina film. However, if oxygen is an efficient p-dopant for metal-phthalocyanines, it is also well known that oxygen induces a degradation of the photoelectrical properties of fullerene and perylene derivatives [32,33]. Therefore, it is clear that before phthalocyanine doping, the oxygen should cross the electron acceptor film in our glass/ITO/CuPc/C_{60} (or perylene derivative)/Al devices, which means that this film should be strongly contaminated by oxygen with the well known decrease in cell performances induced by such contamination [32]. Moreover, in order to cut the discussion we have also probed phthalocyanine free samples, that is to say we have used glass/ITO/PTCDI-C7/Al/P devices. PTCDI-C7 has been chosen because it absorbs highly a part of the solar spectrum. Of course, these cells are Schottky diodes with charge separation at the rectifying contact. Therefore, the photoactive region is very thin (near the rectifying contact), and since both positive and negative photoexcited charges travel through the same material, recombination losses are high. These cells have small efficiency and have been used only to study the specific device properties such as current density regimes through I–V characteristics [34–36] or in the present work to justify our hypothesis. Indeed, if the better efficiency of these cells was around 0.012% the open circuit voltage depended strongly on the encapsulation process. In the case of encapsulation without breaking the vacuum Voc = 0.1 V, while when the cells were encapsulated after breaking the vacuum for 5 min Voc = 0.2 V

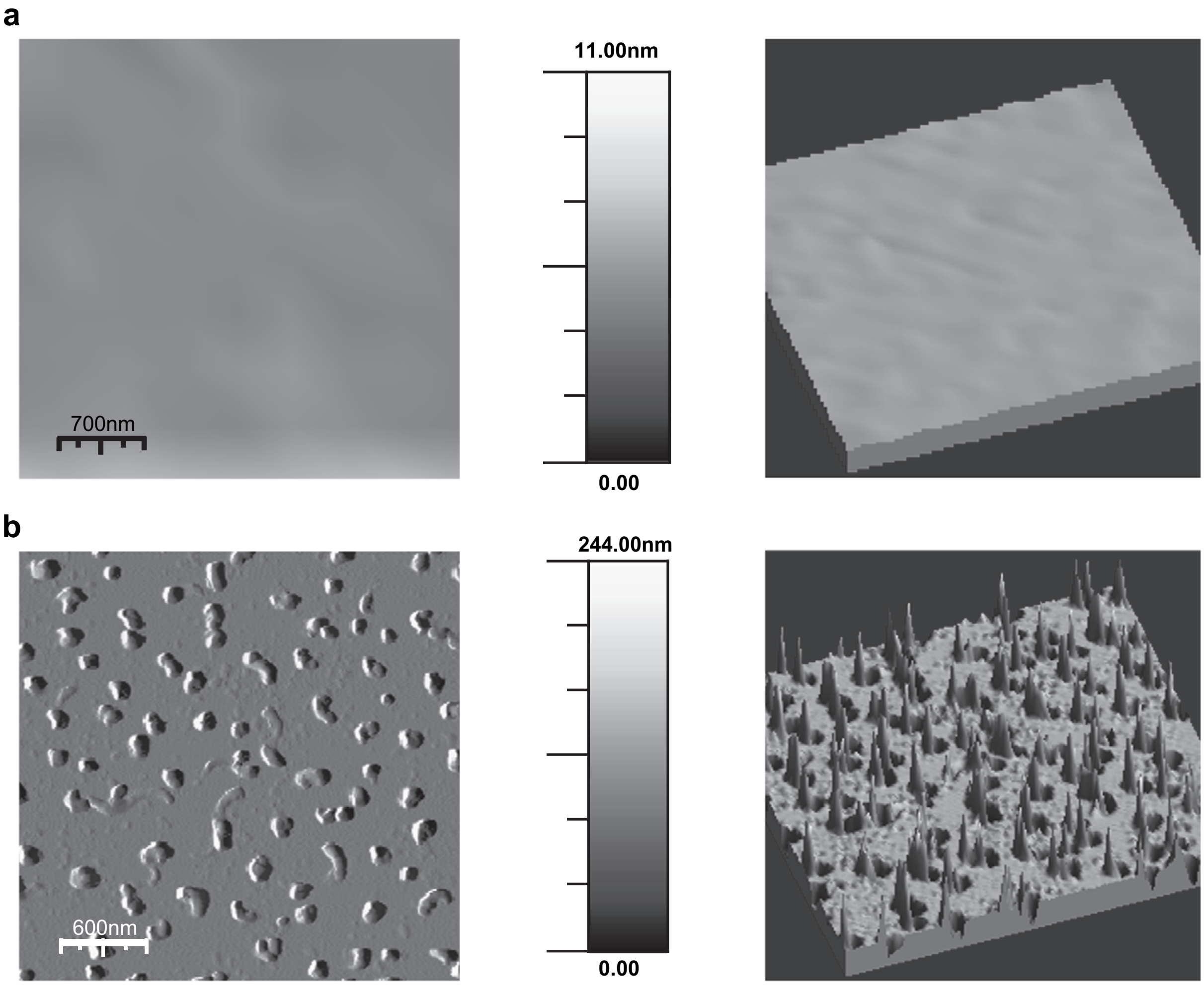

Fig. 6. AFM images of a typical 1,4-DAAQ.

that is to say, with or without phthalocyanine, Voc increases when a thin Al_2O_3 layer is present at the electron acceptor/Al interface.

Therefore, the present study confirms the hypothesis of Singh et al. who have proposed that the presence of a thin Al_2O_3 layer at the interface between the organic semiconductor and the aluminium electrode induces higher photovoltage [12,22].

The influence of the thin Al_2O_3 layer at the interface could be related to an increase of the shunt resistance Rsh, also the presence of this insulating film could prevent any inter-diffusion between the organic material and aluminium.

Indeed, an increase of Rsh improves the Voc value [1]. The slopes at the short circuit point are the inverse values of the shunt resistance of the equivalent circuit scheme of a solar cell [24], therefore, an estimation of the variation of the Rsh values following the encapsulation process can be obtained from the experimental curves. For example, in the case of $CuPc/C_{60}$ based PV cells, Rsh varies from about 90 Ω when the P_I process was used to around 170 Ω when the P_A process

Table 4
Photovoltaic performance data of different solar cells as a function of the encapsulating process

	ZnPc/PTCDA	ZnPc/PTCDI-C7	ZnPc/DAAQ	CuPc/PTCDA	CuPc/DAAQ	CuPc/PTCDI-C7	$CuPc/C_{60}$
$\Delta(HOMO_D - LUMO_A)$ (eV)	0.4	0.9	1.3	0.4	1.3	0.9	0.8
P_I							
Voc (V)	0.2	0.25–0.32	0.6	0.125	0.5	0.17–0.19	0.25–0.4
Jsc		0.60	0.02	0.60	0.03	0.50	4.82
η (%)		0.034	0.0015	0.085	0.002	0.03	0.3
P_A							
Voc (V)	0.6	0.60	0.9–1	0.5	0.9	0.55–0.6	0.5–0.6
Jsc	0.12	0.65	0.02	0.25	0.04	0.90	4.43
η (%)	0.01	0.05	0.002	0.02	0.004	0.075	0.55

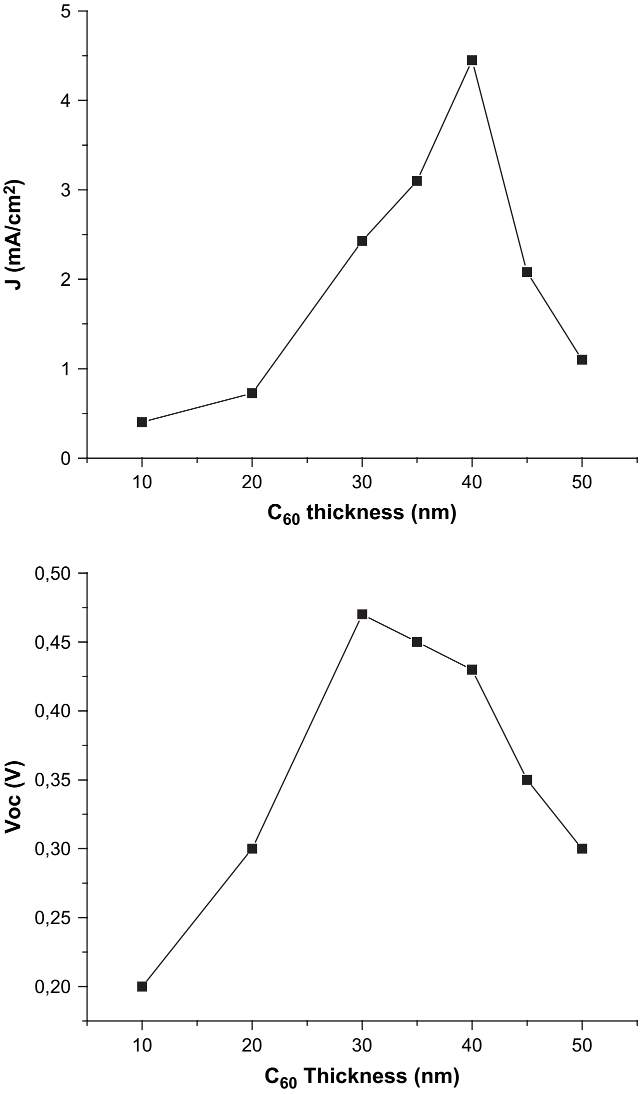

Fig. 7. Optimisation of the C_{60} thickness.

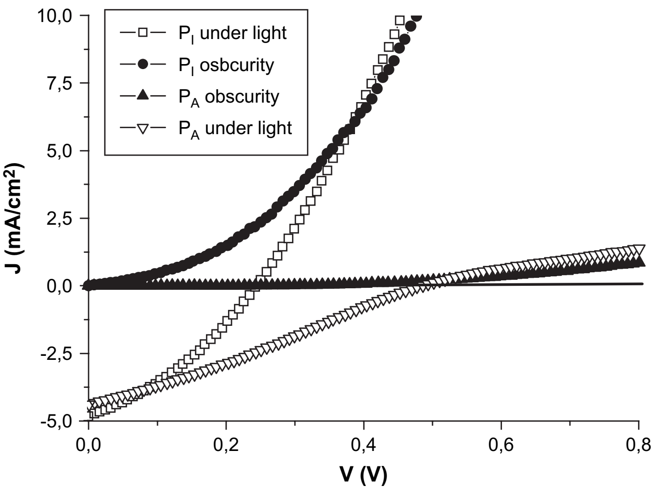

Fig. 8. Typical $J-V$ characteristics of ITO/CuPc/C_{60}/Al/P films with $P = P_I$ or $P = P_A$.

was used (Fig. 8). Therefore, the increase in Voc can be attributed, at least partly, to the increase of the shunt resistance. The principal effect of a thin insulating interfacial layer is the modification of the transmission coefficient for electron tunnelling. However, if this insulating layer is thin enough the photocurrent may not be seriously affected, which should be the case for the natural alumina oxide, even if it can be seen in Fig. 8 that the Isc is slightly smaller when the P_A process was used.

As said above, another contribution can be related to the prohibition or, at least, to the prevention of the pursuit with time of aluminium atoms' diffusion into the organic acceptor after deposition. The negative effect of diffused cathode atoms in C_{60} layers has been already put in evidence [25]. The diffused Al atoms can create states in the energy gap of organic acceptor. There is evidence for a covalent interaction between Al and different organic molecules, while when there is an inter layer at the interface there is no covalent bond formation [26]. It has been shown that Al is found to react at room temperature with oxygen containing anhydride group of the PTCDA molecule. The results of these reactions are interface states in the band gap of PTCDA [27]. More generally, the covalent bond formation induces charge transfer from the aluminium to the organic layer with a dipole formation. All these phenomena can induce some decrease of the open circuit voltage. Therefore, the Al_2O_3 thin film layer acts as a passivation layer at the Al/organic interface, which prevents the organic underlying film from damage and chemical reactivity.

It has been shown in the case of fullerene derivatives [1] that the Voc correlates directly with the acceptor strength, whereas it is insensitive to the work function of the negative electrode because of the Fermi level pinning between fullerenes and metals via surface charges. In our case, it can be proposed that the direct contact of electron acceptor/aluminium induces such Fermi level pinning with small Voc value. However, when the aluminium metallic layer is separated from the organic by a thin oxide layer, there is no more Fermi level pinning.

4. Conclusion

Different electron donor/electron acceptor couples have been probed in heterojunction based organic solar cells. The best results have been obtained with the CuPc/C_{60} couple. The use of different donor/acceptor couples allows to confirm that the open circuit voltage increases with the $\Delta(\mathrm{HOMO_D} - \mathrm{LUMO_A})$ value. Moreover, it is shown that when an in situ encapsulating process is used, which prohibits any oxygen contamination of the electron acceptor/aluminium interface, the values of the open circuit voltage are smaller than the ones encountered into the literature. However, when, before PV cells encapsulation, the cells are kept for 5 min in contact with room air, the measured value of Voc is higher than before, in the range of expected values. This improvement of the cell performances has been attributed to the presence of a small Al_2O_3 thin layer at the organic acceptor/aluminium interface. This layer increases the shunt resistance value and prevents strong chemical reaction between the organic and aluminium.

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