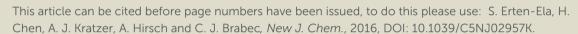


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ARTICLE TYPE

Perovskite Solar Cells using Dicarboxylic Fullerene Derivative

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⁵ Perovskite solar cells were first fabricated in dye sensitized solar cells. But also, perovskite hybrid solar cells were demonstrated to be among the most promising candidates within the emerging photovoltaic materials with their high power conversion efficiencies and low-cost fabrication. In this work, we design and synthesize novel benzoic acid fullerene bis adduct material (BAFB) for the use in perovskite hybrid organic-inorganic solar cells. The obtained maximum efficiency is reported 9.63 % using novel Benzoic acid fullerene bis adduct (BAFB) for perovskite heterojunction solar cells.

Introduction

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Perovskite heterojunction solar cells have attracted considerable attention recently because of their high solar cell efficiency besides their unique properties such as high absorption 15 coefficient, excellent ambipolar charge mobility and small exciton binding energy. 1-10 Kojima et al., first published liquid electrolyte dye sensitized solar cells of perovskite material with 3.8 % PCE solar cell efficiency. Then, solid state dye sensitized solar cell efficiency was reported as 10 %. And today perovskite solar cell 20 was known as highly efficient solar cells. The power conversion efficiencies (PCE) of organometal halide perovskite solar cells have increased from 3.8 % to 20.1%. In other words, the power conversion efficiencies of perovskite solar cells have increased in the past five years. It is very high efficiency for new generation 25 organic photovoltaic technology because efficiency of perovskite solar cells approach the efficiency of commercialized crystalline Si solar cells. The first perovskite solar cells was implemented in dye sensitized solar cells. But, perovskites have also been applied to heterojunction solar cells in low temperature solution 30 processes. The configuration is quite similar to organic photovoltaic cells which consists easy solution processed fabrication techniques. Planar structured perovskite solar cells were fabricated with the efficiency of 10 % by controlling interface engineering. Planar structure is a facile fabrication for 35 low temperature solution processed solar cells. 20-25 The preparation of mesoporous TiO2 is omitted in the planar heterojunction device structure.²⁵⁻³⁰ In the planar structure, mesoporous TiO2 replaces with the hole transport PEDOT:PSS layer, and hole transport spiro-OMETAD replaces with the 40 PCBM layer. Perovskite light absorbing layer is sandwiched between the hole- and electron transporting layers in the planar heterojunction perovskite solar cells. It is very important to control the carrier behaviours and the interface in the whole perovskite solar cells. 30-31 PEDOT:PSS layer is commonly used as 45 hole transport layer in heterojunction solar cells due to its good conductivity and solution processability. Although

mesoporous TiO2 based solid state perovskite solar cells employ the most efficient solar cells, this type of solar cells needs high temperature sintering process at 450 °C. This high temperature 50 process effects the cost production of new generation solar cells and prevents the application in plastic substrates. For this reason, planar heterojunction perovskite solar cells supply an alternative approach for efficient perovskite solar cells with the sandwich geometry between the hole transport layer PEDOT:PSS and 55 electron transport layer [6,6]-phenyl-C₆₁-butyricacid methyl ester (PCBM). This alternative fabrication provides facile operation of solution processed perovskite solar cells. However, the alternative heterojunction perovskite solar cells have not yet reached the device efficiency of conventional high temperature process. One 60 developing step in planar perovskite heterojunction solar cells is to find a good electron acceptor fullerene material instead of PCBM material to control the surface passivation of perovskite and interface properties. 32,33 For this reason, novel Benzoic acid fullerene bis adducts (BAFB) was designed and synthesized for 65 planar heterojunction perovskite solar cells in this paper. Optoelectronic, electrochemical and photovoltaic properties of novel fullerene material have been studied. Novel Benzoic acid fullerene bis adducts (BAFB) pays significantly more attention for engineering perovskite heterojunction solar cells to passivate the 70 defects on surface and grain boundaries of perovskite films. Our photovoltaic results show that Benzoic acid fullerene bis adduct (BAFB) compound is highly promising for the application in heterojunction perovskite solar cells because of its close solar cell efficiency to PCBM material.

Experimental Section

Materials

All chemicals and solvents obtained from suppliers were used without further purification. Reactions were monitored by thin layer chromatography using Merck TLC Silica gel 60 F₂₅₄.

Chromatography on silica gel was performed over Merck Silica gel 60 (particle size: 0.040-0.063 mm, 230-400 mesh ASTM).

Characterization

5 NMR spectroscopy was conducted using Bruker Avance 300 spectrometer. NMR-solvents were purchased from Deutero. The chemical shifts are reported in part per million (ppm) and referenced to the residual solvent. Spectral splitting patterns are designated as "s" (singlet), "d" (doublet), "m" (multiplet). The 10 raw date was processed using MestReNova Lite.

Mass spectrometry (MS) was done on a Shimadzu AXIMA Confidence MALDI-TOF MS-spectrometer (nitrogen UV-laser, 50 Hz, 337 nm). ESI mass spectrometry was carried out on a Bruker maxis 4G UHR TOF MS/MS-Spectrometer.

¹⁵ H NMR spectra were recorded on Bruker DPX 300 (300 MHz) spectrometer and chemical shifts are reported as δ values (ppm) and referenced to residual ¹H signals in deuterated solvents.

Absorption profiles were recorded with a Perkin-Elmer Lambda-35 absorption spectrometer from 350 to 1100 nm. CV $_{20}$ measurements were performed with an Metrohm μ Autolab III/FRA2 potentiostat/galvanostat.

Synthetic Details

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4-carboxybenzaldehyde (0.100 g, 0.666 mmol, 2.0 eq.), C₆₀ (0.240 g, 0.333 mmol, 1.0 eq.), and sarcosine (0.059 g, 0.333 25 mmol, 2.0 eq.) were refluxed overnight under an argon atmosphere in dry toluene (200 mL). The solvent was removed by rotary evaporation under reduced pressure. The crude product was filtered and subsequently purified by column chromatography (SiO₂, toluene → toluene/THF 2:1) to afford a bright brown solid 30 (111 mg, 0.103 mmol, 31%).

¹H-NMR (CDCl₃, 400 MHz): δ [ppm] = 2.78(s), 3.5–3.5(m), 3.62-3.67(m), 3.79-3.82(m), 4.25-4.27(m), 4.43-4.46(m), 4.97-4.99(m), 7.85–7.93(m), 8.09–8.11(m).

¹³C-NMR (CDCl₃, 100 MHz): δ [ppm] = 39.9, 61.7, 64.2, 69.1, 69.2, 70.0, 70.3, 70.5, 70.6, 70.6, 72.5, 83.1, 129.4, 130.1, 130.3, 135.7, 136.0, 136.5, 137.0, 139.6, 140.0, 140.3, 140.3, 141.6, 141.8, 141.9, 142.0, 142.1, 142.1, 142.2, 142.3, 142.5, 142.6, 40 142.8, 143.1, 143.2, 144.4, 144.5, 144.6, 144.8, 145.3, 145.3, 145.4, 145.4, 145.5, 145.6, 145.6, 145.8, 145.9, 146.0, 146.2, 146.2, 146.3, 146.3, 146.6, 147.4, 147.4, 152.8, 153.0, 153.9, 156.1. 166.4.

45 **IR** (ATR, diamond): $[cm^{-1}] = 3446$, 2945, 2872, 2780, 1717, 1700, 1611, 1275, 1099, 668.

UV/Vis (CH₂Cl₂): $\lambda_{\text{max}} = 228$, 264 (sh), 430 nm.

50 **MS** (MALDI, dctb): $m/z = 1074 \text{ [M}^{+}\text{]}.$

HRMS (APPI, toluene): calcd. for $C_{80}H_{22}N_2O_4$ [M⁺]: 1074.18986, found: 1074.1574.

55 ¹H NMR, ¹³C NMR and Mass Spectra of BAFB were available in Electronic Supporting Information (ESI).

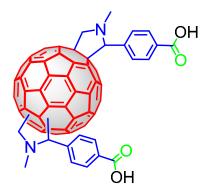


Figure 1. Molecular Structure of Benzoic acid fullerene bis adducts (BAFB)

70 Figure 2. Synthetic procedure of Benzoic acid fullerene bis adducts (BAFB) derivative

Electrochemistry

Cyclic voltammetry measurement of novel fullerene dicarboxylic 75 fullerene derivative was taken by using CH-Instrument 660 B Model Potentiostat equipment. Solution mixture was used o-DCB/ MeCN (4:1). Solution was prepared 10⁻³ M in this solution mixture. A three electrodes-cell was used consisting of Glassy carbon working electrode, Pt wire counter electrode and Ag/AgCl 80 reference electrode, all placed in a glass vessel. Tetrabutylammoniumhexafluorophosphate (TBAPF₆), 0.1 M, was used as supporting electrolyte. Ferrocene was used as internal reference electrode.

Perovskite Solar Cell Device Characterization

85 J-V curves were measured using a source measurement unit from Bo-Test with a Newport Sol1A solar simulator. The EQE spectra were measured using an Enli Technology (Taiwan) EQE measurement system (QE-R). A special mask was used to define active area in the devices and measurements were carried out

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using this special mask. Reproducibility of measurements was checked for many times for the accuracy and precision.

Materials and Preparation:

⁵ PEDOT:PSS and PC₆₁BM (99.5%) were purchased from Clevious and Solenne BV,respectively. PbI₂ and Methyl ammonium iodide (CH₃NH₃I) were purchased from Sigma Aldrich and Dyenamo, respectively.

Device Fabrication:

10 Patterned ITO glass plates were sequentially ultrasonic cleaned with acetone and isopropanol for 20 minutes. Then, the cleaned ITO substrates were spin-coated with PEDOT:PSS and followed by annealing at 140 °C for 10 min in ambient air. PbI₂ and CH₃NH₃I were mixed in a mixture of DMF and DMSO (2:1 v/v) 15 with molar ratio of 1:1 with concentration of 40 wt.%. The solution was spin-coated on to the ITO/PEDOT substrate at 4000 rpm for 35 sec. Then, the substrates were annealed at 100°C for 10 minutes. After that, 10 mg/ml BAFB, PCBM in chlorobenzene solution was solved overnight then solutions filtered using 0.45 20 µm pore size filter. Filtered solutions were coated on to the perovskite layer at 1000 rpm for 30 sec. Subsequently, ZnO solution (nanoparticle suspensions, provided by NanoGrade) was deposited by spin-coating at 2000 rpm. Work function of ZnO is closed to Ag metal electrode work function, n-type ZnO was used 25 for better electron injection to Ag electrode. Finally, 100 nm Ag were thermally evaporated on top. The active area of this electrode is 10.4 mm².

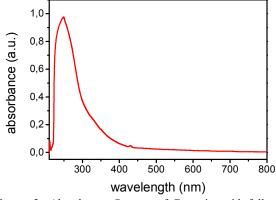
30 Results and Discussion

Optical and Electrochemical Properties of BAFB

The optical absorption spectra of Benzoic acid fullerene bis adducts (BAFB) were recorded in chlorobenzene solution at 25 °C in Figure 3. Absorption of Benzoic acid fullerene bis adducts (BAFB) compound displayed a typical bis adduct derivative absorption pattern at 311 nm that shows weak absorption in visible region and band offset was ended at 450 nm.

Cyclic voltammogram of Benzoic acid fullerene bis adducts (BAFB) derivative has been shown in Figure 4. The table of reduction potentials of Benzoic acid fullerene bis adducts (BAFB) derivative has been presented in Table 1. Cylic voltammogram of BAFB was taken in *o*-DCB/MeCN (4:1) solution mixture. Also, PCBM was taken in the same condition for better comparison of reduction potentials. The lowest unoccupied molecular orbital, bt E_{LUMO} was calculated from the onset potential (Eonset-red) obtained from cylic voltammogram (Figure 4). The E_{LUMO} energy level of BAFB is 3.86 eV, which is raised by 0.04 eV in comparison to that of PCBM (3.90 eV) in the same condition. Our results shows that LUMO level of novel synthesize fullerene derivative, BAFB is only 0.04 eV lower than PCBM material. The

results show that this LUMO value is enough like PCBM material for better electron transfer processes for perovskite solar cells. Schematic energy diagram was also presented in Figure 5.



55 Figure 3. Absorbance Spectra of Benzoic acid fullerene bis adducts (BAFB) derivative in chlorobenzene in DCM

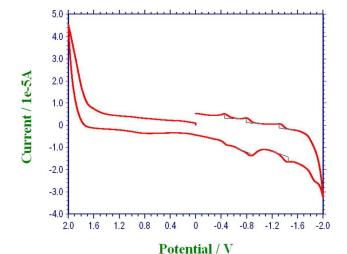


Figure 4. Cyclic Voltammetry Measurements of Benzoic acid fullerene bis adducts (BAFB) derivative

Table 1. Redox Potentials of Benzoic acid fullerene bis adducts (BAFB) derivative

	E ¹ _{red} (V)	E _{red} (V)	E ³ _{red} (V)
BAFB	-0.47	-0.83	-1.38
PCBM	-0.43	-0.82	-1.33

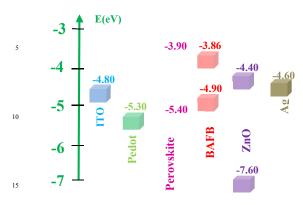


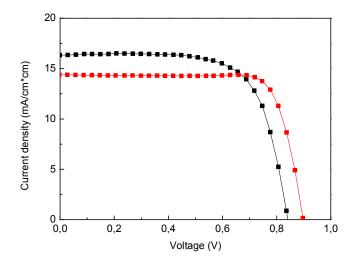
Figure 5. Energy Level Diagrams of Perovskite Solar Cell from the electrochemical data in Table 1.

20 Photovoltaic Performances

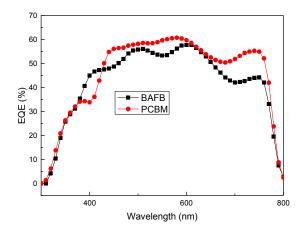
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After investigation of cyclic voltammogram of novel benzoic acid fullerene bis adducts (BAFB), E_{LUMO} level of BAFB was determined as 3.86 eV, which is raised by 0.04 eV in comparison 25 to that of PCBM (3.90 eV). We constructed perovskite bulk heterojunction solar cell device using BAFB material and PCBM as a reference cell at the same conditions. The device architecture consists of indium doped tin oxide (ITO)-coated glass, spincoated PEDOT:PSS, perovskite absorber, Benzoic acid fullerene bis 30 adducts (BAFB), ZnO layer with an evaporated silver contact ITO/PEDOT:PSS /Perovskite/ BAFB/ZnO/Ag. The currentvoltage (J-V) curve of the perovskite bulk heterojunction solar cells was presented in Figure 6. The detailed solar cell performance parameters were summarized in Table 2. The 35 perovskite bulk heterojunction solar cells were prepared in an ITO/PEDOT:PSS/Perovskite layer/ Benzoic acid fullerene bis adducts (BAFB)/ ZnO/ Ag device geometry. Also the reference cell was prepared in an ITO/PEDOT:PSS/Perovskite layer/PCBM/ ZnO/ Ag device geometry. The efficiency for 40 reference cell was displayed a short circuit current density (Jsc) of 15.3 mAcm⁻², an open circuit voltage of 0.87 V, a fill factor of 76.40 % and leading to an overall maximum power conversion efficiency of 10.27 %. The perovskite heterojunction solar cells using benzoic acid fullerene bis adducts (BAFB) presented an 45 overall maximum power conversion efficiency of 9.63 %, a short circuit current density (Jsc) of 16.2 mAcm⁻², an open circuit voltage of 0.82 V, a fill factor of 69.1 %. Jsc, short circuit current density of Benzoic acid fullerene bis adduct (BAFB) based perovskite heterojunction solar cells is 16.2 mAcm⁻². This value is 50 1.05 times higher than PCBM based perovskite heterojunction solar cells (15.3 mAcm⁻²). However, fill factor of PCBM based device was found as 76.40%. Wheras, fill factor of Benzoic acid fullerene bis adducts (BAFB) is 69.10 %. We have worked surface passivation on perovskite surface using dicarboxylic 55 group on fullerene. The carboxylic group may form hydrogen bond with I ion in the perovskite and passivate the surface of perovskite, thus reducing the recombination. 33 Our results show that the efficiency of reference perovskite bulk heterojunction solar cell using PCBM is higher than 1.066 times that of

bis adducts (BAFB). The short circuit current, open circuit voltage and other parameters were obtained from BAFB and reference PCBM perovskite heterojunction solar cells were tabulated with error bars for better photovoltaic performance 65 comparisons in the Table 2. Also, the external quantum efficiency (EQE) spectrum presented for BAFB and PCBM materials in Figure 7. The EQE spectrum gives us information about the response of the active layer upon illumination with the monochromatic light in a large range of wavelengths. In the EQE 70 spectrum, it is shown 55% efficiency in the range between 400-800 nm. The EQE efficiencies of BAFB and PCBM materials are equal at 600 nm. Panchromatic nature of active layer was demonstrated in EQE spectrum. Figure 8 shows the atomic force microscopy image (AFM) of device structure (ITO /PEDOT:PSS 75 /Perovskite/ BAFB/ ZnO/Ag) in wave mode (a) and phase mode (b). AFM image also depicts a uniform deposition along the length of device. Rms value of the perovskite heterojunction solar cell device was found as 11.84 nm.



80 Figure 6. J-V curves of ITO/ PEDOT/ CH₃NH₃PbI₃/ PCBM/ ZnO/ Ag solar cells (red) and ITO/ PEDOT/ CH₃NH₃PbI₃/ BAFB/ ZnO/ Ag solar cells (black) under simulated AM 1.5 solar irradiation at 100 mW cm⁻².



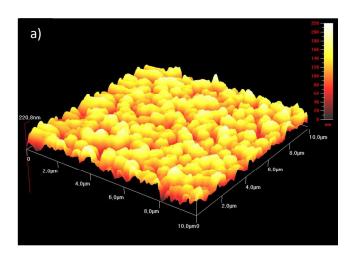
85 Figure 7. The external quantum efficiency (EQE) curves of

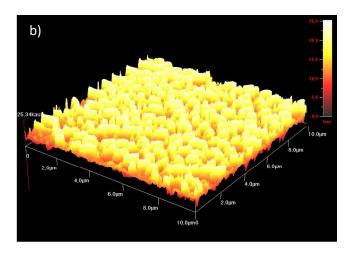
60 perovskite heterojunction solar cells using benzoic acid fullerene

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Table 2. Photovoltaic Performance of Perovskite Solar Cells.

	J_{sc} (mAcm ⁻²)	$V_{oc}\left(\mathbf{V}\right)$	ff	η (%)
BAFB	16.2 ±0.11	0.822±0.01	69.1±1.5	9.21±0.42
PCBM	15.3±1	0.868±0.03	76.4±3	10.13±0.14





¹⁰ **Figure 8.** AFM picture of ITO/PEDOT/Perovskite/BAFB/Ag device, in wave mode (a), in phase mode (b)

15 Conclusion

In conclusion, novel fullerene material based heterojunction perovskite solar cells have been studied. Full material characterizations of BAFB were also supplied (¹H NMR, ¹³C NMR and Mass spectra). Optical, electrochemical and 20 photovoltaic properties were also investigated. Though the absolute conversion efficiency reported here are well below those of solid state dye sensitized solar cells, this novel BAFB material represents a promising efficiency for perovskite heterojunction solar cells because of its close solar cell efficiency to phenyl-C61-25 butyric acid methyl ester material. We investigated the replacement of phenyl-C61-butyric acid methyl ester material with this novel Benzoic acid fullerene bis adducts (BAFB) compound. Our results show that the efficiency of Benzoic acid fullerene bis adducts (BAFB) based perovskite heterojunction 30 solar cells is 9.63% and a short circuit current density is 16.2 mAcm⁻². Phenyl-C61-butyric acid methyl ester based perovskite heterojunction solar cells displayed an efficiency as 10.27 % and 15.3 mAcm⁻² of a short circuit current density.

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Notes and references

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Perovskite solar cells were fabricated using novel benzoic acid fullerene bis adduct material (BAFB). BAFB material was found as a promising material for the use in perovskite hybrid organic-inorganic solar cells. The efficiency was reported 9.63 % for the perovskite solar cell.

