Synthesis and characterization of porphyrin sensitizers with various electron-donating substituents for highly efficient dye-sensitized solar cells†

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A series of porphyrin dyes with an electron-donating group (EDG) attached at a meso-position (YD1–YD8) have been designed and synthesized for use as sensitizers in dye-sensitized solar cells (DSSC). The nature of the EDG exerts a significant influence on the spectral, electrochemical and photovoltaic properties of these sensitizers. Absorption spectra of porphyrins having an amino group show broadened Soret band and red-shifted Q bands with respect to those of reference porphyrin YD0. This phenomenon is more pronounced for porphyrins YD7 and YD8 that have a π-conjugated triphenylamine at the meso-position opposite the anchoring group. Upon introduction of an EDG at the meso-position, the potential for the first oxidation alters significantly to the negative whereas for the first reduction changes inappreciably, indicating a decreased HOMO-LUMO gap. Results of density-functional theory (DFT) calculations support the spectroelectrochemical data for a delocalization of charge between the porphyrin ring and the amino group in the first oxidative state of diarylamino-substituted porphyrins YD1–YD4, which exhibit superior photovoltaic performance among all porphyrins under investigation. With long-chain alkyl groups on the diarylamino substituent, YD2 shows the best cell performance with $J_{sc} = 13.4\ mA\ cm^{-2}$, $V_{oc} = 0.71\ V$, and $FF = 0.69$, giving an overall efficiency 6.6% of power conversion under simulated one-sun AM1.5 illumination.

Introduction

Dye-sensitized solar cells (DSSC) have attracted considerable attention over the past decade because they provide an economic alternative to silicon-based photovoltaic devices. The DSSC of ruthenium polypyridyl complexes produced an efficiency of solar-to-electric conversion up to 11% under standard global AM 1.5 solar conditions with enduring stability.1 Several authors have modified the Ru-bipyridyl complexes to increase their conversion efficiencies since the discovery of the landmark N3 dye,2 but, in view of the cost and environmental concern about ruthenium dyes, much effort has been directed to the development of organic dyes because of their modest cost, large absorption coefficient and the facile modification of their molecular structures.3

Numerous organic dyes have been synthesized for use in DSSC. Organic dyes with large conversion efficiencies are typically composed of a donor-π-conjugated unit-acceptor (D-π-A) structure with a well defined architecture.4 These sensitizers include coumarin,5 indoline,6 oligoene,7 thiophene,8 triarylamino,9 perylene,10 cyanine11 and hemicyanine12 derivatives. Inspired by the efficient energy and electron transfer in the light-harvesting antenna of biological systems,13 porphyrin derivatives14 and analogues15 have been widely used in photovoltaic devices. The intrinsic advantages of porphyrin-based dyes are their rigid molecular structures with large absorption coefficients in the visible region and their many reaction sites, i.e., four meso and eight β positions, available for functionalization. Fine tuning of their optical, physical, and electrochemical properties thus becomes feasible.

On the basis of our previous work on the systematic modification of porphyrin dyes,16 we found that the use of a phenylethynyl (PE) bridging unit between the porphyrin core and the carboxyl anchoring group via the meso-position results in broadening and red shift of absorption bands, and thus gives cell performance better than that of other bridges. In addition, porphyrin dyes substituted with a strong electron-donating diarylamino unit at the meso-position opposite the anchoring group exhibit a satisfactory cell performance comparable with that of the device made of N3 dye. To obtain insight into how the chemical and physical nature of the electron-donating groups influences the efficiency of power conversion, we systematically designed and prepared a series of porphyrin dyes with a D-π-A framework. Herein, we report the synthesis, spectral, electrochemical, and photovoltaic properties of six amine-substituted porphyrin sensitizers YD2–YD6 and YD8, and the reference dyes YD0, YD1, and YD7 (Figure 1).16

Results and discussion

Previous work showed that introduction of hydrophobic alkyl chains onto organic dyes suppresses the electron transfer from
Both the Soret and Q bands for compound YD1 having two hexyl chains on the diphenylamine is expected to act as a more efficient sensitizer in a DSSC than YD1. Alkoxyl groups are considered to be stronger electron-donating groups than alkyl groups; accordingly, porphyrins YD3 and YD4 with methoxyl and pentoxyl groups, respectively, have been synthesized. Another promising strategy to increase the electron-donating ability of the porphyrin dye is to introduce a N-substituent onto the diarylamine moiety; compound YD5 incorporated with a triamine group to the meso-position has also been synthesized. To extend the charge separation between porphyrin and TiO2, porphyrins with a triarylaminogroup, YD6–YD8, have been designed and prepared. Our previous work showed that the cell performance of YD7 is worse than that of YD1. To see if this decreased efficiency might be ascribed to dye aggregation, we prepared YD8 with four tert-butyl groups on triphenylamine. The syntheses of these porphyrin dyes are described in the supporting information.

The absorption spectra of these compounds are summarized in Table 1, and some representative examples are given in Figure 2. All porphyrin dyes in this work exhibit maxima attributed to π–π* transitions in the range 400–500 nm for the Soret band and 550–750 nm for the Q bands. The molar absorption coefficients/10^3 M^-1 cm^-1 for the Soret band of these porphyrin dyes range from 1.36 to 4.98, so fulfilling one requirement for a dye usable in a DSSC, whereas those/10^3 M^-1 cm^-1 of the Q(0,0) band are in the range 16.9–52.7. The Soret and Q bands for YD1–YD8 are broadened and red-shifted relative to those of YD0. Both Soret and Q(0,0) bands of YD1–YD8 have full widths at half maximum (fwhm) height two or three times as large as those for the reference compound YD0; this effect is ascribed to an electronic interaction between the porphyrin core and the amino group. Both the Soret and Q bands for YD1 and YD2 show oscillator strengths comparable with that of YD0, although their absorption coefficients are much smaller than those of YD0. YD5 exhibits a larger oscillator strength for both Soret and Q bands than for YD0. Among these porphyrins, YD7 and YD8 exhibit the most pronounced broadening and bathochromic shift of the Soret band, indicating that the acetylenic link effectively mediates the electronic coupling between the porphyrin and triarylamine units. Similar to the absorption, the emission is red-shifted on incorporation of an amino group onto the porphyrin macrocycle (Table 1).

We employed cyclic voltammetry to determine the redox potentials of these porphyrins; the electrochemical reactions of these compounds were measured under ambient conditions. The electrochemical data are summarized in Table 2. All porphyrin dyes exhibit reversible waves for the first oxidation, corresponding to the HOMO energy of the dye, at a potential greater than that of the I/ I^- couple, which assures regeneration of the oxidized state. Figure 3 shows representative cyclic voltammograms for YD0, YD2, YD4, YD5, YD6, and YD8 in THF containing tetrabutylammonium hexafluorophosphate (TBAPF6, 0.1 M). One reversible oxidation reaction was observed at E_1/2 = +1.04 V corresponding to the formation of [YD0]^+ whereas an irreversible reduction wave was observed at E_{pc} = −1.36 V. The potentials for the first and second oxidations of YD1–YD5 show significant cathodic shifts with increasing electron-donating ability of the amino substituent as compared to those of YD0. For example, the first oxidation occurs at +1.04 V for YD0, shifts to +0.89 V for YD2, and further shifts to +0.77 V for YD4, whereas the reduction potential occurs at E_{pc} = −1.36 V for YD0 and in a small range −1.00 to −1.20 V for YD1–YD8. Incorporation of an amino group onto the porphyrin ring decreases the electrochemical HOMO energy gap, consistent with red shifts of both Soret and Q bands in the absorption spectra. In our previous work, both porphyrin and diarylamino units were responsible for the first and second oxidations of YD1 because the electron density of HOMO and HOMO-I is distributed to these two units. Similarly, the...
first and second oxidations for YD2–YD4 involve charge delocalization through the porphyrin ring and diarylamino moiety, which is further confirmed by DFT calculations.

The cyclic voltammogram of YD5 shows four oxidation waves at \( E_{1/2} = +0.60, +0.80, +1.29 \) and +1.46 V, respectively. The oxidations of the triamine component occur at +0.60, +0.80 and +1.28 V. The first and second oxidation centers for YD5 are thus assignable to the triamine unit, consistent with DFT calculations that show the HOMO and HOMO-1 to be located on the donor subunit. The third and fourth oxidations at +1.29 and +1.46 V might involve both porphyrin and triamine units. Compound YD6 shows two reversible couples at +0.88 and +1.05 V corresponding to oxidations from the triarylamine unit and the porphyrin ring, respectively. The irreversible wave at +1.49 V is assigned to the second electron abstraction from the porphyrin core. Compounds YD7 and YD8 exhibit a similar electrochemical behaviour with two reversible oxidations occurring about +0.90 and +1.05 V. Our previous electrochemical measurements on YD7 showed that the first oxidation occurs at the triarylamine unit and the second electron abstraction corresponds to the oxidation of the porphyrin ring.\(^{15}\)

The excited-state oxidation potentials \( (E_{0.0}^*) \) are obtained from the relation \( E_{0.0}^* = E_{ox1} - E_a0 \), in which \( E_{ox1} \) is the first oxidation potential of a porphyrin dye and \( E_a0 \) is the zero-zero excitation energy obtained from the absorption edge.\(^{15,16}\) The energy levels of these porphyrins are depicted in Figure 4. The calculated \( E_{0.0}^* \) values are all more negative than the conduction edge (–0.50 V vs. NHE) of TiO\(_2\), which is compatible with electron injection from the excited state of the dye to the conduction band.
the porphyrin ring. The separation between the a1u and a2u emission upon introduction of an electron-donating group onto the porphyrin core, which is in accordance with the tendency on the HOMO-LUMO gaps shown in Figures 4 and 5 because the electron correlation and the solvent effect have not been taken into account. Figure 4 shows a reduced HOMO-LUMO gap upon incorporation of an electron-donating group to the porphyrin ring, which is in accordance with the tendency on the change of the HOMO-LUMO gap calculated from DFT.

In the electronic absorption for a porphyrin, both Soret and Q bands arise from σ-σ* transitions, which can be explained by considering the Gouterman four-orbital model: two π orbitals (a1u and a2u) and two degenerate π* orbitals (εp and εp). In compounds YD1–YD8, there is considerable electronic coupling between the electron-donating group and the porphyrin core, thus decreasing the HOMO-LUMO energy gaps relative to YD0. This effect is consistent with the red shift in absorption and emission upon introduction of an electron-donating group onto the porphyrin ring. The separation between the a1u and a2u orbitals increased upon introduction of a donor group whereas the energy splitting of the εp/εp* pair was only slightly perturbed.

According to our previous work, the electronic density of YD1 is significantly distributed into the π-system of the porphyrin ring and the diphenylamino moiety at the HOMO and HOMO-1. Introduction of a more strongly electron-donating group would increase the electronic density on the diarylamino moiety. The HOMO of YD3 shows an increased electron distribution located on the electron-donating diarylamino relative to YD2. Similar to the LUMO of YD2, the π-conjugation is extended to only the porphyrin ring and the phenylene link at the LUMO of YD3. Comparison of YD1, YD3 and YD5 with YD0 shows that increasing the electron-donating ability of the amino substituents results in a decreased HOMO-LUMO gap, which is ascribed to the significantly elevated HOMO as the LUMO is moderately altered. The HOMO electronic densities of YD5 and YD6 are significantly distributed on the triamine and triarylamine moieties, respectively; the LUMO shows an electronic distribution on both the porphyrin and phenylene units. In the case of YD7, for which the triarylamine is attached at the meso-position through an acetylene link, the distribution of electronic density of the molecular orbitals is similar to that of YD6.

The primary step of charge separation in DSSC is electron injection from the excited state of the dye into the conduction band of TiO2, which generates the dye cation. To improve our understanding of the electronic absorption properties of the oxidized species, we investigated the electrochemical reactions of these porphyrin dyes with a spectroelectrochemical method. The spectroelectrochemical properties of YD1 and YD7 have been previously investigated. Upon oxidation, the positive charge of YD1 is delocalized over both the porphyrin and diarylamino units whereas that of YD7 is localized on the triarylamino moiety. According to the DFT calculations, the spectroelectrochemical behaviours of YD2–YD4 are expected to resemble that of YD1. Figure 6 shows scans of absorption spectra of YD2 as a thin layer obtained during electrochemical oxidation at an applied potential from +0.40 to +0.98 V in THF containing TBAPF6 (0.1 M) at 25 °C. As expected, the Soret band at 443 nm decreases while a band at 813 nm and a broad band at 1329 nm increase in the course of electrolysis at applied potential of +0.98 V. The band at 813 nm is characteristic of a porphyrin cation. Such a band in this region has also been observed in a bis(diphenylamino)-substituted porphyrin upon 1 e⁻ oxidation.

As shown in Figure 5, the electron density of HOMO for the YD2 is mainly distributed on the diphenylamine and porphyrin units. The charge would delocalize over the porphyrin ring and the diphenylamine moiety upon 1 e⁻ oxidation. Therefore, the broad band at 1329 nm may correspond to the intervalence state. The first oxidation is essentially reversible as more than 90% of the oxidized species generated at +0.98 V can be reconverted to the neutral form of YD2 according to the intensity of the Q band whereas the second oxidation at +1.35 V is irreversible.

Figure 7 shows the spectral changes of porphyrin YD5 at applied potentials +0.73 and +1.14 V in THF containing TBAPF6 (0.1 M) at 25 °C. In the first oxidation, the Soret band at 444 nm and the signal at 304 nm corresponding to the triamine unit decrease while those at 860 and 1404 nm increase, with clear isosbestic points. The Qx and Qy bands become visible and increase in the course of electrolysis at applied potential of +0.98 V. The band at 813 nm is characteristic of a porphyrin cation. Such a band in this region has also been observed in a bis(diphenylamino)-substituted porphyrin upon 1 e⁻ oxidation.

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Fig. 5  Energy-level diagram and the corresponding molecular orbitals of porphyrins YD0–YD8 calculated at the B3LYP/6-31G(d) level of theory.

Fig. 6  Spectral changes of YD2 in THF containing TBAPF6 (0.1 M) at applied potentials +0.98 V (top), and +1.35 V (bottom); the electrolysis time for each process is about 30 min.

Fig. 7  Spectral changes of YD5 in THF containing TBAPF6 (0.1 M) at applied potentials +0.73 V (top), and +1.14 V (bottom); the electrolysis time for each process is about 30 min.
electron abstraction from the triamine moiety. The broad bands in the near IR region for the oxidized species (~1400 nm for [YD5]+, and ~1010 and ~1400 nm for [YD5]2+)) might be attributed to both the intervalence states within the triamine unit and the charge transfer from the porphyrin ring to the triamine cation. These oxidation processes are reversible because more than 95% of the oxidized species is reconverted to the neutral form of YD5.

The spectroelectrochemical measurements of YD6 were also performed in THF under ambient conditions (Figure 8). Unlike [YD2]+ and [YD5]+ for which a broad band in the near IR region was observed, [YD6]+ shows no absorption at a wavelength greater than 800 nm. Scans of UV-visible spectra exhibit a new band at ~740 nm for the first electron abstraction; furthermore, the intensities of the Soret and Q bands decrease for the first and second oxidations. The first and second oxidations likely correspond to 1 e− abstraction from each of the triarylamino and porphyrin units, respectively, on the basis of DFT calculations.

Fig. 8 Spectral changes of YD6 in THF containing TBAPF6 (0.1 M) at applied potentials +0.98 V (top), and +1.15 V (bottom); the electrolysis time for each process is about 30 min.

Fig. 9 (a) Absorption spectra of films with no added scattering layer (film thickness ~10 μm), (b) incident photon-to-current conversion efficiency (IPCE) spectra of devices, and (c) current-voltage characteristics of devices fabricated from YD0–YD8 sensitized on TiO2 films (film thickness ~(10 + 4) μm).

Porphyrians YD0–YD8 were sensitized onto TiO2 nanoparticulate films to serve as working electrodes of DSSC devices for photovoltaic characterization. Figure 9a shows the transmittance absorption spectra of the sensitized thin-film samples in three parts; Figures 9b and 9c show the corresponding action spectra and current-voltage curves of the devices, respectively. The thin-film samples show much broader absorption (Figure 9a) than their solution counterparts (Figure 1) because of strong intermolecular interactions of the molecules aggregated on TiO2 surface. The large absorbances of the thin-film spectra indicate that the amounts of dye loading on TiO2 films were sufficient for all porphyrin sensitizers. The IPCE action spectra (Figure 9b) reflect the photoelectric conversion efficiency at each wavelength. Although there is a large gap between the Soret and Q bands of the absorption spectra of porphyrins, this feature is not evident in the IPCE spectra because the scattering by TiO2 nanoparticles increases the photocurrents for the weak absorption in that region.

Table 3 summarizes the photovoltaic parameters derived from Figure 9c for devices YD0–YD8. The overall efficiencies of power conversion of the devices exhibit a systematic trend for porphyrins with varied meso-substituents according to three classes. First, the diarylamino groups substituted directly on the meso-position, i.e., YD1–YD4, show a cell performance much better than for our reference cell, YD0. Second, the triarylamino-substituted porphyrins, YD6–YD8, exhibit cell performance comparable to that of YD0. Third, the triamine-substituted porphyrin, YD5, displays cell performance much poorer than that of YD0. We discuss the observed photovoltaic sequence of YD1–YD8 in what follows.

Because the π-conjugation of the diarylamino groups extends the spectral absorption towards long wavelength, the photocurrents of YD1–YD4 are significantly greater than that of YD0; the long-chain hydrocarbons further extend the spectral region of light harvesting and make the performance of YD2 slightly larger than YD1 and that of YD4 slightly greater than YD3. Although both YD3 and YD4 have alkoxyl groups on the diarylamino substituent, the additional electron-donating groups increased no further the device performance. The action spectra shown in
Figure 9b indicate that the efficiency values of YD3 and YD4 are less than those of YD1 and YD2, which accounts for the J_{SC} values of the former being smaller than those of the latter. This decrease might reflect that the additional electron-donating groups raise the energy level of the HOMO (Figures 4 and 5) so that dye regeneration of the porphyrin cations from the electrolyte becomes slower. Such an effect became more pronounced when a more strongly electron-donating group was present. For example, YD5 has a triamine substituent, which is a stronger electron-donating group than a diarylamine, so that the HOMO level is the highest among all porphyrins under investigation. As a result, YD5 shows a small J_{SC} value that is confirmed by its significantly smaller efficiency through the entire action spectrum shown in the middle panel of Figure 9b.

YD7 and YD8 also have excellent \( \pi \)-conjugation between the porphyrin core and the phenyl group through the CC triple-bond bridge so that their thin-film absorption spectra are similar to those of YD1–YD4 (Figure 9a), but their device performances are poorer than those of YD1–YD4. YD7 suffered from dye aggregation in ethanol solution,\(^{16}\) additional tert-butyl groups in YD8 failed to decrease the tendency of aggregation. The smaller J_{SC} and V_{OC} of YD7 and YD8 are thus expected to be due to the effect of dye aggregation so that fewer electrons were injected into the conduction band of TiO\(_2\) after photoexcitation. In contrast, the structure of YD6 has a triarylamino group directly attached at the meso-position of the porphyrin core without a triple bond as a bridge; rotation about the bridged C–C single bond is thus feasible so that effective \( \pi \)-conjugation between the porphyrin core and the triarylamino substituent was unattainable (Figure 5). As a result, the thin-film absorption spectrum of YD6 is similar to that of YD0, but the electron-donating ability of the triarylamino group increases the efficiency of the former relative to the latter to improve the cell performance of the former.

The electron-donating feature of amino substituents in YD1–YD4 and YD6 seems to be responsible for the V_{OC} value (\( \sim 0.71 \) V) being larger than that (0.675 V) of the reference cell (YD0). Among evidence from other work, Durrant and co-workers found that the period for charge recombination of a triarylamino porphyrin/TiO\(_2\) film is 20 times that of a free-base porphyrin counterpart (80 vs. 4 ms);\(^{23}\) Mozer et al. noted that the smaller V_{OC} of porphyrin-sensitized solar cells is due to the decreased electron lifetime related to either a more rapid recombination of electrons with dye cations or I\(_3^-\) ions.\(^{24}\) We therefore expect that the observed larger V_{OC} in YD1–YD4 and YD6 compared to YD0 is due to a diminished recombination between I\(_3^-\) and conduction-band electrons, because I\(_3^-\) might be attached to the positively charged diarylamino moiety far from the TiO\(_2\) surface for the former case. For YD7 and YD8, although the amino group is farther from TiO\(_2\) than in the cases of YD1–YD4 and the electron distribution of the frontier orbitals is appropriate for their use in DSSC (Figure 5), the V_{OC} values of YD7 and YD8 (\( \sim 0.65 \) V) are smaller than those of YD1–YD4, and even smaller than those of YD0 because of the effect of aggregation.

**Conclusions**

We have synthesized new porphyrin dyes with a donor group attached at the meso-position of the porphyrin for dye-sensitized solar cells. The tuning of the HOMO-LUMO energy gap, thus the optical and electrochemical properties, is achievable on varying the structure of the donor group. The electronic interaction between the donor and the porphyrin units in YD1–YD8 significantly affects the spectral features of absorption and electrochemical properties. Our results reveal that direct attachment of an alkyl-substituted diarylamino group to the porphyrin ring results in significant improvement in solar-to-electrical conversion efficiency. This work provides a basis for the future design and synthesis of more efficient porphyrin-based DSSC.

**Acknowledgements**

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**References**


**Table 3** Photovoltaic parameters of porphyrin-based dye-sensitized solar cells under AM1.5 illumination (power 100 mW cm\(^{-2}\)) and active area 0.16 cm\(^2\).

<table>
<thead>
<tr>
<th>Porphyrins</th>
<th>J_{SC}/mA cm(^{-2})</th>
<th>V_{OC}/V</th>
<th>FF</th>
<th>( \eta ) (%)</th>
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