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Synthesis of new dithieno[3,2-b:2′,3′-d]pyrrole (DTP) units for photovoltaic cells

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ABSTRACT

Three new dithieno[3,2-b:2′,3′-d]pyrrole units (DTPA1–3) based on the truxene core have been designed and synthesized for a potential application in photovoltaic cells. One of these units (DTPA1) has been employed to construct a new organic dye (M47), exhibiting a higher solar-to-electrical energy conversion efficiency when compared to reference dyes (M48 and M49) under the same conditions. Absorption spectra, electrochemical cyclic voltammetry, theoretical calculations, current voltage curves and controlled intensity modulated photovoltage spectroscopy have been investigated. M47 sensitized solar cells employing a cobalt electrolyte afford a short circuit photocurrent of 12.1 mA cm^{-2}, an open circuit voltage of 758 mV, and a fill factor of 0.56, corresponding to an overall power conversion efficiency of 5.1% under standard AM 1.5 sunlight. These results suggest that the truxene-substituted dithieno[3,2-b:2′,3′-d]pyrrole derivatives are promising candidates for photovoltaic cells.

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1. Introduction

Since Grätzel and O'Regan significantly improved the efficiency of dye-sensitized solar cells (DSCs) in 1991 [1], large research efforts have been put towards realization of this cheap alternative to today's commercially available solar technologies. Upon photoexcitation, electron transfer from the photosensitizers into the conduction band of TiO_2. The photosensitizer, therefore, has a pivotal role in the cell conversion efficiency. Besides the ruthenium [2] and porphyrin [3] dyes, metal-free dyes have been demonstrated as efficient light-harvesters for DSCs owing to synthetic feasibility, low cost, and the strong light harvesting capacity [4–46]. To date, the efficiency of DSCs based on metal free dyes has reached more than 12% power conversion efficiency (PCE) [47–50].

Recently, tremendous research has been focused on the design and synthesis of new coplanar π-linkers. It has been found that incorporation of additional electron-withdrawing units into the π bridge as internal acceptors, termed the D–A–π–A configuration, distinct improvement of photovoltaic performance and stability [51]. On the other hand, fused-ring thiophenes [4] and multifused thiophenes [52] have been widely exploited as crucial building blocks for organic dyes, since better π-conjugation can be readily achieved by increasing the coplanarity of bridged π-systems.

In the previous work, we have developed a series of dithieno[3,2-b:2′,3′-d]pyrrole (DTP) segments for metal free organic dyes [53–55], resulting in good photovoltaic performances. Apart from their electron richness, the DTP units are attractive as π bridges because modification of the substituent is readily achieved, which may produce new photovoltaic materials with interesting properties. In this contribution, we report the design and synthesis of three novel DTP segments DTPA1–3 by the Buchwald–Hartwig amination. These building blocks can be used for rational design of conjugated organic polymers and photosensitizers toward Field effect transistors (FETs), organic photovoltaic (OPV) devices, and light-emitting diodes (LEDs). Moreover, DTPA1–3 offer a way to construct new materials with rod-, cone- and starburst-shaped

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configuration. Herein, DTPA1 was employed as the π bridge to synthesis a new D–π–A organic sensitizer M47. To further assess the merit of DTPA1 in terms of photovoltaic performance, we also prepared its counterparts M48 and M49 (Fig. 1) bearing the hexyl-substituted DTP (HDTP) and 2,2′-bithiophene spacer, respectively, as references.

2. Experimental

2.1. Materials and instruments

The synthetic routes for DTPA1–3 and M47–49 are shown in Scheme 1. BTNAP, Xphos, P(*t*-Bu), *t*-BuONa and cyanoacetic acid were purchased from Energy Chemical (China). $Pd_2(dba)_3$ was purchased from J&K Chemical. All other solvents and chemicals used in this work were analytical grade and used without further purification.

^1H NMR and ^{13}C NMR spectra were recorded on a Bruker AM-300 or AM-400 spectrometer. The reported chemical shifts were against TMS. High resolution mass spectra (HRMS) were obtained with a Micro mass GCT–TOF mass spectrometer. The melting point was taken on a RY-1 thermometer and temperatures were uncorrected.

2.2. Optical and electrochemical measurements

The UV–vis spectra of dyes and sensitized films were measured by SHIMADZU UV-2600 spectrophotometer. Fluorescence measurements were carried out with a HITACHI F-4500 fluorescence spectrophotometer.

Cyclic voltammetry (CV) measurements for sensitized films were performed on a Zennium electrochemical workstation (ZAHNER, Germany), with sensitized electrodes as the working electrode, Pt-wires as the counter electrode, and an Ag/AgCl electrode as the reference electrode at a scan rate of 10 mV s^{-1}. Tetrabutylammonium perchlorate (TBAP, 0.1 M) and MeCN were used as supporting electrolyte and solvent, respectively. The results were calibrated using ferrocene as standard.

Electrochemical impedance spectroscopy (EIS) in the frequency range from 200 mHz to 100 kHz was performed with a Zennium electrochemical workstation (ZAHNER, Germany) in the dark with the alternate current amplitude set at 10 mV.

Charge densities at open circuit and intensity modulated photovoltage spectroscopy (IMVS) were performed on a Zennium electrochemical workstation (ZAHNER, Germany), which includes a green light emitting diode (LED, 532 nm) and the corresponding control system. The intensity-modulated spectra were measured at room temperature with light intensity ranging from 5 to 75 W m^{-2}, in modulation frequency ranging from 0.1 Hz to 10 kHz, and with modulation amplitude less than 5% of the light intensity.

2.3. Fabrication and characterization of DSCs

The TiO_2 paste (particle size, 20 nm) was printed on a conducting glass (Nippon Sheet Glass, Hyogo, Japan, fluorine-doped SnO_2 over layer, sheet resistance of 10 Ω/sq) using a screen printing technique. TiO_2 particle used in this work was synthesized according to the method reported by Ito et al. [56]. The film was dried in air at 120 °C for 30 min and calcined at 500 °C for 30 min under flowing oxygen before cooling to room temperature. The heated electrodes were impregnated with a 0.05 M titanium tetrachloride solution in a water-saturated desiccator at 70 °C for 30 min and fired again to give a ca. 3 μm (for cobalt cells) thick mesoscopic TiO_2 film. The TiO_2 electrode was stained by immersing it into a 0.3 mM dye solution in a mixture of DCM/EtOH (v/v, 1:1) and kept at room temperature for 12 h. The sensitized electrodes were then rinsed with dry ethanol and dried by a dry air flow. Pt catalyst was deposited on the FTO glass by coating with a drop of H_2PtCl_6 solution (40 mM in ethanol) with the heat treatment at 395 °C for 15 min to give photoanode. The sensitized electrode and Pt-counter electrode were assembled into a sandwich type cell by a 25 μm-thick Surlyn (DuPont) hot-melt gasket and sealed up by heating. The Co-bpy electrolyte, is composed of

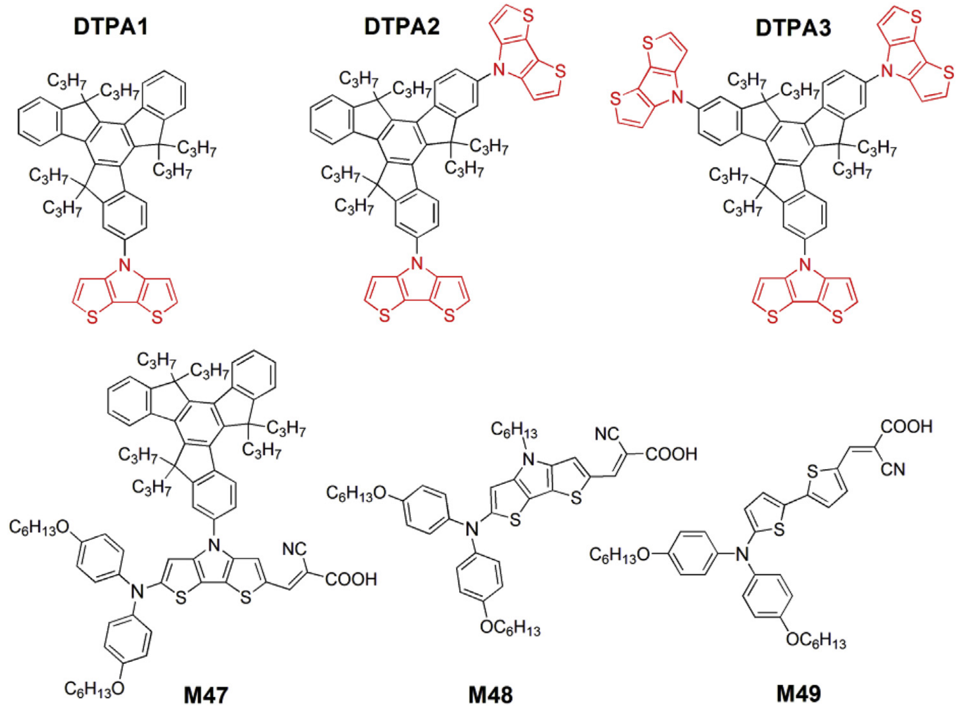

Fig. 1. Chemical structures of DTPA1–3 and M47–49.

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