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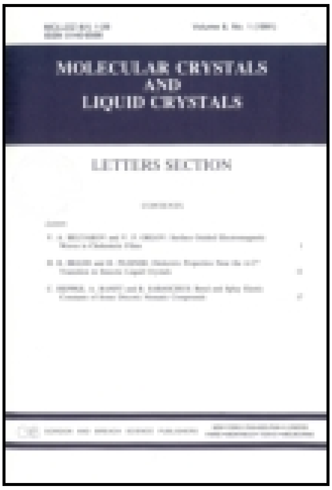

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Effect of Phthalimide in 2,1,3-Benzooxadiazole Based Copolymer on the Performances of Solar Cells

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Effect of Phthalimide in 2,1,3-Benzooxadiazole Based Copolymer on the Performances of Solar Cells

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Two newly designed donor-acceptor (D-A) conjugated polymers were prepared from the Stille coupling reactions by incorporating phthalimide derivative as a co-acceptor unit into 2,1,3-benzooxadiazole (BO) and thiophene with different ratios of 5 mol.% and 10 mol.% of TBO-I 5, TBO-I 10, respectively. Polymers possess moderate molecular weights and excellent thermal properties with a 5% weight loss temperatures (T_d) around 300°C. Compared to poly{5,6-bis(decyloxy)-4-(thiophen-2-yl)benzo[c][1,2,5]oxadiazole} (TBO), adding phthalimide unit in TBO-I 5 and TBO-I 10 shifted UV-Vis absorption spectra and changed the HOMO and LUMO levels of polymers. Bulk hetero junction (BHJ) polymer solar cells (PSCs) based on polymers blended with [6,6]-phenyl-C61-butyric acid methyl ester ($PC_{61}BM$) were fabricated to investigate photovoltaic properties. As a results, with increasing the composition of phthalimide, the J_{sc} *of the devices based on polymers were reduced in the order of TBO (4.74 mA/cm^2) > TBO-I 5 (3.94 mA/cm^2) > TBO-I 10 (1.90 mA/cm^2) due to the blue-shifted in absorption spectra and twisting in polymer main chain of TBO-I polymers. The decrease in* J_{sc} *led to low photovoltaic performances and the device based on TBO showed higher performances than those of TBO-I 5 and TBO-I 10 with the PCE of 1.66%, the* J_{sc} *of 4.74 mA/cm^2, the V_{oc} of 0.82 V and the FF of 42.4% under the illumination of AM 1.5G, 100 mW/cm^2.*

Keywords Polymer solar cell; π-conjugated polymer; 2,1,3-benzooxadiazole; phthalimide; Stille coupling reaction

1. Introduction

Promising to become a renewable energy with outstanding advantages of low cost, light weight, and potential of being fabricated by a roll-to-roll process and large-area devices, PSCs have been intensely investigated in recent years [1, 2]. The best PSC reported to date is commonly composed of a layer of a conjugated polymer as a p-type semiconductor and

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a fullerene derivative as an n-type semiconductor BHJ composite sandwiched between a transparent conducting electrode and a low work function metal electrode [3]. However, their low power conversion efficiency (PCE) is a major barrier to commercialization. For increasing the performances, great efforts have been devoted to the design and synthesis of new conjugated polymer for donor and new fullerene derivative for acceptor materials. Recently, donor-acceptor conjugated polymers have been extensively studied because this has been proven to be one of the most successful strategies to get the ideal conjugated polymer with desired characteristics such as broad absorption in visible (narrower band gap) for large solar photon harvest, higher hole mobility, and relatively lower-lying the HOMO (the highest occupied molecular orbital) energy levels for high open-circuit voltage, appropriate energy offset between the lowest unoccupied molecular orbital (LUMO) of the donor polymer and that of the fullerene for effcient charge separation [4]. Among the acceptor units, benzooxadiazole (BO) has been widely implemented as an electron-deficient co-monomer of low band gap polymers; it shows strong electron-withdrawing property of two imine nitrogens and relatively stable quinoid form. PSC devices with the blend of BO-containing conjugated polymer and $PC_{61}BM$ as the active layer have shown PCEs of over 5% in several reports [5–7]. Besides, phthalimide derivatives have been used as acceptor units in organic electronics due to their good electron withdrawing and π-π stacking properties [8]. Phthalimide-based donor/acceptor copolymers were found to have a field-effect hole mobility as high as 0.28 cm^2/Vs [9], clearly suggesting its investigation as a solar harvesting material. Furthermore, the HOMO energy level of phthalimide is lower than other typical electron withdrawing materials, such as benzothiadiazole, quinoxaline, oxadiazole [10] which is benefit to enhance the open circuit voltage (V_{oc}) value of PSC devices. From this point of view, two D-A type of polymers containing BO and phthalimide derivatives as co-acceptor units and thiophene as a donor unit with different ratios were synthesized through a Stille coupling reaction. In addition, we also investigated the effect of the amount of phthalimide on optical, electrochemical, and photovoltaic properties of copolymers.

2. Experimental

2.1. Synthesis of Monomers

Compounds 1, 2, 3, 4, and 5 were synthesized according to the literature procedure [11, 12].

2.2. Synthesis of Polymers

Poly{5,6-bis(decyloxy)-4-(thiophen-2-yl)benzo[c][1,2,5]oxadiazole} (TBO). Compound **4** (118 mg, 0.2 mmol), 2,5-bis(trimethylstannyl)thiophene (81.6 mg, 0.2 mmol), Pd_2dba_3 (9.12 mg, 10.0 μmol) and tri-(*o*-tolyl)phosphine (24.3 mg, 80.0 μmol) were mixed in dry degassed toluene (6.0 mL). The reaction mixture was heated to reflux for 48 hours under the nitrogen atmosphere. After cooling to room temperature, the mixture was poured into 200 mL methanol and the polymer was allowed to precipitate. The polymer was filtered and purified by Soxhlet extraction using methanol, hexane and chloroform. The chloroform fraction was concentrated by rotary evaporator and then poured into methanol to precipitate again. The polymer was filtered and dried in vacuum to give TBO (71.4 mg, 69.6%) as black solid. ^{1}H-NMR (400 MHz, $CDCl_3$, ppm): δ 8.67–8.24 (br, 2H), 4.43–4.21 (br, 4H), 2.26–2.18 (br, 4H), 2.13–1.97 (br, 4H), 1.61–1.40 (br, 4H), 1.31–1.23 (br, 20H), 0.86–0.84 (br, 6H).

Poly{4-[5-(5,6-Bis-decyloxy-benzo[1,2,5]oxadiazol-4-yl)-thiophen-2-yl]-co-2-butyl-5,6-bis-decyloxy-isoindole-1,3-dione} (TBO-I 5 and TBO-I 10)

TBO-I 5 was synthesized by the Stille coupling reaction between compound **4** (106 mg, 0.18 mmol), compound **9** (13.5 mg, 0.02 mmol) and 2,5-bis(trimethylstannyl)thiophene (81.6 mg, 0.2 mmol). The same reaction condition and procedure were used as in the polymerization of TBO. The yield of the polymer was 63.4 mg (61.2%) as black solid. ^{1}H-NMR (400 MHz, $CDCl_3$, ppm): δ 8.72–7.98 (br, 2H), 4.56–4.16 (br, 4H), 4.11–3.89 (br, 4H), 3.67–3.48 (br, 2H), 2.30–1.92 (br, 6H), 1.63–1.01 (br, 62H), 0.95–0.75 (br, 15H).

TBO-I 10 was synthesized by the Stille coupling reaction between compound **4** (94.5 mg, 0.16 mmol), compound **9** (26.9 mg, 0.04 mmol) and 2,5-bis(trimethylstannyl)thiophene (81.6 mg, 0.2 mmol). The same reaction condition and procedure were used as in the polymerization of TBO. The yield of the polymer was 53.1 mg (50.3%) as black solid. ^{1}H-NMR (400 MHz, $CDCl_3$, ppm): δ 8.67–8.01 (br, 2H), 4.51–4.16 (br, 4H), 4.14–3.87 (br, 4H), 3.66–3.49 (br, 2H), 2.22–1.95 (br, 6H), 1.66–0.99 (br, 62H), 0.94–0.76 (br, 15H).

2.3. Fabrication of Devices

PSCs were fabricated with a device configuration of ITO/PEDOT:PSS/polymer:PCBM/Al. ITO-glass substrates were pre-cleaned, dried on a hot-plate at 150°C for 10 min and treated by UV/O_3 for 120 sec. The solution of poly(3,4-ethylenedioxythiophene):poly(styrenesulfonate) (PEDOT:PSS, Clevios P) (diluted with 2-propanol, PEDOT:2-propanol = 1:2 by volume) was spin-coated on ITO substrate (sheet resistance = 15 Ω/sq) and the thickness of this layer is about 40 nm. After being baked at 150°C for 10 min under the air, the active layer was spin-cast from the blend solution of polymer/PCBM at 600 rpm for 120 s. The blend of polymer was prepared from 5 mg of polymer and 10 mg of PCBM were dissolved in 1 mL of chloroform (CF). The blended solution was stirred for overnight at 60°C in the glove box. Prior to spin coating, the active solution was filtered through a 0.45 μm membrane filter. Then, the aluminum cathode was deposited with a thickness of 110 nm through a shadow mask with a device area of 0.13 cm^2 at 2×10^{-6} Torr.

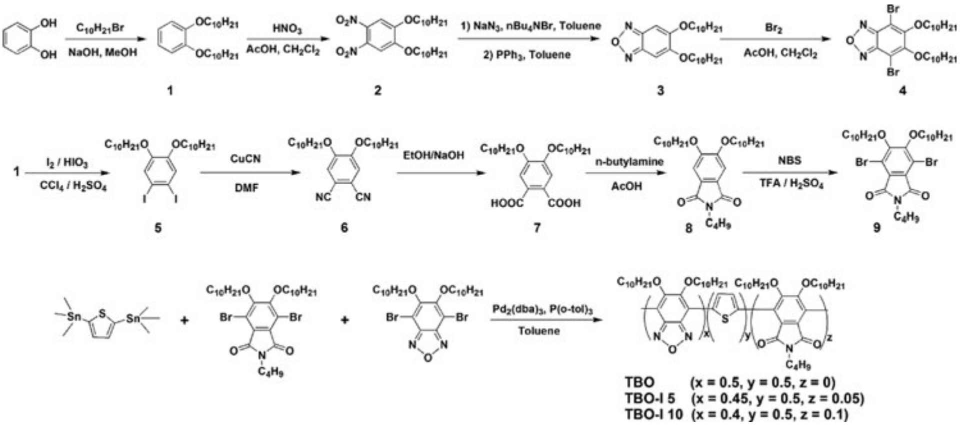
2.4. Measurement

Synthesized compounds were characterized by ^{1}H-NMR and ^{13}C-NMR spectrum, which were obtained with a JEOL JNM ECP-400 spectrometer. FTIR spectra of the prepared polymers on silicon substrates were obtained using a Jasco Model FTIR 4100 spectrophotometer at room temperature. Gel permeation chromatography (GPC) measurements were conducted by GPC system equipped with a Varian 212-LC pump, a Rheodyne 6-port sample injection valve, a Waters Temperature Control Module, a Waters 410 differential RI detector, and two Waters Styragel HR4E columns. The monodispersed polystyrene was used as standards and toluene used as an eluent. The thermogravimetric analysis (TGA) was carried out under the nitrogen atmosphere at a heating rate of 10°C/min with a Perkin-Elmer TGA 7 thermal analyzer. The UV-Vis spectrum was recorded using a JASCO V-530 UV-Vis Spectrophotometer. The cyclic voltammetry (CV) was performed by an Ivium B14406 with a three electrode cell in a 0.1 M n-Bu_4NPF_6 solution in acetonitrile at a scan rate of 100 mV/s. A Pt coil was used as the counter electrode and dip-polymer on a Pt wire was used as the working and a Ag/Ag$^+$ electrode was used as the reference electrode. The *J*–V measurements under 1.0 sun (100 mW/cm^2) condition from a 150 W Xe lamp with an AM 1.5G filter were performed using a KEITHLEY Model 2400 source-measure unit. A calibrated Si reference cell with a KG5 filter certified by National Institute of Advanced Industrial Science and Technology was used to confirm 1.0 sun condition.

3. Results and Discussion

3.1. Synthesis and Characterization

General synthetic strategy for monomers and polymer is outlined in Scheme 1. The three polymers were synthesized by the Pd-catalyzed Stille-coupling reaction. The polymers were good soluble in common organic solvents such as chloroform, chlorobenzene, and 1,2-dichlorobenzene. The chemical structures of polymers were confirmed by ^{1}H NMR and FTIR. Figure 1 exhibits the FT-IR spectra of TBO, TBO-I 5 and TBO-I 10. The vibration bands around 1720-1690 cm^{-1} are attributed from the stretching vibrations of the C=O groups on the phthalimide moiety. The result indicates the successful preparation of the polymers. The molecular weight of the polymers was determined by the GPC. The number average molecular weight (M$_n$) of TBO, TBO-I 5 and TBO-I 10 were 68500, 33400 and 25200 g/mol, respectively with polydispersity indexes (PDI) of 1.73, 1.76 and 1.86, respectively. The TGA (Figure 2) tests showed that the decomposition temperatures at 5% weight loss of TBO, TBO-I 5 and TBO-I 10 are 308, 297 and 298°C, respectively, implying that these polymers have adequate thermal stability for the applications in photovoltaic cells.



Scheme 1. Synthesis route of monomers and polymers.

3.2. Optical and Electrochemical Properties

The photo-physical characteristics of the polymer films were investigated by the UV-Vis absorption spectroscopy. The UV–Vis absorption spectra and optical properties were showed in Figure 3 and Table 1. All of the absorption spectra exhibited two absorption bands: the shorter wavelength at 310-380 nm corresponds to localized π-π^* transition of the conjugated polymer backbone and the other boarder band at 460-720 nm corresponds to intramolecular charge transfer (ICT) between the donor and acceptor units [13]. When

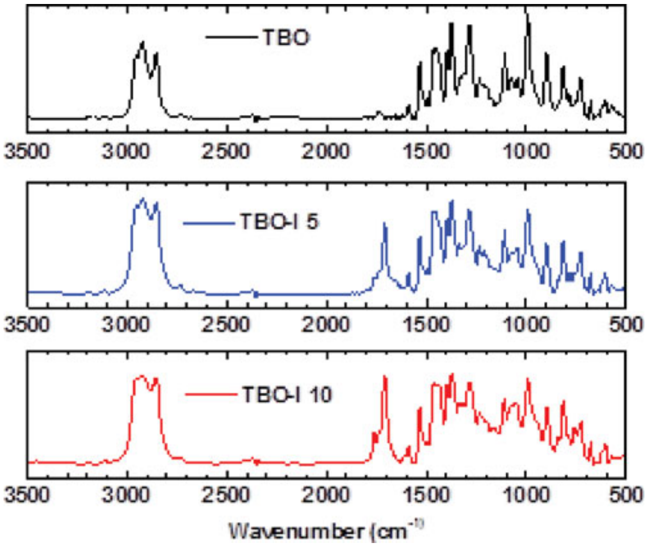


Figure 1. IR spectra of polymers.

increasing the amount of phthalimide, the absorption maximum wavelength of the polymers TBO-I 5 and TBO-I 10 are blue shifted compared to that of TBO, which might be ascribed to a relatively weak acceptor unit of phthalimide [14]. In addition, the main chain of the polymer may cause a steric interaction between the alkyl chains which results in twisting of the polymer main chain which could lead to a large band gap, low charge carrier mobility [15]. The absorption spectrum of TBO shows two maximum absorption peaks at 606 and 663 nm. Whereas TBO-I 5, TBO-I 10 show the maximum absorption peaks at 606, 603 nm, respectively; and shoulder peaks appear around 640–680 nm. Increasing the ingredient of phthalimide leads to decrease the intensity of shoulder peak. This absorption behavior

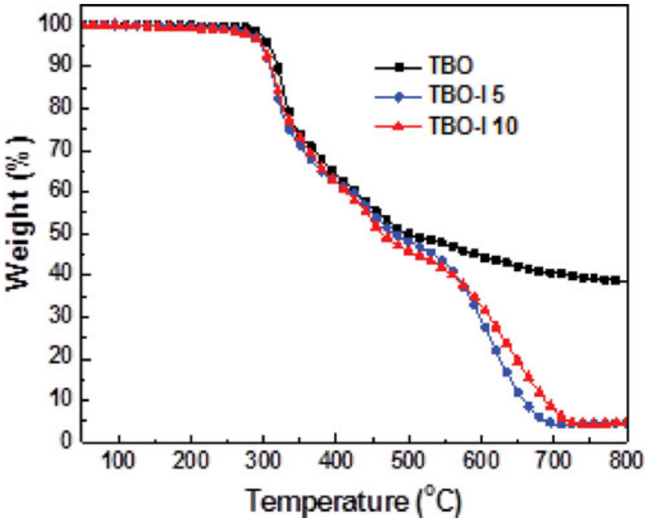


Figure 2. TGA thermograms of polymers.

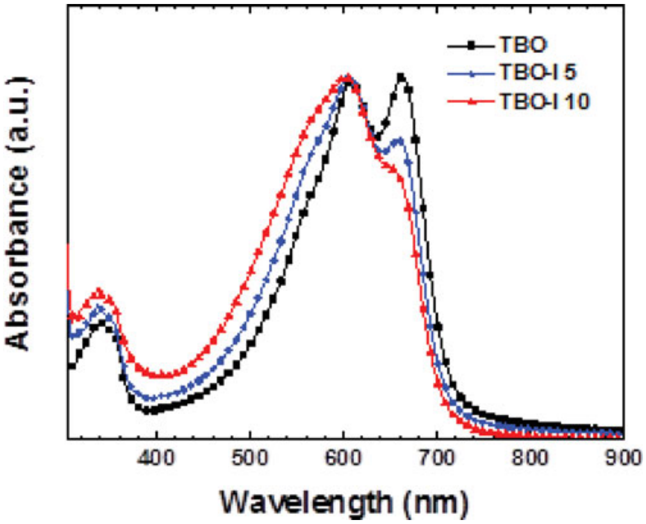


Figure 3. UV-Vis absorption spectra of polymers.

indicates that TBO aggregates more strongly than polymers containing phthalimide and there exist less ordered structure in the polymer films of TBO-I 5 and TBO-I 10, which is detrimental to hole mobility and photovoltaic performance of the polymers [16]. The optical band gap of polymers TBO, TBO-I 5 and TBO-I 10 estimated from the absorption edge in the UV-Vis spectra of the thin films are 1.74, 1.75 and 1.76 eV, respectively.

The HOMO levels figured out from the cyclic voltammograms (Fig. 4) of TBO, TBO-I 5 and TBO-I 10 were −5.53, −5.62 and −5.68 eV, respectively. As mentioned above, the HOMO energy level of phthalimide is lower than that of BO, hence the HOMO energy level of polymers containing phthalimde are lowered. It was reported that a stronger electron-deficient unit resulting in a lower LUMO energy level [17]. Therefore, increasing the amount of phthalimide moiety in the polymer leads to increase the LUMO energy levels of polymers. The electrochemical band gap of TBO, TBO-I 5 and TBO-I 10 were 1.85, 1.98 and 2.06 eV, respectively, which were determined by the difference between the HOMO and the LUMO energy levels of the polymers.

3.3. *Photovoltaic Properties*

Devices were fabricated a blend of polymer/PCBM (1:2) and the concentration of polymer solution was kept at 5 mg/mL. The photovoltaic parameters of PSCs based on polymer:PCBM blend films and *J*-V curves were depicted in Table 2 and Figure 5, respectively.

Table 1. Optical absorption and electrochemical properties of polymers

Polymer	λ_{max} (nm)	E_g^{opt}(eV)	HOMO (eV)	LUMO (eV)	E_g^{cv}(eV)[a]
TBO	608/663	1.74	−5.53	−3.68	1.85
TBO-I 5	606	1.75	−5.62	−3.64	1.98
TBO-I 10	603	1.76	−5.68	−3.62	2.06

[a] The electrochemical band gap was calculated according the equation: E_g^{cv} = LUMO − HOMO

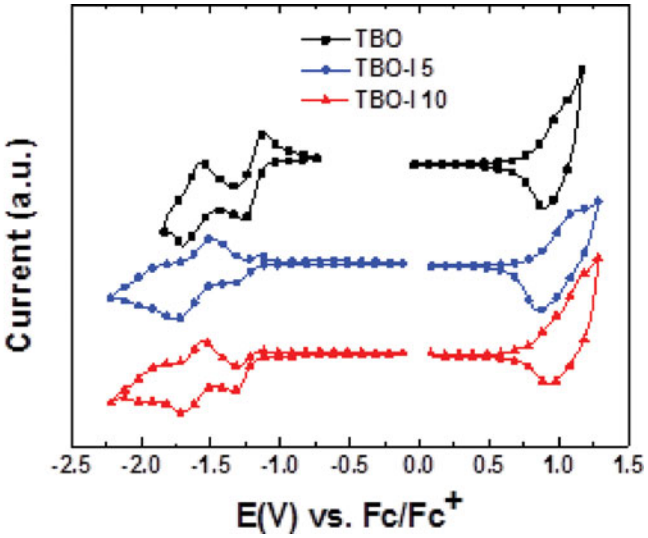


Figure 4. Cyclic voltammograms of polymers.

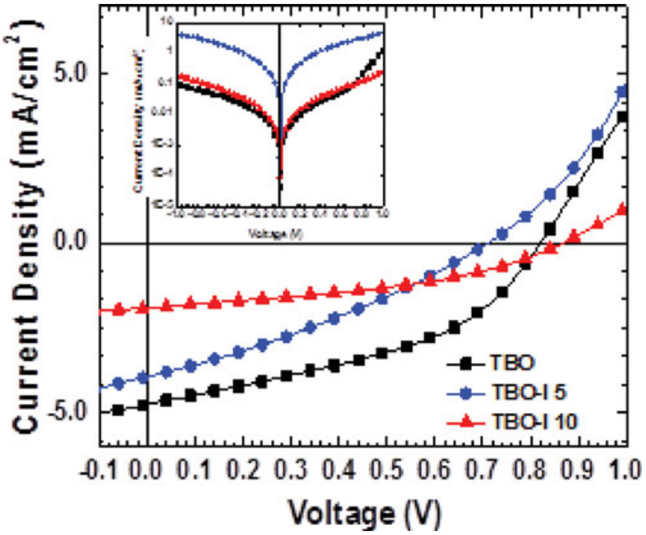


Figure 5. *J*-V curves of conventional type PSCs based on TBO, TBO-I 5 and TBO-I 10 under the illumination of AM 1.5G, 100 mW/cm^2.

Table 2. Photovoltaic parameters of PSCs based on polymer:PCBM blend films

Polymer:PCBM	J_{sc} (mA/cm^2)	V_{oc} (V)	FF (%)	PCE (%)
TBO	−4.74	0.82	42.4	1.66
TBO-I 5	−3.94	0.71	30.8	0.86
TBO-I 10	−1.90	0.87	40.1	0.66

The devices prepared from PCBM blended with TBO, TBO-I 5 and TBO-I 10 exhibit the V_{oc} of 0.82, 0.71 and 0.87 V, respectively; which are related to the difference between the HOMO energy level of the polymer and the LUMO of PCBM. The device with TBO-I 10 shows a higher V_{oc} than that of the device based on TBO which benefits from its a lower-lying HOMO energy level. Surprisingly, the devices based on TBO-I 5 display a lower V_{oc} than that of TBO, even though TBO-I 5 has lower-lying HOMO energy level. It is probably due to the difference in V_{oc} of devices is not only affected by their HOMO levels but also other factors such as morphology and carrier recombination rate [18]. The J_{sc} of the devices based on polymers were in order of TBO (4.74 mA/cm^2) $>$ TBO-I 5 (3.94 mA/cm^2) $>$ TBO-I 10 (1.90 mA/cm^2). This might be ascribed to the blue-shifted in absorption spectra and twisting in polymer main chain of TBO-I polymers which could lead to a large band gap, low charge carrier mobility. Besides, TBO has a higher molecular weight than those of TBO-I 5 and TBO-I 10. It has been reported that the low J_{sc} of solar cells fabricated using a low molecular weight polymer originates mainly from the reduced charge carriers mobility in the donor phase of the heterojunction [19]. The relatively low V_{oc} and FF of TBO-I 5 based devices indicate that the lack of optimization in morphology, inter-penetrating network which affect to charge recombination in the bulk of active layer [20]. The disadvantage in absorption spectra and polymer main chain of polymer containing phthalimide leads to low photovoltaic performances. PCE of TBO, TBO-I 5 and TBO-I 10 are 1.66, 0.86 and 0.66%, respectively.

4. Conclusions

A series of D-A polymers based on thiophene, BO and phthalimide with different ratios were synthesized by the palladium (0)-catalyzed Stille coupling reaction. The polymers exhibited good solubility in common organic solvents and good thermal stability. This work demonstrated that the optical, electrochemical and photovoltaic properties of polymers could be easily tuned by incorporating different acceptor moiety ratios into the polymer backbone. With increasing the composition of phthalimide, the absorption spectrum of polymer was blue-shifted due to the weaker electron accepting ability of phthalimide unit compared to that of BO unit. Besides, the HOMO and LUMO energy levels of polymers were shifted by introducing phthalimide and in the range of $-5.53 \sim -5.68$ eV and $-3.62 \sim -3.68$ eV, respectively. The blue-shifted absorption spectra and less ordered structure of TBO-I 5 and TBO-I 10 mainly contributed to low performances of devices based on these polymers. The result showed that the device based on TBO:PC$_{61}$BM (1:2, w/w) displayed the best photovoltaic performance with a PCE of 1.66%, a V_{oc} of 0.82 V, an J_{sc} of 4.74 mA/cm^2, and an FF of 42.4%, under the illumination of AM 1.5G, 100 mW/cm^2.

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