

Research Article

Laser Beam Induced Current Technique of Polymer Solar Cells Based on New Poly(Azomethine) or Poly(3-Hexylthiophene)

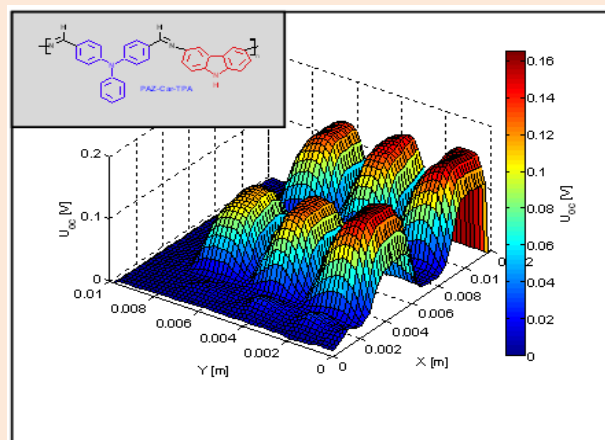
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Abstract

We synthesized new, air- and thermally stable poly(azomethine) with carbazole and triphenylamine moieties (PAZ-Car-TPA) and characterized their photovoltaic properties by Solar Simulator Model SS100AAA with AM 1.5G with an irradiation intensity of 100 mW/cm². The bulk heterojunction polymer solar cells with an active layer based on PAZ-Car-TPA as a donor and [6,6]-phenyl C61 butyric acid methyl ester (PCBM) as an acceptor were fabricated and studied. Moreover, devices with poly(3-hexylthiophene) (P3HT) were constructed and compared with polymer solar cells based on PAZ-Car-TPA. The laser beam induced current (LBIC) technique with a 420 nm laser with a beam size of the diameter of 100 μm was used to investigate electrical properties of constructed polymer solar cells. Additionally, a morphology of PAZ-Car-TPA, P3HT, PAZ-Car-TPA:PCBM and P3HT:PCBM was investigated by the atomic force microscopy.



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Introduction

Recently the organic solar cells are widely investigated in two main specific directions: (i) application of new polymers, copolymers or small compounds in an active layer of photovoltaic (PV) devices, and (ii) application of new device architecture [e.g. 1-16]. Considering the polymer structure used as an active layer in the construction of polymeric photovoltaic (PV) devices, poly(3-hexylthiophene) (P3HT) and poly[N-9'-heptadecan-2,7-carbazole-alt-5,5-(4',7'-di-2-thienyl-2',1',3'-benzothiadiazole)] (PCDTBT) have primarily been investigated as a donor material [1-16]. Presently, polymer solar cells based on P3HT:PCBM mixture receive the value of the power conversion efficiency (PCE) from below 1% to more than 6%, dependently on the specific architecture of devices, type of a hole transporting layer (HTL), and applied conditions (especially construction of a device in the air or in the glove-box). Taking into consideration the fact that P3HT is able to collect only about 22.4% of the available solar photons, new polymers and organic compounds are tested in terms of the application in PSCs [1, 17]. A lot of scientists work on the design of new polymers with a small value of the energy gap ($E_g \sim 1.4-1.5$ eV), soluble in such solvents as chloroform or 1,2-dichlorobenzene and stable in the air [1-16]. However, many important tasks remain still unsolved. For example, stability of polymer solar cells in the air and their lifetime are still the issue. For this reason, a lot of scientist investigated in details the morphology and defects of an active layer in polymer solar cells and interactions between layers in polymer solar cells [18-24].

Poly(azomethine)s (PAZ), are presently investigated by a few scientific groups for use as components of the active layer in organic solar cells [25–35]. In our previous works we reported the value of PCE equal to 0.56% which was reached for PAZ with triphenylamine moieties [32] and for PAZ with three thiophene rings [34]. It was the highest value of PCE received for this group of polymers at the present time, to the best of our knowledge.

The laser beam induced current (LBIC) technique method offers several advantages compared to other techniques. It is a nondestructive method and allows for detection of defects which significantly impact on solar cells energy and current efficiency. It also gives information about the uniformity of the parameters of the solar cell. The LBIC technique was used to investigate properties of inorganic solar cells [e.g. 36, 37]. Brooks *et al.* [36, 37] studied by triple wavelength LBIC technique the photovoltaic properties of $\text{Cd}_{1-x}\text{Zn}_x\text{S}/\text{CdTe}$ solar cells taking into consideration the influence of the thickness of $\text{Cd}_{1-x}\text{Zn}_x\text{S}$ on the PCE and V_{oc} .

In this work we propose the utilization of new poly(azomethine) with carbozole and triphenylamine moieties for application in bulk heterojunction (BHJ) polymer solar cells. For the first time the laser beam induced current (LBIC) technique is used to analyze electrical properties of constructed polymer solar cells based on poly(azomethine) or P3HT and PCBM. Finally, morphology of the active layers and polymers is investigated based on the atomic force microscopy (AFM) technique.

Experimental

Materials and synthesis procedure

4,4'-diformyltriphenylamine, 3,6-diaminocarbazole, *N,N*-dimethylacetamide (DMA), chloroform, poly(3-hexylthiophene) (P3HT) and [6,6]-phenyl C_{60} butyric acid methyl ester (PCBM) were purchased from Sigma-Aldrich and used as received. ITO and poly(3,4-ethylenedioxythiophene):poly(styrenesulfonate) (PEDOT:PSS) were purchased from the Ossila Company. The surface resistance of ITO was approximately 20 ohm/square.

Poly(azomethine) synthesis

The polymer was obtained using a one-step high temperature condensation technique with anhydrous CaSO_4 as the water trap. A single-neck flask with a magnetic stir bar was charged with dialdehyde (1 mmol), diamine (1 mmol), *p*-toluenesulfonic acid (PTS) and 10 mL of DMA. The reaction mixture was allowed to stir for 22 h at 160 °C in an oil bath. The polymer solution was precipitated in methanol, and the polymer was collected by filtration. The solid was washed with hot methanol and hot acetone. The final polymer was dried overnight at 80 °C.

PAZ-Car-TPA: yield 57%, dark green powder. ^1H NMR (300 MHz, CDCl_3), δ [ppm]: 9.89 (HC=O), 8.61 (HC=N), 8.13–7.13 (Ar). FT-IR, ν [cm^{-1}]: 3413 (N-H stretching), 3037 (aromatic C-H stretching), 1682 (C=O stretching from residue CHO groups), 1620 (C=N imine stretching), 1588–1458 (stretching in the aromatic ring), 1317–1280 (C-N stretching), 810 (bending C-H out of plane).

Characterization

The obtained polymer was characterized by the nuclear magnetic resonance (^1H NMR) spectra recorded on a Bruker DRX 300 MHz spectrometer using chloroform (CDCl_3) as the solvent and TMS as the internal standard, and Fourier infrared (ATR FTIR) spectra acquired on a Nicolet 5700 (ThermoElectron). Thermogravimetric analyses (TGA) were performed on a Mettler-Toledo AG apparatus at the heating rate of 10 °C/min under nitrogen. Conductivity (σ) of PAZ-Car-TPA layer was measured for ITO/PAZ-Car-TPA/Al structure at 25, 50 and 75 °C for 100, 200 and 500 mV. The thickness of the PAZ-Car-TPA layer was determined by the AFM technique by measuring the height of the edge developed using a sharp copper blade.

The surface morphology studies of the PAZ-Car-TPA (P3HT) and their mixture with PCBM were performed on air using a commercial Innova AFM system from Bruker (former Veeco) company. Measurements were done on air

using a Tapping Mode along with the phase imaging feature. A typical cantilever (about 40 N/m and < 10 nm tip curvature) was used. Films on the glass substrate have been obtained by dissolving the mixture in chloroform.

Construction and characterization of polymer solar cells

The solar cells were constructed on an indium tin oxide (ITO)-coated glass substrate with a ITO/PEDOT:PSS/PAZ-Car-TPA (P3HT):PCBM/Al structure in an air atmosphere. First, the ITO-coated glass substrate was cleaned with deionized water and then ultrasonicated in isopropanol for approximately 20 minutes. PEDOT:PSS was spin cast (2500 rounds per minute, 25 sec) from an aqueous solution to form a film on the ITO substrate. Then, a mixture of PAZ-Car-TPA (P3HT):PCBM in a chloroform solution with a weight ratio of 1:1 was spin cast on top of the PEDOT:PSS layer. An aluminium electrode was deposited by thermal evaporation in a vacuum of approximately 5×10^{-5} Torr. The solar cells were annealed for 30 minutes at 130 °C with (for P3HT) and without (for PAZ-Car-TPA) the Al electrode. The current density–voltage (J–V) characteristics of the solar cells were measured using a Solar Simulator Model SS100AAA with AM 1.5G. To simulate solar irradiation, a xenon lamp with an irradiation intensity of 100 mW/cm² was used. The area of one photovoltaic pixel was approximately 4.5 mm².

The experimental set up which was applied for the measurements of the laser beam induced current (LBIC) maps is presented in **Figure 1**. It consists of the 50 mW green 532 nm or blue 420 nm laser with a beam size of the diameter of 100 µm, the mirror which directs the light beam to the investigated solar cell, X–Y stages for the cell moving, the BNC-2120 module through which a signal is passed to the fast NI USB-6255 measuring card and a personal computer with a dedicated acquire – control application written in the C++ programming language. All measurements were computer controlled and performed at room temperature. The wavelength of the applied illuminating laser was $\lambda = 420$ nm.

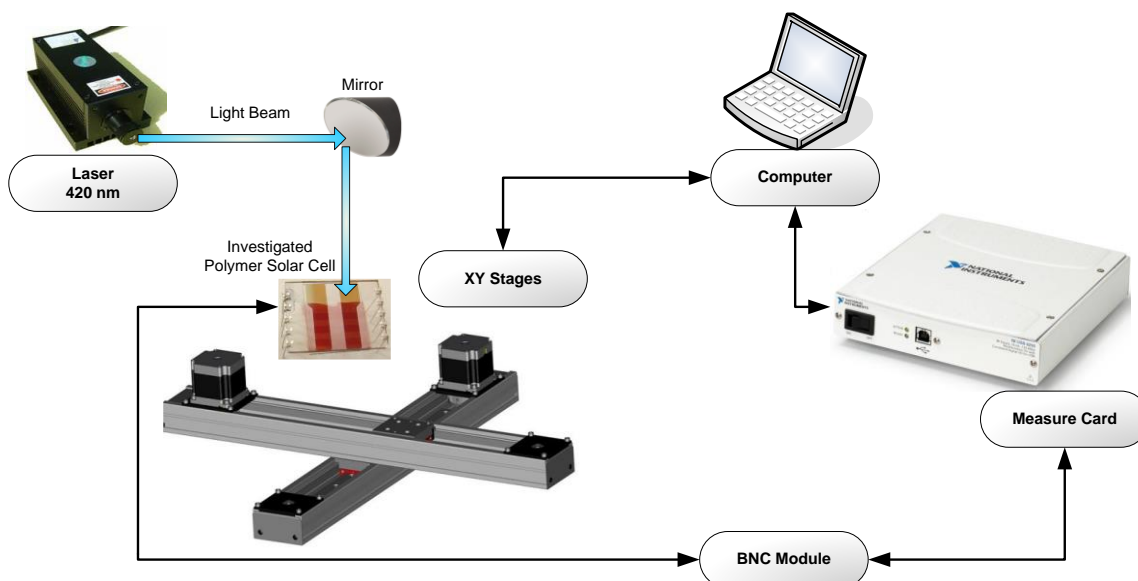


Figure 1 Experimental set up for laser beam induced current (LBIC) measurements

Result and Discussion

Basic characterisations

The poly(azomethine) PAZ-Car-TPA (**Figure 2**) was prepared using a simple one-step high-temperature condensation procedure with the catalyst in solution. The structure of the poly(azomethine) was confirmed by FT-IR and ¹H NMR spectroscopy, and the results were in agreement with the proposed chemical structure. In particular, in the proton NMR spectrum of the investigated polymer, the azomethine proton signal at approximately 8.61 ppm was observed, as expected. The presence of imine groups was confirmed by FT-IR spectroscopy, and the band characteristic of the -

HC=N- stretching deformations at 1620 cm^{-1} was observed. Consistent with the ^1H NMR data, the band characteristic of the carbonyl end group was also observed at approximately 1682 cm^{-1} . The FT-IR spectrum of PAZ-Car-TPA is shown in Figure 2.

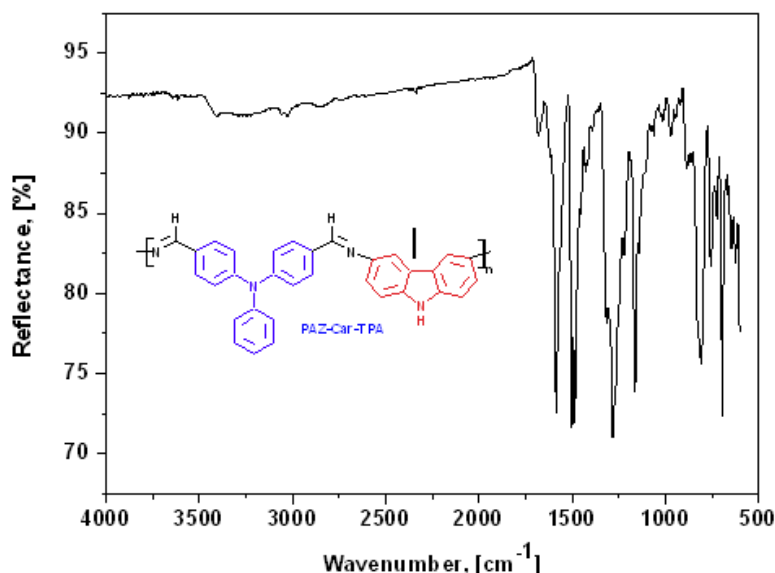


Figure 2 FT-IR spectrum of PAZ-Car-TPA along with chemical structure of PAZ-Car-TPA

The results from the TGA analysis in a nitrogen atmosphere suggested that PAZ-Car-TPA possessed a good thermal stability with a 5 % weight loss at $438\text{ }^{\circ}\text{C}$. For P3HT, 5 % weight loss at $456\text{ }^{\circ}\text{C}$ was found. The high thermal stability of the polymers prevents deformation of their morphology and is important for polymeric photovoltaic device applications.

The conductivity (σ) of PAZ-Car-TPA at $25\text{ }^{\circ}\text{C}$ was approximately $1.35 \times 10^{-10}\text{ S/cm}$. As the voltage increased from 100 to 500 mV, an increase in the conductivity of the polymer was observed (**Table 1**). Moreover, along with the increase of the temperature from 25 to $75\text{ }^{\circ}\text{C}$ a small increase in the conductivity was found. The conductivity of P3HT at $25\text{ }^{\circ}\text{C}$ was approximately $6.23 \times 10^{-8}\text{ S/cm}$. As the voltage increased from 100 to 500 mV, an increase in the conductivity of the polymer was observed and was found about $1.27 \times 10^{-7}\text{ S/cm}$ for 500 mV.

Table 1 Conductivity of PAZ-Car-TPA

V [mV]	σ [S/cm]		
	T=25 $^{\circ}\text{C}$	T=50 $^{\circ}\text{C}$	T=75 $^{\circ}\text{C}$
100	1.35×10^{-10}	1.33×10^{-10}	1.44×10^{-10}
200	1.88×10^{-10}	1.99×10^{-10}	2.09×10^{-10}
500	5.72×10^{-10}	5.24×10^{-10}	5.12×10^{-10}

AFM method was used to analyse morphology of both polymers and their mixture with PCBM. **Figure 3** shows typical, planar view of the topography, the Sobel transform of the topography which we used to provide a better view of fine structures and features, and angular spectra indicating the presence of a privileged orientation of the morphological features. Typical parameters obtained from AFM studies, such as roughness (S_a , S_q), skew (S_{sk}), and kurtosis (S_{ku}) of blends on the glass substrate, are presented in **Table 2**. Moreover, the surface area ratio (S_{dr}) has been determined, as a significant factor in terms of chemical and optical behaviour.

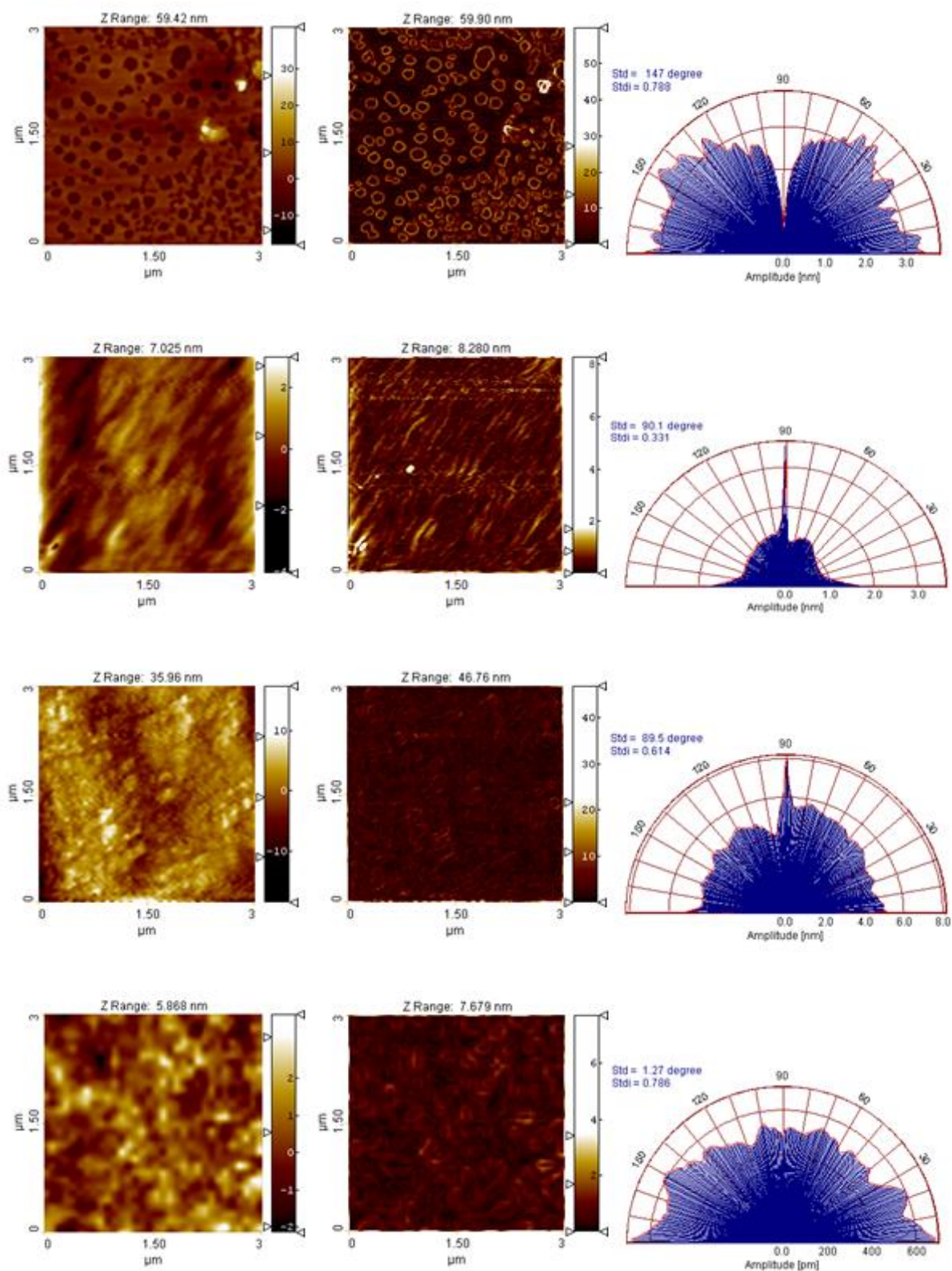


Figure 3 Topography, Sobel transform of the topography and Angular spectra of PAZ-Car-TPA, PAZ-Car-TPA:PCBM, P3HT and P3HT:PCBM (from top to bottom)

The angular spectra of all films were determined for the scanning field $3\ \mu\text{m} \times 3\ \mu\text{m}$ (Figure 3). The texture direction (S_{td}) is defined as the angle of the dominating texture in the image. For images consisting of parallel ridges, the texture direction is parallel to the direction of the ridges. This parameter is only meaningful if there are morphological objects oriented in a privileged direction. The texture direction index (S_{tdi}) is a measure of how dominant the preferential direction is, and is defined as the average amplitude sum divided by the amplitude sum of the preferential direction.

Table 2 The roughness parameters of investigated compounds by AFM

Sample	S_a [nm]	q [nm]	S_{sk}	k_u	S_{dr} [%]
PAZ-Car-TPA	3.140	4.229	0.328	9.015	1.123
PAZ-Car-TPA:PCBM	0.565	0.724	0.352	3.630	0.006
P3HT	2.286	2.889	-0.221	3.310	0.354
P3HT:PCBM	0.630	0.790	0.323	3.004	0.007
The roughness parameters were determined for scan area $3\ \mu\text{m} \times 3\ \mu\text{m}$					

PAZ-Car-TPA sample contains the pores in size from 50 to 300 nm in diameter (see Figure 3). The variety of the shapes of the pores includes round and oval, sometimes due to proximity effect few pores are connected creating more complex features. The depth of the pores is equal to the thickness of the film, therefore at their bottom, the substrate is exposed. One draws such a conclusion from both: topographical data and phase imaging (not presented here), revealing similar energy dissipation at the bottom of the holes and the substrate after the film was removed by scratching. The morphology of the PAZ-Car-TPA:PCBM film reveals presence of the linear, highly oriented structures in width 100-200 nm and length 500-2000 nm (Figure 3). Due to smooth shapes of the structures and their location at that same level, the roughness of the surface is very small. The angular spectrum shows the specific direction of the ordering of the features.

P3HT sample contains relatively homogenous grainy round and oval objects revealing some level of the ordering revealed by the angular spectrum graph. The range of the structures diameter can be found from 50-200 nm (Figure 3). P3HT:PCBM film is a very flat surface containing relatively homogeneously dispersed small round and oval grains of size in diameter 20-100 nm (Figure 3). No particular ordering was detected.

For all investigated materials we found the presence of submicron level of organization. Additionally, the S_{td} and S_{tdi} factors indicate certain level of ordering of the features, which change along with the change of the polymer structure and PCBM presence/ absence.

Photovoltaic properties

The current density–voltage (J–V) characteristics of the constructed poly(azomethine) solar cells were measured first using a Solar Simulator Model SS100AAA with AM 1.5G with an irradiation intensity of $100\ \text{mW}/\text{cm}^2$. The area of one photovoltaic pixel was approximately $4.5\ \text{mm}^2$. On the one glass substrate there were six pixels as is presented in **Figure 4**. The PV parameters for the PAZ-Car-TPA:PCBM BHJ polymer solar cells are listed in **Table 3**. The J–V (current–voltage) characteristics of the poly(azomethine) BHJ solar cells along with photo of the constructed polymer solar cell based on PAZ-Car-TPA are shown in Figure 4.

The device composed of the PAZ-Car-TPA with PCBM (1:1) showed an open circuit voltage (V_{oc}) of 0.135 V, a short circuit current density (J_{sc}) of $0.185\ \text{mA}/\text{cm}^2$, and a fill factor (FF) of 0.34, giving a power-conversion efficiency (PCE) of 0.008 % under an illumination of $100\ \text{mW}/\text{cm}^2$ with an AM1.5G (see Table 3). Obtained PV parameters were very poor, however well reproducible for each of the 6 pixels. Value of PCE of BHJ solar cells with P3HT:PCBM (1:0.8) was about 220 times higher than for the device with PAZ-Car-TPA:PCBM (Table 3).

Series resistance (R_s) and a shunt resistance (R_{sh}) values for the BHJ devices are listed in Table 3. The highest values of resistance were found for R_{sh} than for R_s for both kinds of devices. Both resistances of devices with PAZ-

Car-TPA:PCBM were bigger than for the solar cell with P3HT:PCBM. The higher R_s value of devices with PAZ-Car-TPA:PCBM is probably related to lower charge mobility and in consequence in this case the lowest value of PCE was found (see Table 3). The lowest value of series resistance was detected for devices with P3HT:PCBM, and exhibited the highest PCE value.

V_{oc} is also demonstrated to decrease in the devices with PAZ-Car-TPA:PCBM, however not related to the reduced shunt resistance in devices (see Table 3). This means that recombination in poly(azomethine) solar cell is not the main aspect to reduce efficiency of devices.

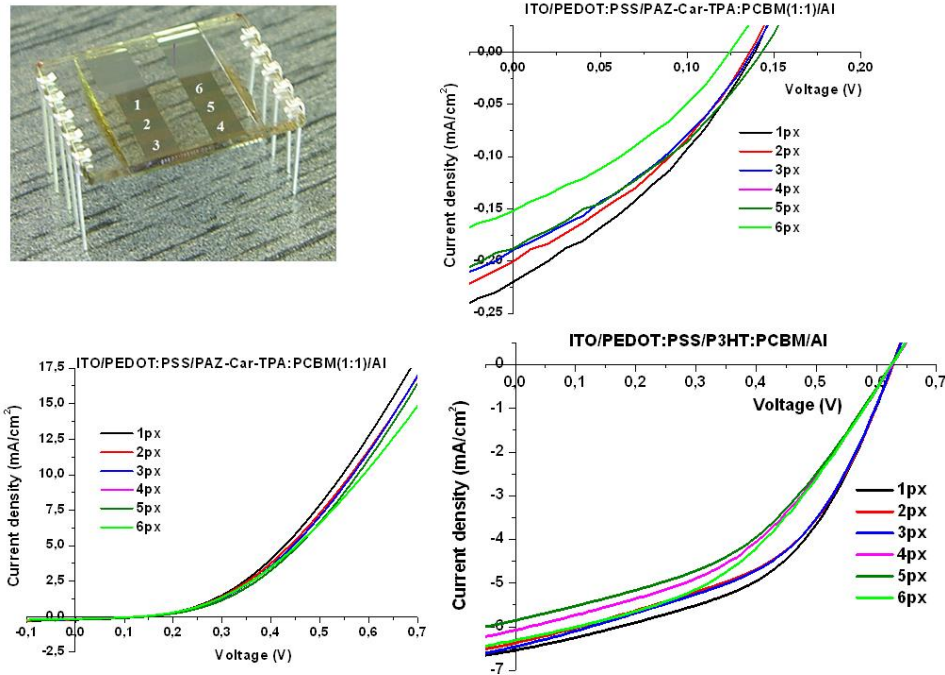


Figure 4 Photo of the constructed polymer solar cell based on PAZ-Car-TPA along with J-V curves of BHJ polymer solar cells under AM 1.5 G-simulated solar illumination (100 mW/cm^2)

Table 3 PV performance for BHJ polymer solar cells under AM 1.5 G-simulated solar illumination (100 mW/cm^2)

Pixel	J_{sc} [mA/cm ²]	V_{oc} [V]	FF [-]	PCE [%]	I_{sc} [μA]	R_{sh} [kΩ]	R_s [kΩ]	P_{max} [μW]
ITO/PEDOT:PSS/PAZ-Car-TPA (P3HT):PCBM/Al								
1	0.216 (6.53)	0.140 (0.627)	0.34 (0.49)	0.010 (2.01)	9.706 (294.1)	23.31 (8.084)	6.314 (0.585)	0.459 (90.52)
2	0.198 (6.38)	0.137 (0.627)	0.34 (0.48)	0.009 (1.92)	8.888 (286.8)	25.40 (8.467)	6.655 (0.591)	0.416 (86.03)
3	0.187 (6.44)	0.140 (0.626)	0.34 (0.48)	0.009 (1.94)	8.436 (290.3)	26.29 (8.199)	7.303 (0.601)	0.395 (86.38)
4	0.184 (6.07)	0.143 (0.626)	0.34 (0.43)	0.009 (1.63)	8.258 (273.3)	27.06 (7.996)	7.213 (1.002)	0.401 (73.15)
5	0.178 (5.84)	0.123 (0.624)	0.33 (0.43)	0.007 (1.57)	8.037 (263.5)	24.36 (10.53)	7.051 (0.999)	0.329 (71.07)
6	0.149 (6.31)	0.126 (0.625)	0.34 (0.43)	0.006 (1.70)	6.695 (283.8)	31.13 (6.505)	8.050 (0.979)	0.284 (75.90)
average	0.185 (6.26)	0.135 (0.626)	0.34 (0.46)	0.008 (1.79)	8.337 (282.0)	26.26 (8.417)	7.098 (0.793)	0.381 (80.51)

deviation	0.022	0.008	0.00	0.001	0.998	2.735	0.596	0.063
	(0.26)	(0.001)	(0.03)	(0.18)	(11.48)	(1.304)	(0.220)	(8.120)
%	12.0	6.1	1.2	16.8	12.0	10.4	8.4	16.5
	(4.1)	(0.2)	(6.4)	(10.2)	(4.1)	(15.5)	(27.7)	(10.1)

V_{oc} - open circuit voltage, J_{sc} - short circuit current density, I_{sc} - short circuit current, FF - fill factor, PCE - power conversion efficiency, R_s - series resistance, R_{sh} - shunt resistance, R_s and R_{sh} were calculated from the slope of the J-V characteristic curves

The main question is why PAZ-Car-TPA is not suitable to be applied in polymer solar cells? To solve this problem, constructed devices were investigated by laser beam induced current (LBIC) measurements. The spatial distributions along with map of the open circuit voltage $V_{oc}(x,y)$ values of the investigated polymer solar cell based on PAZ-Car-TPA:PCBM and P3HT:PCBM are presented in **Figure 5**. The 420 nm wavelength of the illuminating laser was used.

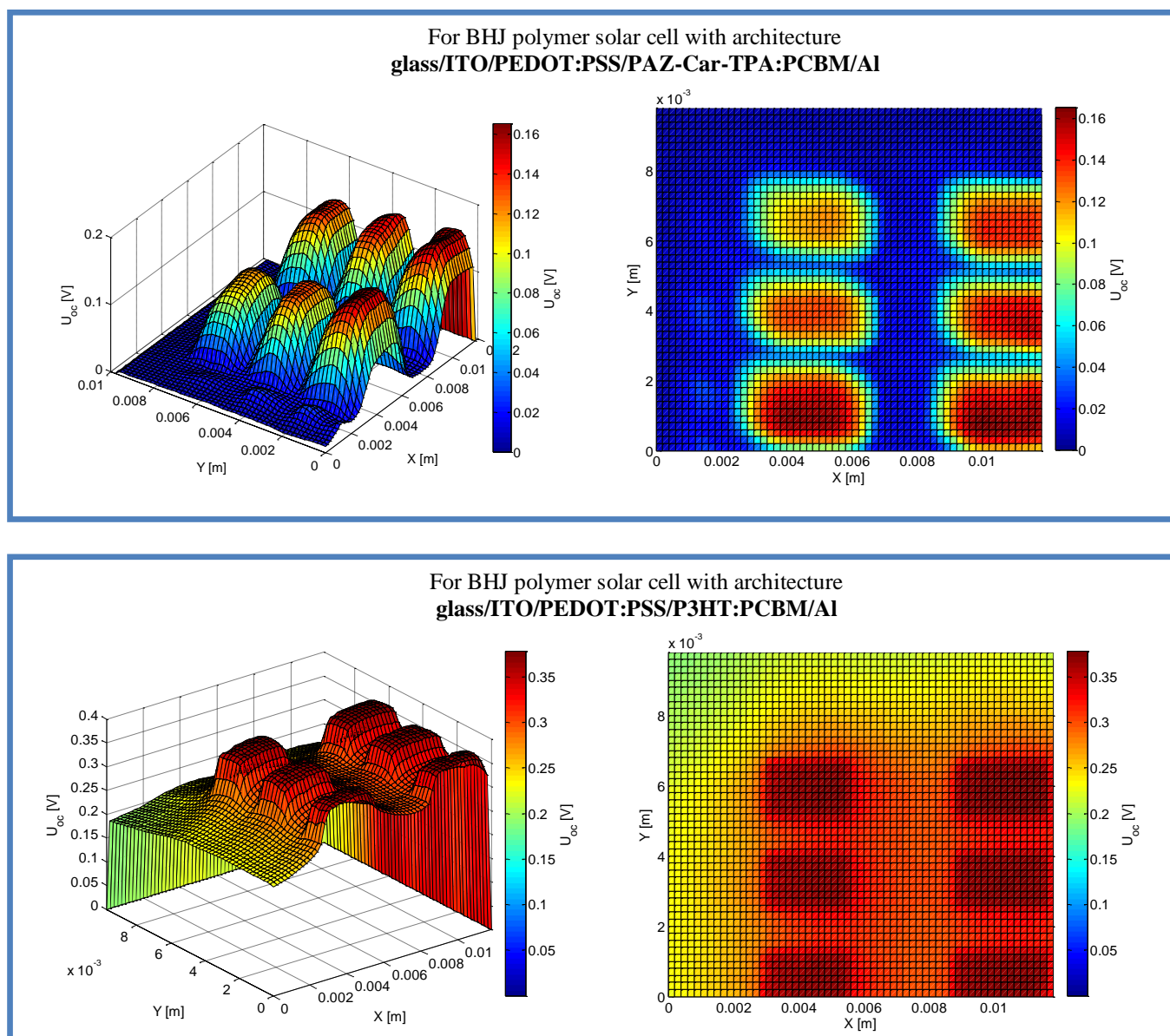


Figure 5 Surface plot along with map of the open circuit LBIC voltage $V_{oc}(x,y)$ values obtained for the investigated polymer solar cell based on PAZ-Car-TPA (top) and P3HT (bottom) at the blue laser illumination

From Figure 5 one can see that the open circuit voltage V_{oc} of each of six individual pixels in the polymer solar cell is equal to about 0.16 V for PAZ-Car-TPA:PCBM and about 0.35 V for P3HT:PCBM. The spatial distributions along with map of the short circuit current $I_{sc}(x,y)$ of the investigated polymer solar cell based on PAZ-Car-TPA:PCBM are presented in **Figure 6**.

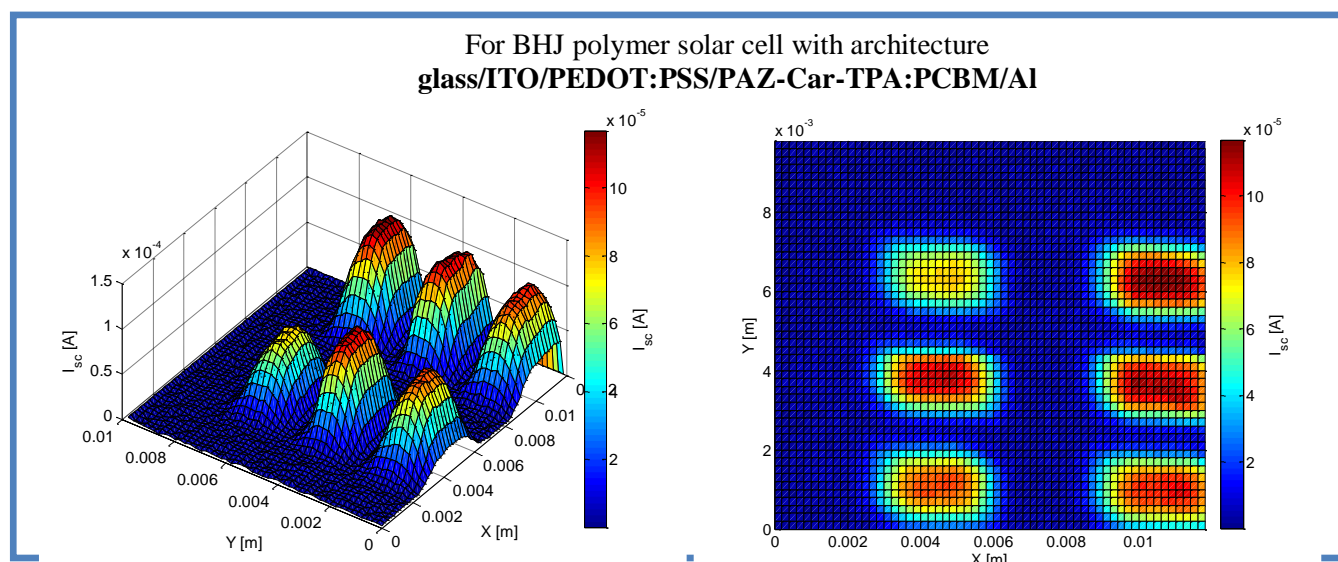


Figure 6 Surface plot along with map of the short circuit LBIC current $I_{sc}(x,y)$ values obtained for the investigated poly(azomethine) solar cell at the blue laser illumination

From Figure 6 one can see that the short circuit current of each of 6 individual pixels of polymer solar cell based on PAZ-Car-TPA:PCBM equals to about 80 μA while for devices with P3HT:PCBM was found about 1300 μA . Of course it depends on the intensity of illumination of the cell. A serial or parallel connection of the individual solar cells can considerably increase both the current and the voltage output of the system.

Conclusions

A new poly(azomethine) with carbazole and triphenylamine moieties with a quite good solubility in chloroform was synthesized as a donor component of an active layer of BHJ polymer solar cells. Devices based on PAZ-Car-TPA:PCBM exhibited a very poor value of PCE compared to P3HT:PCBM, however well reproducible for each of the 6 pixels. We have studied the electrical properties of constructed polymer solar cells by laser beam induced current technique taking into consideration the applied polymer. It was a surprise for us that V_{oc} detected by LBIC for a device with P3HT:PCBM was two times lower than detected under AM 1.5 G-simulated solar illumination. On the other hand a device with PAZ-Car-TPA:PCBM exhibited a little higher V_{oc} value detected by LBIC than under AM 1.5 G-simulated solar illumination. Moreover, V_{oc} of each of six individual pixels in polymer solar cell based on PAZ-Car-TPA:PCBM was about two times lower than for devices with P3HT:PCBM as was detected by LBIC. On the other hand, V_{oc} of poly(azomethine) solar cells under AM 1.5 G-simulated solar illumination was about five time lower than for the device based on P3HT. However, independently on the light of source used in the both cases, V_{oc} was higher for device with P3HT:PCBM than for devices with PAZ-Car-TPA:PCBM active layer. These differences probably could be explained by various sources of illumination applied: laser ($\sim 630 \text{ W/cm}^2$) in the first case and a xenon lamp (0.1 W/cm^2) in the second case. Probably at extremely high light intensity surface recombination in a device with P3HT:PCBM under laser illumination happened.

The AFM study showed that both polymers exhibited various morphology. PAZ-Car-TPA exhibited root-mean-square (S_q) roughness value at 4.23 nm, whereas P3HT possess S_q at $\sim 2.89 \text{ nm}$. On the other hand, a mixture of each

of polymer with PCBM caused that value of S_q is comparable. This behavior can be probably explained by a similar miscibility of both polymers in an active layer with PCBM.

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