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FOTOGENERACE NÁBOJE V ORGANICKÝCH POLOVODIČÍCH

PHOTOGENERATION OF CHARGE CARRIERS IN ORGANIC SEMICONDUCTORS

AUTOREFERÁT DIZERTAČNÍ PRÁCE
SUMMARY OF DOCTORAL THESES

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ABSTRAKT

S nástupem komerční výroby organických solárních článků roste i zájem o detailní poznání základních elektronových procesů souvisejících s fotogenerací náboje, které umožní dosáhnout vyšší účinnosti fotovoltaické konverze. Tato práce se zabývá studiem fotogenerace nosičů náboje v organických polovodičích, především v π -konjugovaných polymerních materiálech. První část práce popisuje současné poznatky o fotogeneraci nosičů náboje v polymerních solárních člancích. Následuje experimentální a výsledková část, která se věnuje studiu polymerních solárních článků připravených z elektron donorních polymerů MDMO-PPV, Tg-PPV, PCDTBT a PCBTDP a elektron akceptorních derivátů fullerenu PC₆₀BM a PC₇₀BM. Výsledky práce jsou rozděleny do tří hlavních částí: 1) studium přenosu náboje mezi elektron donorním a elektron akceptorním materiálem optickými metodami, 2) studium přenosu náboje mezi elektron donorním a elektron akceptorním materiálem optoelektrickými metodami a 3) vývoj organických solárních článků na flexibilních substrátech, zabývající se z velké části depozičními metodami tenkých vrstev funkčních materiálů.

ABSTRACT

The interest in the detail knowledge about elementary electronic processes during photogeneration of charge carriers, which allow achieving higher efficiency of organic solar cells, grows with advent of the commercial organic solar cells production. The thesis is focused on study of photogeneration of charge carriers in organic semiconductors, especially in π -conjugated polymer materials. First part of the thesis summarizes state of the art in studies of photogeneration of charge carriers in polymer solar cells. Subsequent experimental and results part are focused on study of polymeric solar cells prepared from electron donor polymers MDMO-PPV, Tg-PPV, PCDTBT and PCBTDP and electron acceptor derivatives of fullerenes PC₆₀BM and PC₇₀BM. Results of the thesis are divided in tree main parts: 1) study of charge transfer between electron donor and electron acceptor materials by optical methods, 2) study of charge transfer between electron donor and electron acceptor materials by optoelectrical methods and 3) development of organic solar cells on flexible substrates. The last part is focused largely on deposition methods of active materials thin layer.

KLÍČOVÁ SLOVA

π -konjugované polymery, fullereny, absorpce světla, optoelektrické vlastnosti, fotogenerace nábojů, textilní solární články, organická fotovoltaika, přenos náboje, stavy s přenosem náboje, přenos energie

KEYWORDS

π -conjugated polymers, fullerenes, light absorption, optoelectrical properties, charge photogeneration, textile solar cells, organic photovoltaics, charge transfer, charge transfer states, energy transfer

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1 INTRODUCTION

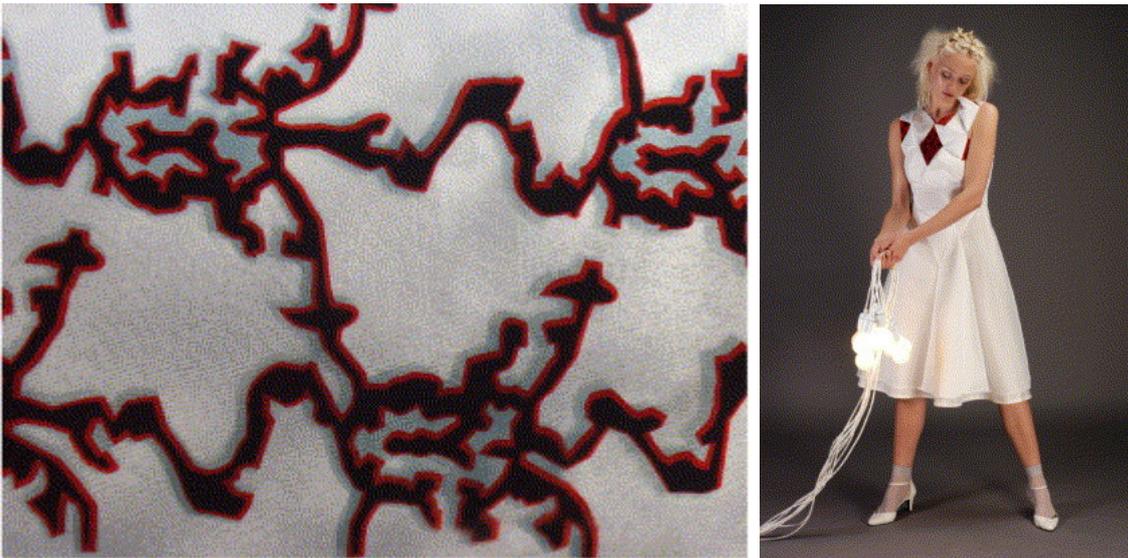


Fig. 1: Illustration photos of design proposals of textile solar cell implementation in clothes. Designed by Tine Hertz and Maria Langberg from Danmarks Designskole (Copenhagen, Denmark) [1]

Photovoltaic solar energy conversion is widely studied as one of the sources of renewable energy. Thin layer organic solar cells are alternative to silicon solar cells, and possess many desirable features, which silicon solar cells cannot offer. Organic solar cells are cheaper to production, easier to dispose at the end of life, can be made mechanically flexible, light transparent and can also work in the interiors thanks to their higher light absorption efficiency. On the other hand disadvantages of organic solar cells are the lower lifetime and lower energy conversion efficiency in comparison with the silicon solar cells.



Fig. 2: Concept car using organic photovoltaic and other organic electronic devices [2].

Nevertheless, the interest in the development of organic solar cells grows with the possibility of their new applications. As one of many examples may serve the solar cells incorporation into textiles as *Fig. 1* and *Fig. 2* illustrate. These can be used for smart clothes, luggage, as energy source elements in houses or transportation vessels' interiors, etc.

Photogeneration efficiency of organic solar cells is currently achieving 10 % [3]. These data are coming as well from academic laboratories as from privately held companies. For example, we can mention companies such as Heliatek, GmbH; Mitsubishi Chemical, Co.; Polyera, Co.; Solamer Energy, Inc and other [4–6], who presented the most significant progress in organic photovoltaic development in the last four years. These facts show on the overall growing interest in commercial fabrication of organic solar cells and give the reasons, why we deal with research of organic solar cells.

1.1 Goals of the Thesis

This thesis is focused on:

- Basic research of photogeneration processes in polymeric solar cells primarily focused on study of charge transfer processes and charge transfer states.
- Applied research, focused on development of OSC on text substrates realized in the framework of FP7 project Dephotex.

1.2 Methodological Approach

Charge transfer and energy transfer processes were investigated by optical and optoelectrical methods to elucidate their contribution to photovoltaic phenomena in polymeric solar cells.

Fluorescence measurements were used for investigation of charge transfer and energy transfer processes. The results of fluorescence quenching experiments and detection of charge transfer states by measurement of near infrared fluorescence are presented.

Charge transfer states and their influence on photovoltaic conversion efficiency were studied by optoelectrical methods: spectrally resolved photocurrent measurements and electroluminescence measurements.

The applied research focused on development of organic photovoltaic devices on textile substrate was realized in international cooperation with 15 European partners in the framework of the FP7 the project DEPHOTEX (NMP Theme – Nanosciences, Nanotechnologies, Materials & New Production Technologies). Dephotex project, research on photovoltaic textiles based on novel fibres was carried out for 3 years (since November 2008 to October 2011). This work involved primarily optimization of thin layers deposition and therefore this part of work is focused largely on comparison of properties

of optoelectrical devices prepared by spin-coating, inkjet print and electrophoretic deposition.

1.3 Working Principle

Organic solar cells are usually consisted of an active layer, two transport layers for both types of charges and finally of electrodes. General scheme of organic photovoltaic cell is shown on *Fig. 3*.

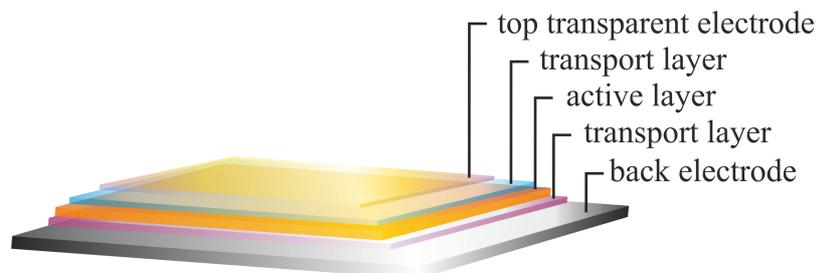
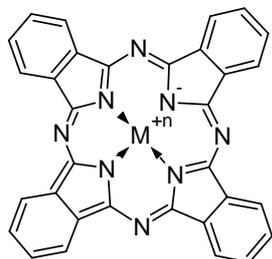


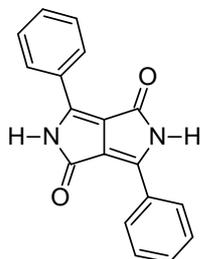
Fig. 3: general structure of organic solar cells which consist of active layer, charge transport layers and electrodes.

The charge carriers are generated in the active layer, and then selectively transported by transport layers to the electrodes. One of the electrodes has to be transparent for light.

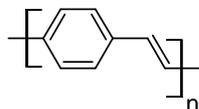
Electron donors



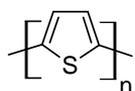
phthalocyanine



diphenyl-
diketopyrrolo-pyrrole

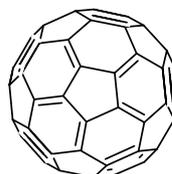


poly(para-phenylenevinylene)



poly(thiophene)

Electron acceptors



fullerene



perylene

Fig. 4: Examples of electron donor and electron acceptor materials.

The active layer is consisting of electron donor and electron acceptor materials. Examples of some materials for organic photovoltaics are depicted in the *Fig. 4*. Both low-molecular and polymeric materials are studied. Low-molecular-weight materials have presented generally higher overall photo-

and chemical stability while polymeric materials have shown better film-forming properties. Structures of active layers are illustrated on Fig. 5.



Fig. 5: Possible arrangement of electron donor and electron acceptor materials in active layer: A) bilayer, B) disordered bulk heterojunction and C) ordered bulk heterojunction.

Photogenerated free charge carriers are transported from the active layer through electron or hole transport layers to the respective electrodes. Transport layers are used for achieving the necessary lower energy barrier between active layer and electrodes. Hole transport layers is typically prepared from PEDOT:PSS (poly(3,4-ethylenedioxythiophene):poly(styrenesulfonate)). Electron transport layers can be created by TiO₂ [7], ZnO [8, 9] and others.

Electrodes are chosen according to their work function, i.e. to their ability to create electrical potential suitable for separation of electron-hole pairs. Frequently used materials are aluminium, silver and gold. One of the electrodes in the organic solar cell needs to be transparent for light. The generally utilized material is ITO (indium tin oxide).

All described layers of organic solar cells have to be well combined and optimized for achievement of high efficiency of photogeneration. This is the generally a complex task and deep research of function principles of organic solar cells is necessary for development of high efficient devices.

1.3.1 Charge Photogeneration

The primary process in organic solar cells is the conversion of incident solar energy to separated charge carriers. The process is complex and consists of series of sequential steps as illustrated Fig. 6.

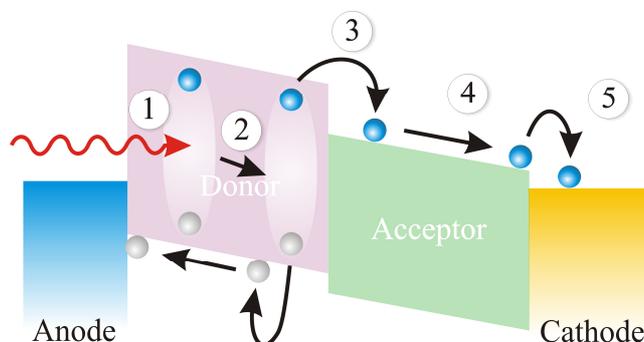


Fig. 6: Scheme of the charge photogeneration and transport processes in organic solar cell, where: 1 – photon absorption by electron donor, 2 – exciton migration to interphase of donor and acceptor, 3 – charge transfer between electron donor and acceptor with separation of free charge carriers, 4 – charge carriers transport to electrodes, 5 – charge carriers collection on electrode.

These steps are:

- 1) absorption of photon with energy $h\nu$ by electron donor D and generation of an exciton D^*



- 2) exciton diffusion to electron donor and electron acceptor interphase $[D^*A]$



- 3) charge transfer (CT) of electron from donor to acceptor resulting in generation of Coulombically bounded electron-hole pair $[D^{*\cdot}A^{\cdot}]$ in CT state.



which has to be dissociated onto free charge carriers – hole on donor $D^{*\cdot}$ and electron on acceptor A^{\cdot} ,



- 4) carriers' transport,
- 5) collection of charge carriers on the electrodes.

1.3.2 Charge Transfer States and Their Experimental Observation

Similar as excitons on donor or acceptor molecule, excited CT states can be deactivated by several pathways. Relaxation pathways of excited CT states are illustrated by *Fig. 7*. Dissociation of CT states in free charge carriers is the most desirable, because contributes to photovoltaic conversion. If CT states cannot be dissociated during their life time, they will be geminately recombined [10]. The loss process is either non-radiative or has a very low emission energy [11–13].

Example of fluorescence emission spectrum of CT states is presented the *Fig. 8* [11], where the energy excited levels S_1 of donor (MDMO-PPV) and of acceptor (PC₆₀BM) molecules and energy level of CT states compared with measured fluorescence spectrum are shown. Emission peak with maximum energy cca 1.3 eV (\approx 955 nm) is the fluorescence of CT states.

Because the direct fluorescence emission of CT states is very weak, the CT states are investigated by methods based on studies of fluorescence quenching. [14–17]. This method is indirect and quenching can be simultaneously caused by other processes, for example by energy transfer [16, 18].

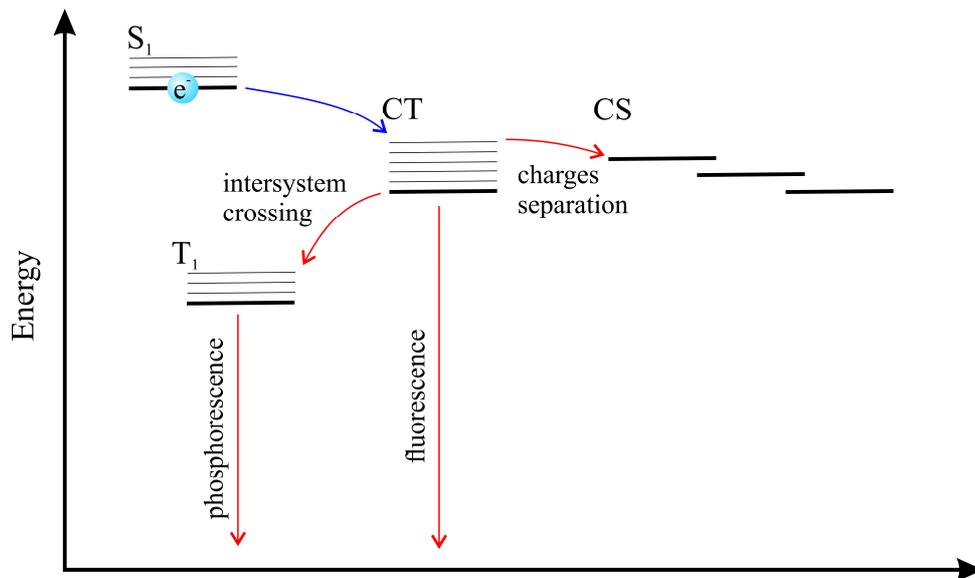


Fig. 7: Scheme of relaxation pathways of excited CT state.

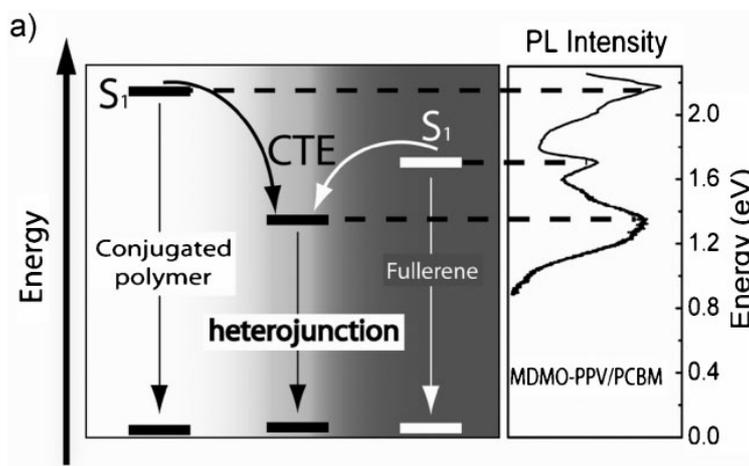


Fig. 8: Fluorescence emission spectrum of MDMO-PPV:PC₆₀BM blend compared with energy levels of excited states of compounds and CT state [11].

Further recombination process can take place through electron transfer from CT state to triplet excited state T₁ of donor or acceptor. Such process is possible to happen if triplet state performs smaller energy than the CT state [12].

Emission spectra of CT states can be also observed by electroluminescence measurements on solar cells devices [19, 20]. Electroluminescence is caused by bimolecular recombination of charge carriers injected in diode devices through electrodes. According to Tvingstedt, K. et al. [20], recombination takes place on the interface of donor and acceptor of heterojunction devices. The observed spectrum is thus created only by emission of CT states and not by pristine material, like in the case of fluorescence spectrum, which is the superposition of all emission states.

Further way to research the CT states is to investigate the CT ground states. In this context it is spoken about charge transfer complexes (CTC). Vandewal

et al. [12] reported observation of CTC ground states band in external quantum efficiency (EQE) spectra. The example of EQE spectra is depicted in the left part of the Fig. 9, where spectra of MDMO-PPV, PC₆₀BM and their blends are shown. The right part of the Fig. 9 shows absorption coefficients spectra which demonstrate very low absorption coefficient of CTC states [12]. Absorption coefficients of CTC were determined using photothermal deflection spectroscopy by Goris et al. [21]. Presented absorption spectra of MDMO-PPV, PC₆₀BM and their blends (see Fig. 9 on right) are very similar to EQE spectra.

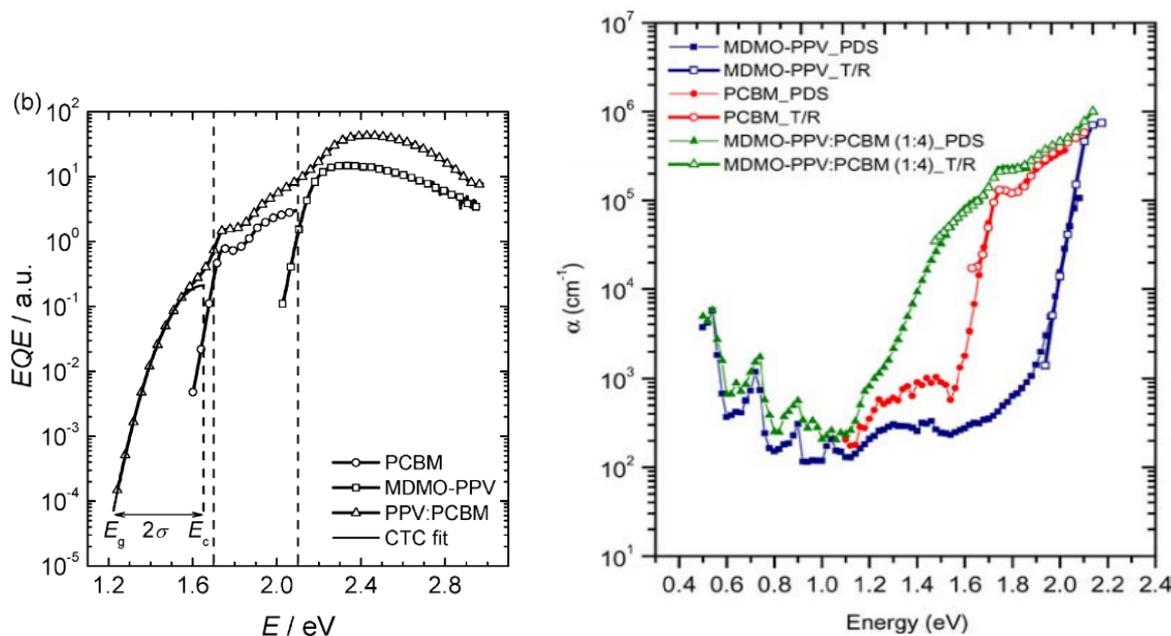


Fig. 9: On left: EQE spectra of MDMO-PPV, PC₆₀BM and MDMO-PPV:PC₆₀BM blend which shows low energy band of CT state [12]. On right: Absorption coefficients spectra of MDMO-PPV, PC₆₀BM and MDMO-PPV:PC₆₀BM blend which shows low energy band of CT state [22].

2 STUDIED MATERIALS

The photogeneration processes were studied in this thesis on these electron donating π -conjugated polymers:

- a) polyaromatic polymers – MDMO-PPV and Tg-PPV,
- b) donor-acceptor alternating copolymers with internal charge transfer states – PCDTBT and PCBTDPP,

For chemical structures see Fig. 11. These polymers were chosen for wide spectrum of optical and electrical properties which allowed the study of photogeneration processes in these polymers in rather general way.

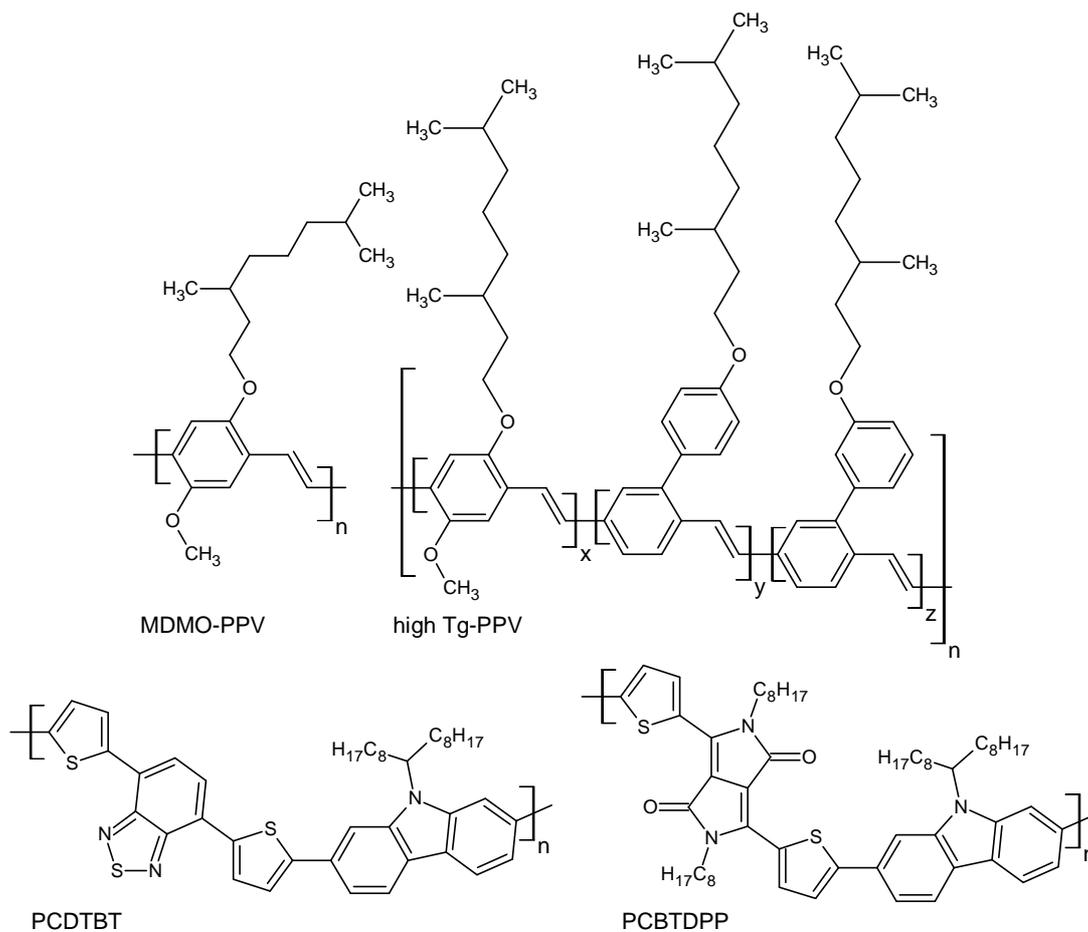


Fig. 10: Studied electron donating polymers.

The fullerenes are dominantly investigated group of electron acceptors; especially soluble derivatives PC₆₀BM ([6,6]-phenyl-C₆₁-butyric-acid-methyl ester) and PC₇₀BM ([6,6]-phenyl-C₇₁-butyric-acid-methyl ester) (see Fig. 11), which were also studied in this work.

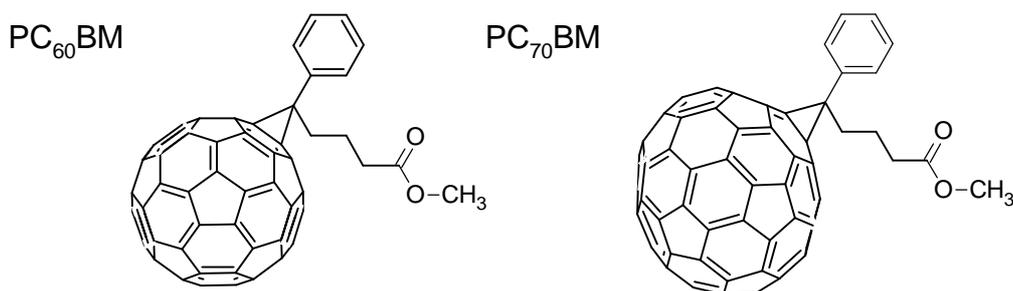


Fig. 11: Dominantly used electron acceptors in organic photovoltaics are soluble derivatives of C₆₀ and C₇₀ fullerenes.

Table 1 summarizes the best published properties of photovoltaic devices prepared from these studied materials. Similar photovoltaic structures were prepared and characterized in this work.

Table 1: Properties of photovoltaic devices prepared on ITO/PEDOT:PSS and with top Al electrode from studied polymers and fullerenes.

Devices	j_{sc} (mA cm⁻²)	V_{oc} (V)	FF (%)	μ (%)	Source
MDMO-PPV:PC ₆₀ BM 1:4	5.25	0.82	61	2.5	Shaheen et al.[23]
MDMO-PPV:PC ₇₀ BM 1:4	7.6	0.77	51	3.0	Wienk et al. [24]
Tg-PPV:PC ₆₀ BM 1:4	3.1	0.82	40	1.0	Vandewal et al. [12]
Tg-PPV:PC ₇₀ BM	-	-	-	-	-
PCDTBT:PC ₆₀ BM 1:4	6.92	0.89	63	3.6	Blouin et al. [25]
PCDTBT:PC ₇₀ BM 1:4	10.3	0.88	63	5.8	Ossila [26]
PCBTDPP:PC ₆₀ BM 1:3	5.2	0.85	37	1.6	Huo et al. [27]
PCBTDPP:PC ₇₀ BM 1:3	8.02	0.76	40	2.45	Jo et al. [28]

3 EXPERIMENTAL PART

This part of the thesis presents the results of experiments focused on studies of photogeneration of charge carriers in polymer semiconductors, as well as the applied research focused on the preparation of flexible polymer solar cells on textile substrates realized in our group within the European FP7 project DEPHOTEX.

These results are presented in three sections:

1. Optical studies of photogeneration processes – present the research of charge transfer and energy transfer between donor and acceptor in solutions and thin films by fluorescence spectrometry.
2. Detection and studies of CT states by optoelectrical methods – OSC devices were characterized by current voltage characteristics, spectrally resolved photocurrent measurements and electroluminescence measurements to assess the influence of charge transfer states on photogeneration efficiency.
3. Textile OSC application – this last section present results of experiments focused on preparation of thin films by different deposition methods: spin-coating, electrophoretic deposition and inkjet printing. Based on these findings the textile organic solar cells were proposed.

3.1 Optical Methods for Study of Photogeneration Processes

Charge transfer and resonance energy transfer processes were investigate by fluorescence spectrometry. In particular, the studies of fluorescence quenching and the analysis of fluorescence emission in near infrared region caused by relaxation of excited CT states are utilized. In order to find suitable donor-acceptor pairs, the several studies based on optical methods were realized, subsequently several parameters of photovoltaic devices prepared from these materials were estimated.

3.1.1 Fluorescence Quenching

The charge transfer and energy transfer processes between polymers and fullerenes were studied in details by fluorescence quenching. Initially, the studies were realized in solution, as the results can be evaluated by standard fluorescence quenching models based on Stern-Volmer equation and Perrin equation.

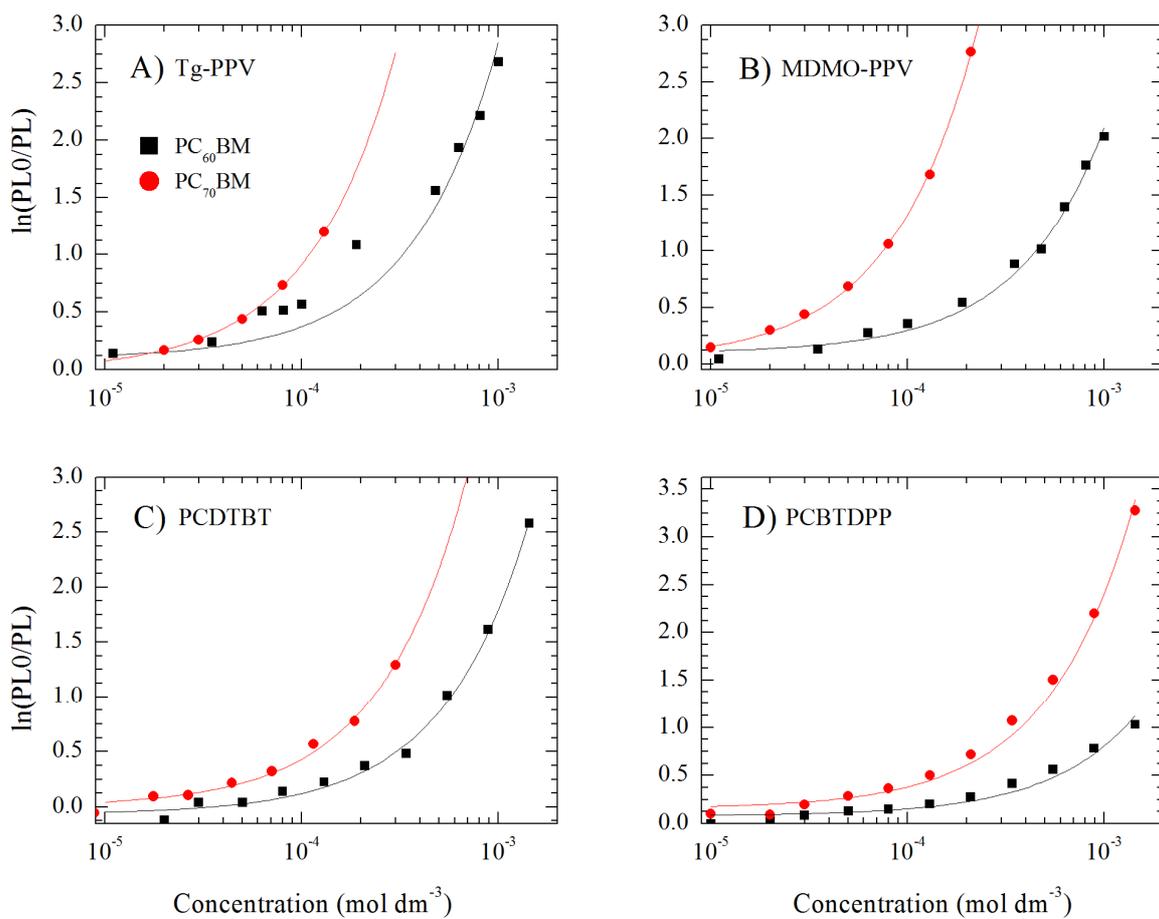


Fig. 12: Perrin plots of relative fluorescence decay of different polymers A) Tg-PPV B) MDMO-PPV C) PCDTBT D) PCBTDPP on concentration of PC₆₀BM (squares) and PC₇₀BM (circles).

Table 2: Gibbs free energy ΔG of the charge transfer between the electron donating polymer and PC₆₀BM and PC₇₀BM electron acceptors, the calculated Förster critical distance R_{RET} , the experimental values of quenching equilibrium constant K calculated from the Perrin plots, and radius R_{eff} of the Perrin action sphere.

Fullerene	Polymer	ΔG (eV)	Ω (mol·dm ⁻³ m ⁻²)	R_{RET} (nm)	K (mol ⁻¹ dm ³)	R_{eff} (nm)
PC ₆₀ BM	MDMO-PPV	-1.28	$1.96 \cdot 10^{-11}$	8.5 ± 0.4	1900 ± 50	9.1 ± 0.1
	Tg-PPV	-1.44	$2.70 \cdot 10^{-11}$	10 ± 1	2380 ± 150	9.8 ± 0.2
	PCDTBT	-0.73	$2.49 \cdot 10^{-11}$	8.7 ± 0.8	1690 ± 50	8.8 ± 0.1
	PCBTDPP	-0.5	$0.86 \cdot 10^{-11}$	7 ± 1	740 ± 50	6.6 ± 0.1
PC ₇₀ BM	MDMO-PPV	-1.2	$1.55 \cdot 10^{-10}$	12 ± 1	6500 ± 600	13.7 ± 0.5
	Tg-PPV	-1.36	$2.20 \cdot 10^{-10}$	14 ± 1	9300 ± 500	15.4 ± 0.5
	PCDTBT	-0.7	$1.40 \cdot 10^{-10}$	12 ± 1	4300 ± 100	12.0 ± 0.1
	PCBTDPP	-0.4	$0.42 \cdot 10^{-10}$	10 ± 1	2400 ± 70	9.8 ± 0.1

Fig. 12 shows plots obtained in this experiment, when fluorescence quenching was described by Perrin model. It was found by data analysis that resonance energy transfer plays a dominant role with respect to fluorescence

quenching under the conditions when π -conjugated polymers and fullerenes are separated at such a distance that direct contact and electron exchange is not possible. This was concluded on the basis of the similarity between the Förster critical distance R_{RET} obtained from the spectral analysis of RET efficiency and the R_{eff} values obtained from the analysis of the Perrin model of static quenching, see *Table 2*. Resonance energy transfer is more pronounced for PC₇₀BM, as it has a higher absorption coefficient compared to PC₆₀BM.

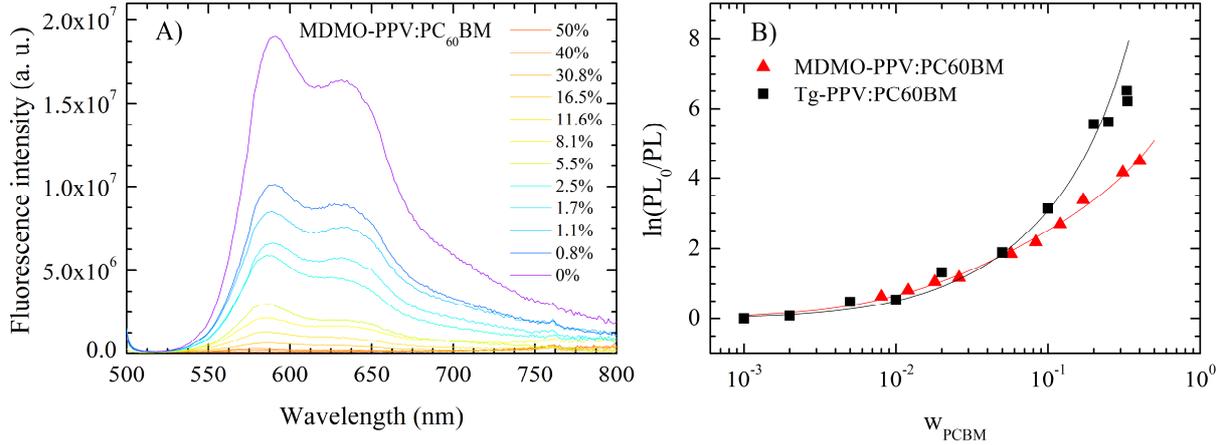


Fig. 13: A) Fluorescence spectra of MDMO-PPV with varying concentration of PC₆₀BM. B) Qualitative evaluation of fluorescence quenching efficiency.

Based on gained results, the studies were extended on thin layers to achieve a deeper insight to the interaction of electron donor and electron acceptor in photovoltaic devices.

Table 3: Parameters describing fluorescence quenching of polymer in thin films.

	MDMO-PPV	Tg-PPV
K_1 (M^{-1})	1.4 ± 0.3	4.2 ± 0.6
R_{eff} (nm)	0.8 ± 0.2	1.2 ± 0.2
R_{RET} (nm)	2.1 ± 0.1	2.0 ± 0.1
K_2	74 ± 6	90 ± 20
τ_0 (s)	$(2.8 \pm 0.3) \cdot 10^{-10}$	$(3.7 \pm 0.6) \cdot 10^{-10}$
k_{diff} (s^{-1})	$(2.7 \pm 0.4) \cdot 10^{11}$	$(2.5 \pm 0.7) \cdot 10^{11}$
L (nm)	6 ± 1^a	6 ± 1^a
R_C (nm)	0.5 ± 0.2	0.7 ± 0.2

a) Diffusion length of MDMO-PPV exciton determined by Scully et al.[29].

It was shown that experimentally obtained dependencies of fluorescence intensities on fullerenes concentration, see *Fig. 13*, can be evaluated by standard models for fluorescence quenching (Stern-Volmer and Perrin equations), which was modified with respect to the expression of fullerene amount in thin layers by mass fraction w . The obtained quenching constants were deeply analyzed in order to identify the presence of charge

transfer and energy transfer. Results are summarized in *Table 3*. Thus, the studies allows to obtain information (range of dynamic and static quenching) which can help to optimize active layer morphology (materials domain size) for achievement of high photogeneration efficiency of charge carries in polymeric photovoltaic devices.

The results of fluorescence quenching experiments in solutions have been published in *Chemical Physics Letters* [30], the results of the thin film studies are prepared for publication in the same journal.

3.1.2 Emission of CT States

The fluorescence of CT states was observed on thin films of the studied blends. Despite the co-emission of fullerenes and CT states, the positions of CT states emission maxima were determined based on spectra analysis. More straightforward results were obtained for PCBTDP:PCBM thin layers, because polymer PCBTDP is non-fluorescent in thin films and have smaller optical band gap than fullerenes, which allow excitation of polymer without simultaneous excitation of fullerenes.

3.1.3 Determination of Photovoltaic Devices Properties

The modelling of photovoltaic parameters can be used for estimation of suitability of new donor or acceptor materials for application in photovoltaic devices. Therefore utilization of optical spectra of studied polymer:fullerenes blends for estimation of photovoltaic devices parameters, in particular open circuit voltage V_{OC} and short current density j_{SC} , were investigated. Values of V_{OC} were determined from fluorescence spectra of CT states and values of j_{SC} were calculated from absorption spectra of thin films by application of Shockley-Queisser limit model. These parameters are usually estimated from molecular energy levels (energy of band gap, HOMO and LUMO levels) of used materials [31]. Both approaches to modelling of photovoltaic devices parameters were utilized and obtained parameters were compared with parameters of real devices, see *Table 4*.

The calculated parameters from spectral data showed better agreement with real devices than the results calculated from molecular orbital energy levels. Values of j_{SC} are very poorly predictable without determination of losses during charge carrier transport. The study of transport phenomenon was outside the scope of this work. The values of V_{OC} may be estimated relatively accurately from the energies of CT emission. It emphasizes the importance of research of CT states.

Table 4: Comparison of estimated values of short current density j_{SC} and open circuit voltage V_{OC} estimated from optical parameters with values determined on real photovoltaic devices.

Device	According to energy levels		According to optical spectra		Literature data*	
	$j_{sc_{lim}}$ (mA/cm ²)	$V_{oc_{lim}}$ (V)	j_{sc_a} (mA/cm ²)	$V_{oc_{teofl}}$ (V)	j_{sc} (mA/cm ²)	V_{oc} (V)
MDMO-PPV:PC ₆₀ BM 1:4	13	0.82	12	0.87	5.3	0.82
MDMO-PPV:PC ₇₀ BM 1:4	13	0.90	20	0.89	7.6	0.77
Tg-PPV:PC ₆₀ BM 1:4	10	0.82	10	0.86	3.1	0.82
Tg-PPV:PC ₇₀ BM 1:4	10	0.90	17			
PCDTBT:PC ₆₀ BM 1:4	18	1.07	13	0.81	6.9	0.89
PCDTBT:PC ₇₀ BM 1:4	18	1.15	15	0.82	10.3	0.88
PCBTDP:PC ₆₀ BM 1:3	23	1.12	12	0.79	5.2	0.85
PCBTDP:PC ₇₀ BM 1:3	23	1.20	14	0.81	8.0	0.76

*for reference see Table 1.

3.2 Detection and Study of Charge Transfer States by Optoelectrical Methods

The studied polymer:fullerenes blends were used for preparation of organic photovoltaic devices based on bulk heterojunction. The current-voltage characteristic and spectrally resolved photocurrents measurements were evaluated with respect to the parameters describing efficiency of photovoltaic conversion. Subsequently, optoelectrical experiments focused on detection of CT states based on spectrally resolved photocurrent and electroluminescence measurements were realized. Finally, these results were evaluated in order to determine the influence of CT states on photovoltaic parameters.

3.2.1 Photovoltaic Efficiency

Prepared photovoltaic devices were characterized by current voltage measurement under illumination by solar simulator and photovoltaic conversion efficiency was evaluated. Obtained parameters are summarized in Table 5.

Table 5: The achieved photovoltaic parameters of prepared devices and thickness (d) of their active layers.

Device	d (nm)	j_{sc} (mA/cm ²)	V_{oc} (V)	FF (%)	η (%)
MDMO-PPV	94 ± 5	(8 ± 2) · 10 ⁻³	1.03 ± 0.02	26 ± 1	(4 ± 1) · 10 ⁻³
MDMO-PPV:PC ₆₀ BM 1:4	130 ± 20	2.04 ± 0.08	0.76 ± 0.03	42 ± 2	0.65 ± 0.07
MDMO-PPV:PC ₇₀ BM 1:4	260 ± 10	1.81 ± 0.09	0.85 ± 0.01	40 ± 4	0.65 ± 0.07
Tg-PPV	420 ± 5	(3.1 ± 0.1) · 10 ⁻⁴	1.05 ± 0.05	29 ± 1	(1.1 ± 0.5) · 10 ⁻⁴
Tg-PPV:PC ₆₀ BM 1:4	140 ± 20	2.2 ± 0.1	0.68 ± 0.06	30 ± 2	0.46 ± 0.09
Tg-PPV:PC ₇₀ BM 1:4	123 ± 5	3.9 ± 0.2	0.74 ± 0.02	44 ± 2	1.3 ± 0.1
PCDTBT	63 ± 5	(31 ± 2) · 10 ⁻³	0.92 ± 0.03	24.7 ± 0.3	(7 ± 1) · 10 ⁻³
PCDTBT:PC ₆₀ BM 1:4	53 ± 5	7.8 ± 0.2	0.75 ± 0.01	45 ± 2	2.6 ± 0.1
PCDTBT:PC ₇₀ BM 1:4	52 ± 5	11.0 ± 0.5	0.74 ± 0.01	46 ± 1	3.7 ± 0.1
PCBTDPP	40 ± 10	(70 ± 1) · 10 ⁻³	0.82 ± 0.01	26 ± 1	0.02 ± 0.01
PCBTDPP:PC ₆₀ BM 1:3	62 ± 5	4.4 ± 0.4	0.71 ± 0.02	37 ± 1	1.2 ± 0.1
PCBTDPP:PC ₇₀ BM 1:3	86 ± 5	6.1 ± 0.2	0.67 ± 0.02	37 ± 1	1.53 ± 0.06
PC ₆₀ BM	30 ± 10	0.02 ± 0.01	0.13 ± 0.01	32 ± 1	(0.9 ± 0.2) · 10 ⁻⁴
PC ₇₀ BM	40 ± 10	0.26 ± 0.01	0.59 ± 0.01	36 ± 2	0.05 ± 0.02

3.2.2 External Quantum Efficiency Spectra

In order to investigate spectral response of photovoltaic devices, all prepared solar cells were characterized by measurements of the photocurrent spectra at zero voltage. Obtained photocurrent spectra were recalculated in terms of external quantum efficiency (EQE), which is defined as ratio between number of generated charges N_e and number of incident photons N_{ph} :

$$EQE = \int \frac{N_e(\lambda)}{N_{ph}(\lambda)} d\lambda. \quad (5)$$

Examples of EQE spectra of devices prepared from polymer PCDTBT are on Fig. 14.

The utilization of spectrally resolved photocurrent measurement to detection of CT states absorption bands was investigated. However, the detection limits of our apparatus used for spectrally resolved photocurrent experiments didn't allow observation of these CT bands.

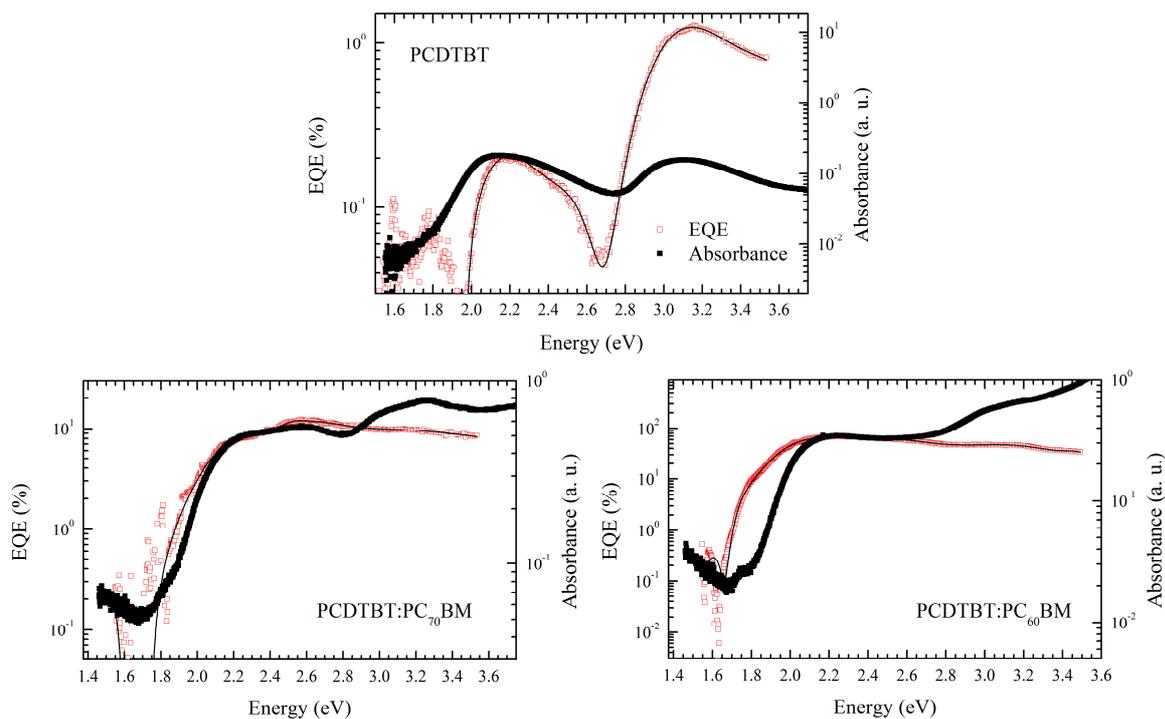


Fig. 14: EQE spectra and absorption spectra of PCDTBT and then of photovoltaic devices PCDTBT:PC₆₀BM 1:4 and PCDTBT:PC₇₀BM 1:4.

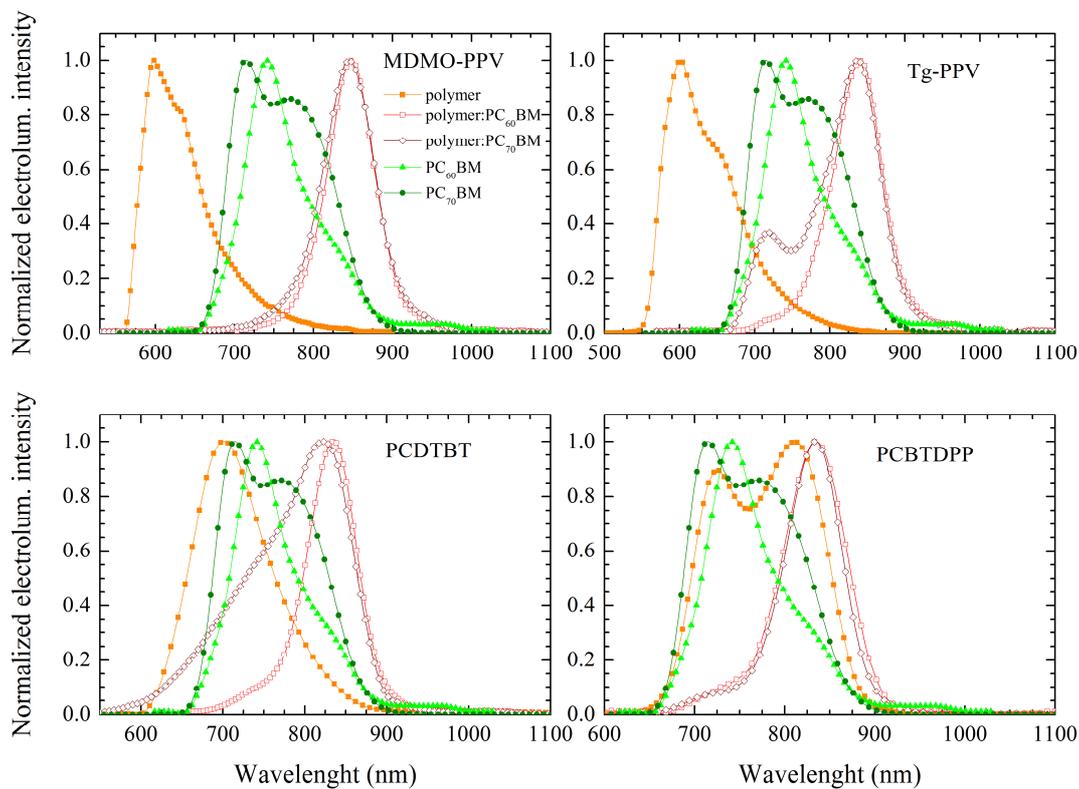


Fig. 15: Smoothed electroluminescence spectra of pristine materials and their blends (orange curves – polymers, green curves – fullerenes, red curves – blends).

3.2.3 Studies of Electroluminescence of Photovoltaic Devices

In order to detect the bimolecular recombination, the intensity of electroluminescence was measured simultaneously with current voltage characterization of photovoltaic devices in dark. Simultaneously electroluminescence spectra were detected, see Fig. 15.

Electroluminescence spectra of pristine materials were in good agreement with fluorescence spectra, while spectra of blends showed unanimously maximum emission at 1.48 eV independently on used donor and acceptor material. It was decided that electroluminescence rather came from triplet states of fullerenes although this result was unexpected with regard to previous published results by other authors [19, 20]. This suggests that there are more conditions driving the origin of electroluminescence in photovoltaic devices, for example the layer morphology. Investigation of influence of bulk heterojunction morphology of active layer on energy levels of CT states is beyond the aims of this thesis; however interesting topic for further work.

3.2.4 Reciprocity between External Quantum Efficiency and Electroluminescence Spectra

The ground CT states according to literature [19] can affect photogeneration efficiency. The aim of this chapter is to identify the CT absorption band by computational procedure based on recalculation of emission spectra. For verification of this fact, CT absorption bands were calculated from electroluminescence spectra according to equation [32]:

$$EQE(E) = \frac{EL(E)}{\phi_{bb}(E)} \frac{1}{\exp\left(\frac{qV_{bi}}{kT}\right) - 1}, \quad (6)$$

where ϕ_{bb} is spectral photon density of black body and V_{bi} stands for built-in voltage determined from difference of LUMO energy level of acceptor and HOMO energy level of donor [33].

The contribution of light absorption in near IR region was verified through calculation of j_{SCrad} and V_{OCrad} from these extended EQE spectra.

Values of short current density j_{SCrad} were obtained by integration of EQE spectra and solar emission intensity spectra (where the AM 1.5 spectrum with integral intensity 1000 W/m² was used):

$$j_{SCrad} = q \int EQE(E) \phi_{Sun}(E) dE \quad (7)$$

Open circuit voltage V_{OCrad} were determined according to following term:

$$V_{\text{OCrad}} = \frac{kT}{q} \ln \left(\frac{j_{\text{SC}}}{j_{\text{rad0}}} + 1 \right), \quad (8)$$

where j_{rad0} is the saturation current density for radiative recombination of the [32, 55].

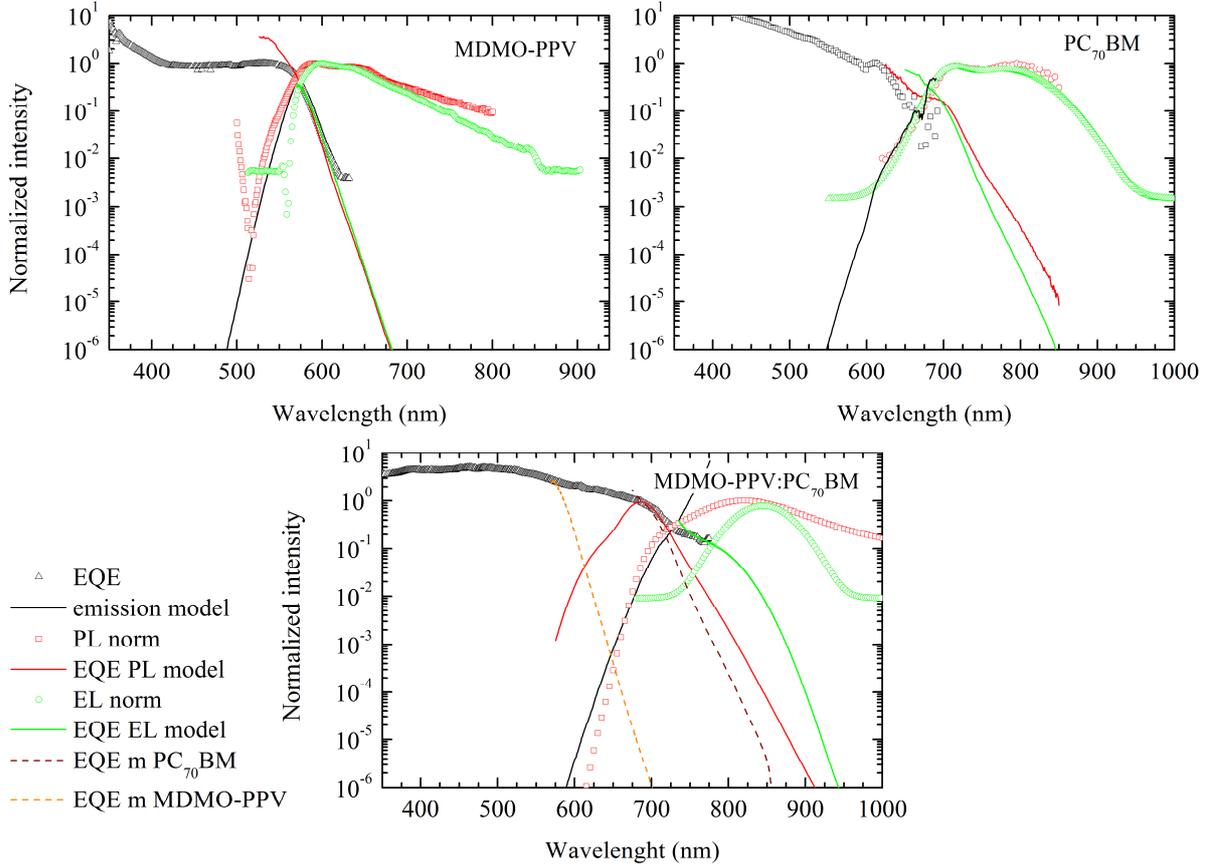


Fig. 16: Normalized EQE, fluorescence and electroluminescence spectra with calculated edges according to Eq. (6) for the reciprocity of EQE and electroluminescence spectra.

$j_{\text{SCrad EQE}}$ and $V_{\text{OCrad EQE}}$ were calculated from experimentally obtained EQE spectra as well as from EQE spectra with near infrared absorption tail calculated from either electroluminescence (parameters marked $V_{\text{OCrad EQE+EL}}$, $j_{\text{SCrad EQE+EL}}$) or from photoluminescence (parameters marked $V_{\text{OCrad EQE+PL}}$, $j_{\text{SCrad EQE+PL}}$) experiments. The results are presented on Fig. 17, Fig. 18. The calculated parameters are compared with parameters calculated from current voltage measurements.

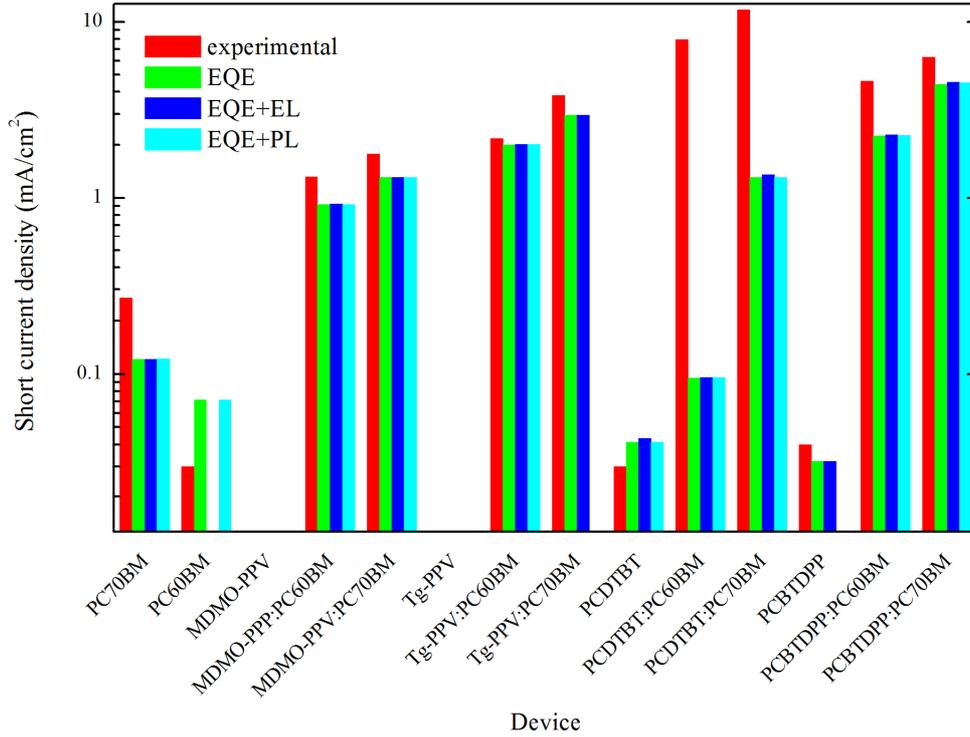


Fig. 17: The comparison of short current density j_{sc} values obtained from current voltage characteristics (red), by integration of EQE spectra (green) and EQE spectra with IR tail calculated from electroluminescence data (dark blue) and from photoluminescence data (light blue).

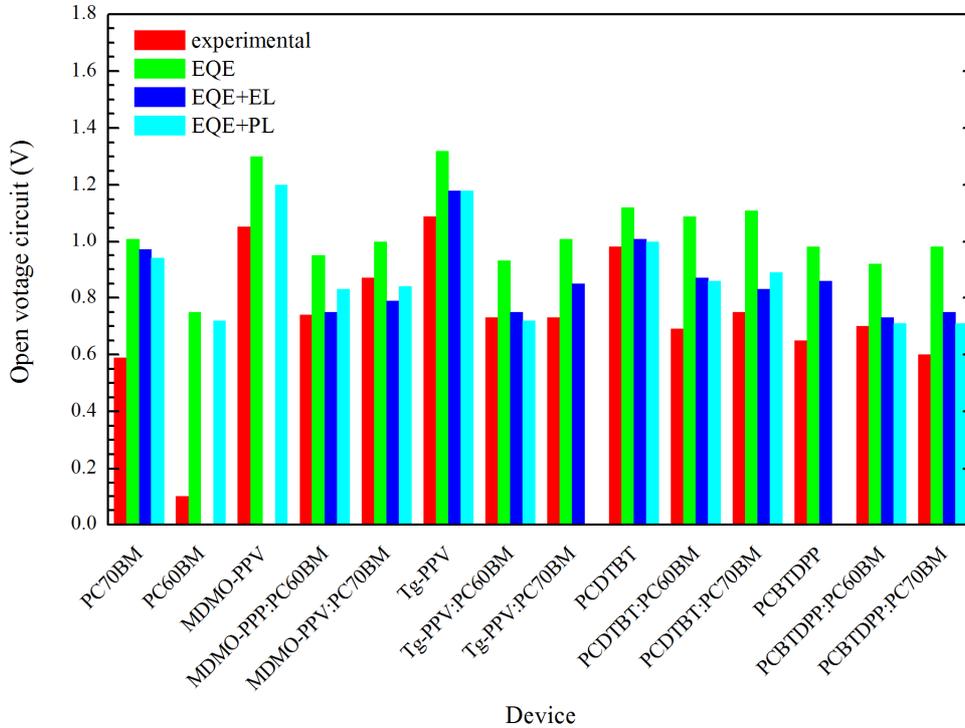


Fig. 18: The comparison of open circuit voltage V_{oc} values obtained from current voltage characteristics (red), by calculation from EQE spectra obtained from photocurrent spectra measurements (green), from EQE spectra with IR tail calculated from electroluminescence data (blue) and from EQE spectra with IR tail calculated from photoluminescence data (cyan).

The calculations showed that emission spectra can be used for completion of missing parts of EQE spectra, which are experimentally inaccessible due to detection limits. Simultaneously, significant influence of CT states on the photogeneration efficiency was confirmed, when it was shown that low band gap of CT states reduces the V_{OC} value of photovoltaic devices.

3.2.5 The Comparison of Open Circuit Voltage Determined from Optical and Optoelectrical Characteristics

The demonstrated optical and optoelectrical experiments were focused on the detection of CT states and on contribution of these states to the parameters of photovoltaic devices. It was turned out that CT states influence the V_{OC} more than the j_{sc} . Simultaneously, V_{OC} values are connected with CT states band gap by the empiric relation [12]:

$$V_{OC_{teofl}} = E_{CT} - 0.47 \text{ eV}. \quad (9)$$

Therefore, V_{OC} values determined from energy level of materials, from optical spectra and from optoelectrical spectra are compared with the experimental values in this paragraph.

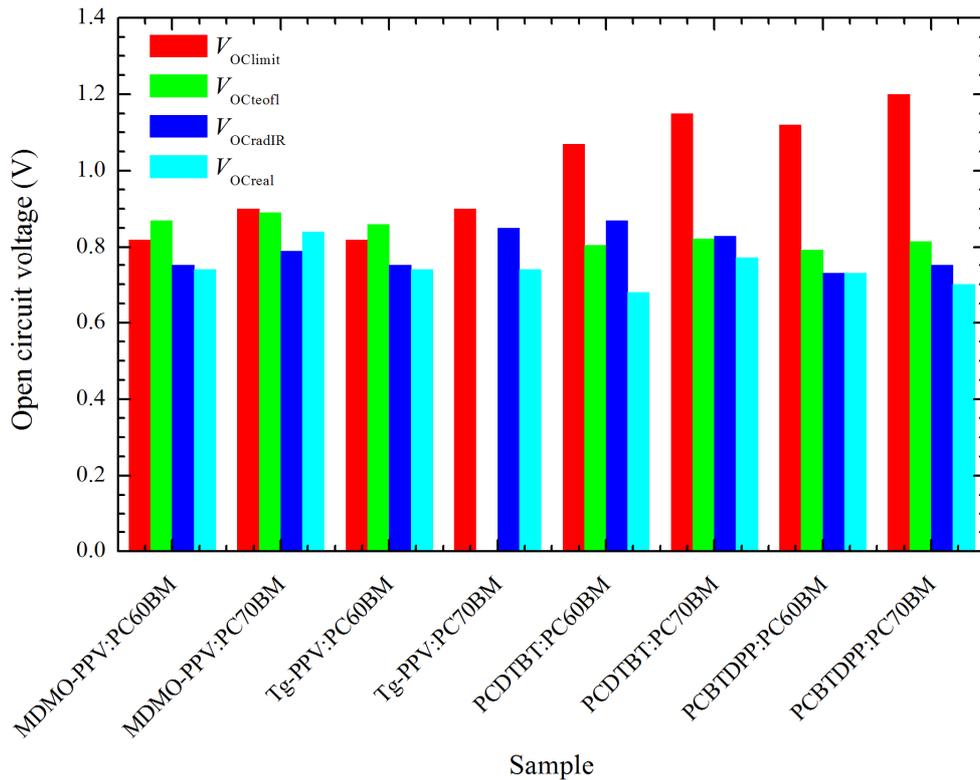


Fig. 19: Diagram comparing the values of open circuit voltage V_{OC} determined by different ways.

Determined values of open circuit voltage are compared in diagram on Fig 19. There is a good agreement between $V_{OC_{real}}$ and $V_{OC_{teofl}}$ as well as between $V_{OC_{real}}$ and $V_{OC_{radIR}}$. Values of $V_{OC_{limit}}$ are absolutely inconsistent with $V_{OC_{real}}$ for photovoltaic devices prepared from low band gap polymers.

These results reveal the importance of direct CT states detection in the solar cell's active layer for the estimation of the V_{oc} parameters.

3.3 Textile Organic Solar Cells

In the Introduction the advanced features of organic electronic devices were described. One of them is mechanical flexibility. It is the reason why part of work was paid to development of textile-based solar cell. The development was a part of solution of European project DEPHOTEX realized in our working group.

The general structure of developed device is shown on *Fig. 20*. Devices consist of conductive fabric, active layer, top transparent electrode and protective transparent polymer layer. The conductive fabrics were supplied by partners of project DEPHOTEX. Active layer was created by polymer Tg-PPV or by Tg-PPV:PC₆₀BM blend. Efficient hole transport was provided by transport layer from PEDOT:PSS.

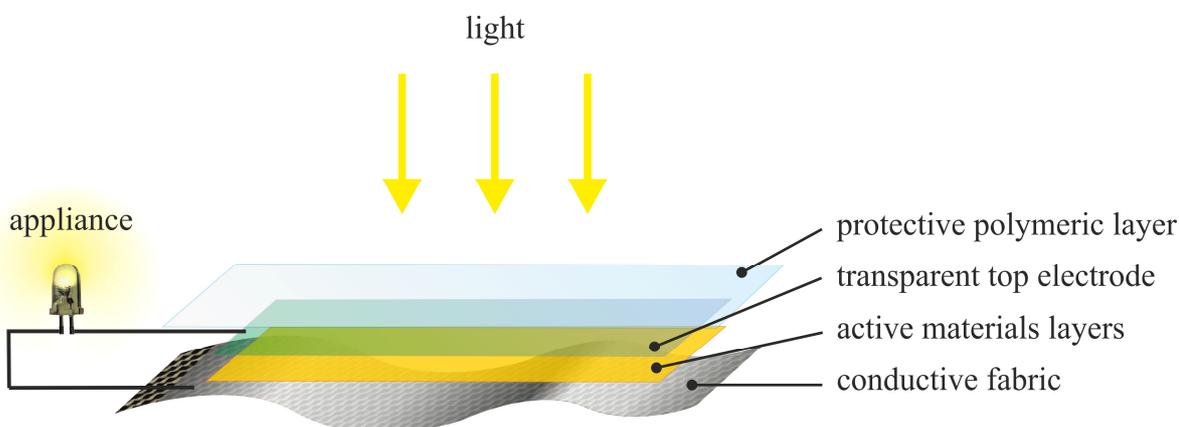


Fig. 20: General structure of textile solar cell is consisting of conductive fabric layer, active layer, transparent top electrode and protective polymer layer.

The development of textile photovoltaic devices required solution of wide range of tasks. Our effort was focused especially on the development of deposition methods for multi-layered OSC structure and optimization of preparation conditions.

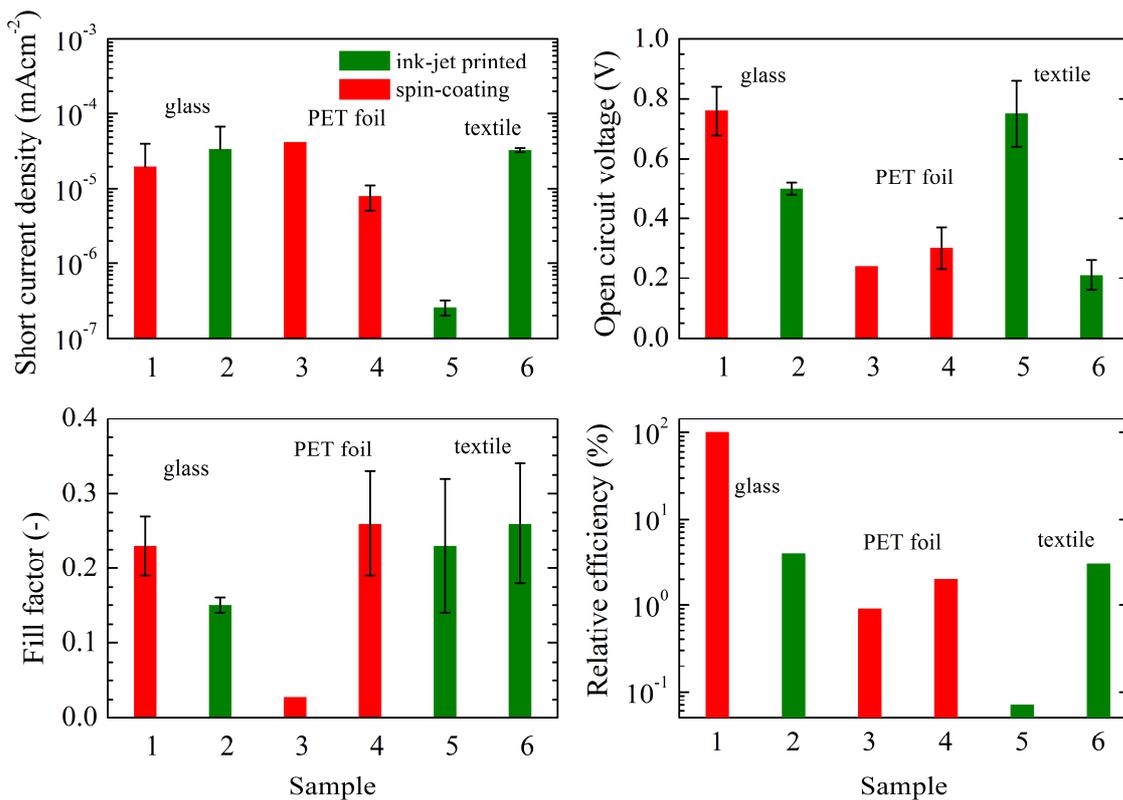


Fig. 21: Statistics of parameters of solar cells calculated from current voltage characteristics. From upper left: Short current density, open circuit voltage, fill factor and relative photogeneration efficiency at the maximal power. Numbers of samples present: 1 - glass/ITO/PEDOT/Tg-PPV:PCBM 1:4/Al, 2 - glass/ITO/ Tg-PPV:PCBM 1:4 (12 lay.)/Al, 3 - PET foil/ITO/ PEDOT/Tg-PPV:PCBM 1:4/Al, 4 - PET foil/ITO/ PEDOT/Tg-PPV:PCBM 1:4/Al/Ag-PES PU textile, 5 - textile/Al/ Tg-PPV:PCBM 1:4 (12 lay.)/Au, 6 - textile/Al/ Tg-PPV:PCBM 1:4 (12 lay.)/PEDOT/Au

Inkjet print was found as optimal deposition method for preparation of active layer of textile devices.

Achieved efficiencies of prepared textile devices are presented by the charts on the Fig. 21, where are compared with similar structures prepared by spin-coating and/or other substrates (glass, PET foil). Results summarized in these charts are considered as important, since they showed that achieved photogeneration efficiency of textile solar cells were near to the inkjet printed solar cells prepared on rigid substrates.

Inkjet print provides simple up-scale of photovoltaic device area and for this reason textile solar cells with active area to 7.5 cm² were prepared as Fig. 22 shows. The largest properly working solar cells were of 1 cm² large.

These experiments showed that preparation of OSC devices on textile by inkjet printing is possible. However a significant effort must be paid to reach the parameters suitable for practical applications.

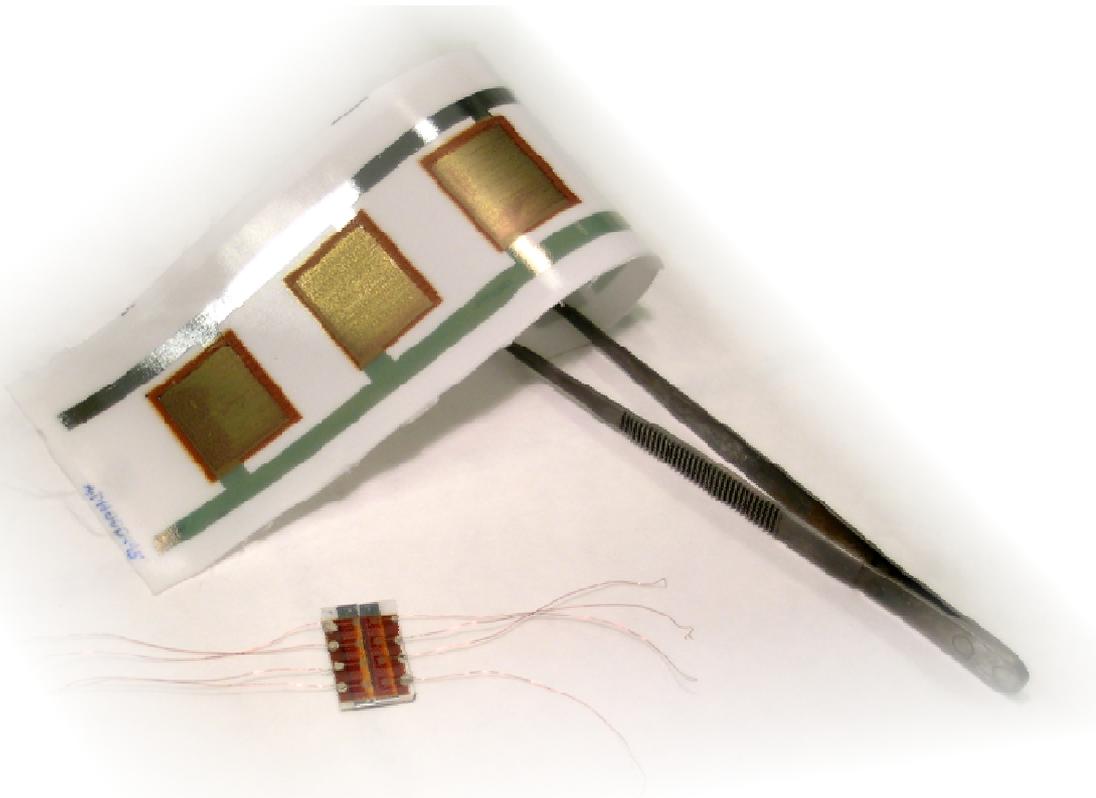


Fig. 22: The flexible module of four solar cells with 7.5 cm^2 active area prepared by inkjet printing on textile substrate. Standard sample (2.5×1.5) cm^2 with eight small electrodes (area 0.03 cm^2) is depicted for comparison too.

4 SUMMARY

The photogeneration processes especially charge transfer and resonance energy transfer in polymeric solar cells were studied by optical and optoelectrical methods. Four electron donating π -conjugated polymer MDMO-PPV, Tg-PPV, PCDTBT and PCBDPP and two electron accepting fullerenes PC₆₀BM and PC₇₀BM were used in experiments.

The research of electronic interaction between electron donating and electron accepting materials by optical methods were realized by fluorescence quenching experiments in solution and thin films. Results of the studies in solutions showed significant contribution of resonance energy transfer from polymer to fullerenes in comparison with charge transfer driven by exciton diffusion. In addition the studies of fluorescence quenching based on the modelling by Stern-Volmer and Perrin model enable to delineate these processes in thin layers.

Another goal of optical studies was focused on detection of CT states by analysis of fluorescence emission. Based on this analysis, the energy of maximum of emission peak originating from relaxation of excited CT states were determined.

Furthermore parameters of photovoltaic devices prepared from studied blends were estimated from optical spectra and molecular energy levels of materials.

Subsequently, the charge transfer states were studied by optoelectrical methods including the analysis of spectrally resolved external quantum efficiency and electroluminescence.

Significant contribution of light absorption of fullerenes to photogeneration efficiency was found by EQE spectra study. These spectra also showed photogeneration losses caused by light interferences in active layer.

Analysis of electroluminescence spectra allowed determination of energies of bimolecular recombination centres. It was concluded that value of energies rather correspond to energy level of fullerenes triplet states than CT states.

In order to identify of absorption CT states band, reciprocal relation between EQE spectra and electroluminescence spectra were studied. Electroluminescence spectra were substituted by photoluminescence spectra, which enable to determine the emission from CT states. Extended EQE spectra were used for estimation of open circuit voltage and calculation of short current density of prepared devices, which was in good agreement with parameters obtained by standard procedure from current voltage measurements.

Finally values of open circuit voltage determined from molecular energy levels, fluorescence spectra of CT states, extended EQE spectra were compared with values obtained from current voltage measurements and it was confirmed direct relation between energy of CT states and value of open circuit voltage.

In last part of the thesis results of textile organic solar cells development were demonstrated. The successful samples of photovoltaic devices prepared by inkjet print on textile substrates were presented.

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Given Speeches:

HEINRICHOVÁ, P.; VALA, M.; WEITER, M. The study of pi- conjugated polymers for photovoltaic application by fluorescence quenching experiments. *ReAdMat*. Pardubice (2013).

Selected Activities:

ISOPHOS2010 - International School on Organic Photovoltaics, Ventotene, Italy, 21.–27. 9. 2010. – poster presentation

10th IUVESTA School, 30. 5. 2011–04. 6. 2011- International Summer School on Physics at Nanoscale – poster presentation

5. Chemistry and life at FCH VUT Brno, 14-16. 9. 2011 – poster presentation

17th International School on Condensed Matter Physics, Bulharsko Varna, 2.-7. 9. 2012 – poster presentation

International Days of Materials Science, Pardubice 24.-25. 9. 2013 – presentation

Letní škola molekulové spektrometrie, Spektroskopická společnost Jana Marka Marci a MU Brno, 12.-14. 6. 2013

Teaching of: Laboratory classes in Physics (2009–2013), Laboratory classes in Instrumental and Structure Analysis – laboratory exercise “Organic photovoltaic cell” (2009–2014), leading of BSc. and MSc. graduating students in the laboratories during realization of experimental parts of their theses (2009–2014). Summer School of Nanotechnology – laboratory exercise of preparation and characterization of organic photovoltaic (2011-2013). Popular science: Noc vědců at FCH VUT (Brno), 2010 - 2013; Pátky s vědou at Hvězdárna a planetárium Brno, 11. 4. a 23. 5. 2014.