Upconverting nanophosphor incorporated photoanodes for improved photoelectric performances of quantum dot sensitized solar cells

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ABSTRACT
We herein provide a strategy to improve the solar cell performance by merging upconverting nanophosphors (UCNPs), Mn\textsuperscript{2+}, Yb\textsuperscript{3+}, Er\textsuperscript{3+} tri-doped NaYF\textsubscript{4} into TiO\textsubscript{2} photoanodes of quantum dot-sensitized solar cell (QDSSCs). The best performance was achieved at the UCNPs content of 8 wt\% based on the systematic investigation of the power conversion efficiency (PCE) performance dependent on UCNPs amount. The short-circuit photocurrent ($J_{sc}$) (21.30 mA cm\textsuperscript{-2}) and open circuit photovoltage ($V_{oc}$) (0.54 V) of QDSSC were obviously improved compared with those of pure TiO\textsubscript{2} photoanode ($J_{sc}$ of 14.67 mA cm\textsuperscript{-2}, $V_{oc}$ of 0.48 V) under one sun illumination, resulting in a 42.6\% enhancement in PCE.

IMPACT STATEMENT
The short-circuit photocurrent ($J_{sc}$) (21.30 mA cm) and open circuit photovoltage ($V_{oc}$) (0.54 V) of QDSSC using the upconverting nanophosphors incorporated TiO\textsubscript{2} photoelectrodes were remarkably improved under one sun illumination, resulting in a 42.6\% enhancement in power conversion efficiency.

In order to solve environmental pollution problems caused by extensive fossil fuels consumption and the increasing serious energy crisis, much attention has been dedicated to alternative renewable energy and the photovoltaic devices, especially for solar power conversion [1]. On 1991, O’Regan and Grätzel [2] made breakthrough of lowering light harvesting cost compared to traditional silicon-based solar cells by coating a monolayer of charge transfer ruthenium on colloidal TiO\textsubscript{2}. In the recent decade, as a derivative of dye-sensitized solar cells (DSSCs) [3], quantum dot-sensitized solar cells (QDSSCs) [4] have attracted great attention. Compared with organic dyes, semiconductor quantum dots (QDs) exhibit several appealing advantages for application in solar cells: (1) easy tunable band gaps by adjusting the size and composition of QDs [5]; (2) high molar extinction coefficients [6]; (3) higher stability [7,8]; (4) large intrinsic dipole moments [9]; (5) multiple exciton generation effect [10] and (6) impact ionization effect. These points promote the theoretically predicted maximum thermodynamic efficiency up to 44\% based on quantum dot-sensitized solar cells, beyond the Shockley–Queisser limitation of 33\% for thin film solar cells. In the QDSSCs, the photoanode is crucial for collecting photo-generated...
Table 1. Review of literature reports on TiO$_2$/CdS/CdSe/ZnS or upconversion application in the solar cells.

| QD          | Upconversion application                      | Electrolyte | Counter electrodes | PCE (%) | Ref |
|-------------|-----------------------------------------------|-------------|--------------------|---------|-----|
| CdS/CdSe/ZnS | Mn$^{2+}$-NaYF$_4$:Yb$^{3+}$,Er$^{3+}$ Nanophosphors (QDSSC) | 2 M S$^{2-}$/Sn$^{2-}$ | CuS              | 4.75    | Ourwork |
| CdS/CdSe/ZnS | –                                           | 2 M S$^{2-}$/Sn$^{2-}$ | Pt | 2.56 | [23] |
| TEn/CdS/CdSe/ZnS | –                          | 2 M S$^{2-}$/Sn$^{2-}$ | 1 M NaOH | PbS | 4.68 | [26] |
| CdS(5)/CdSe(EP)/ZnS(5) | –                  | 2 M S$^{2-}$/Sn$^{2-}$ | 0.1 M NaOH | Cu$_2$S | 4.7 | [27] |
| CdS(3)/CdSe(4)/ZnS(4) | –                     | 1 M S$^{2-}$/Sn$^{2-}$ | Au | 4.22 | [4] |
| ZnS/CdS/CdSe | Ln-NaYF$_4$ (QDSSC) | 1 M S$^{2-}$/Sn$^{2-}$ | 0.2 M NaOH | PbS | 4.37 | [15] |
| CdS/CdSe/ZnS | Yb$^{3+}$-Er$^{3+}$ codoped ZrO$_2$ nanoparticles (QDSSC) | 1 M S$^{2-}$/Sn$^{2-}$ | 0.2 M NaOH | Cu$_2$S | 3.0 | [16] |

Nevertheless, the solar energy absorbed by QDSSCs is strongly dependent on the species of used QDs, and only represents in part of the total solar energy especially in the near-infrared range (NIR). Although some efforts have been dedicated to utilization of QDs whose absorption fall in the NIR such as PbS and PbSe [11], the full range absorption of sunlight in QDSSCs is still challenging. In addition, great efforts have been made to enhance the capability for the light harvesting of the near-infrared regions through other approaches such as introducing scattering layer or enhancing surface area [12,13].

As a matter of fact, while upconverting nanophosphors (UCNPs) which could convert sub-band gap photons (in near-infrared-NIR range) into above-band gap photons (in visible or longer range), have been intensively investigated for applications in bio-imaging [14] and optical thermometry [15], their application in solar cells are very limited. The UCNPs could not only undertake frequency upconversion avoiding the NIR transmission loss, but also the photons emitted in shorter wavelength range can be reabsorbed by QDs thus producing more electrons. Therefore, employing UCNPs in QDSSCs could be reasonably expected to effectively overcome the limitation of lower absorption efficiency of NIR and visible light [16–18].

Wang et al. incorporated Y$_2$O$_3$:Er$^{3+}$ into DSSCs and improved the efficiency of photoelectric conversion [19]. Li et al. used surface plasmon resonance (SPR) techniques to improve multi-shell-modified upconversion NaYF$_4$:Yb$^{3+}$, Er$^{3+}$@SiO$_2$@Au@TiO$_2$ crystallites for DSSCs and improved the photovoltaic conversion efficiency (PCE) by 28.1% [20].

Wang et al. increased the scattering ability of light by doping TiO$_2$ hollow spheres with dual-functional upconverters to improve the light absorption capacity [21]. Wu et al. introduced Y$_{6.78}$Yb$_{0.20}$Er$_{0.02}$F$_3$ into TiO$_2$ photoelectrode and improved the PCE by 35% [22]. However, these works are mainly focusing on the DSSC system and the species of UCNPs would strongly affect the performance of cells.

Therefore, further investigation of UCNPs enhanced PCE of QDSSCs is still necessary.

Herein we developed a compositive nanostructure of Mn$^{2+}$, Yb$^{3+}$ and Er$^{3+}$ tri-doped NaYF$_4$/TiO$_2$ (UCNPs/TiO$_2$) and expanded its application as an efficient photoanode for QDSSCs. Our results showed that present photoanode structure exhibited the best improvement in PCE compared with previous reports for UCNPs application in QDSSCs as shown in Table 1 which is lateral comparison between present work with similar previous reports for QDSSCs. Due to the very limited exploration of upconversion application in QDSSCs, some reports without addition of UC materials are also listed here for better understanding. The light-electric conversion properties of QDSSCs dependent on the different doping contents of UCNPs were systematically investigated. The maximum conversion efficiency of 4.75% with an improvement of 42.6% compared to the UCNPs-free TiO$_2$ photoanode (3.33%) was achieved at the optimized composition of 8 wt% UCNPs/TiO$_2$. The photoelectrode structure is shown in Scheme 1. The detailed preparation procedures of UCNPs, solar cell device as well as the photoanode were described in supplemental files.

Figure 1 shows morphologies of TiO$_2$ and UCNPs/TiO$_2$ photoelectrodes. The SEM top view of TiO$_2$ electrode in Figure 1(a) exhibits a typical porous structure due to sintering. The thickness of film could be confirmed.
Figure 1. Morphologies of TiO2/FTO and UCNPs/TiO2/FTO photoelectrodes: typical top view SEM images (a) and cross-sectional view (b) and TEM (c) of TiO2/FTO photoelectrode; typical top view SEM images (d) and cross-sectional view (e) and TEM (f) of UCNPs/TiO2/FTO photoelectrode; (g) and (h) TEM and HRTEM image of NaYF4:Yb3+, Er3+, Mn2+; (i) the HRTEM image of UCNPs/TiO2; (j) EDX mapping shows uniform distribution of O, Ti, Na, Y, Er, Yb and Mn.

from the cross-sectional view of SEM in Figure 1(b) to be ∼8 μm, and the film is composed of small P25 particles. The TEM image of P25 used in present work is shown in Figure 1(c), the average size of P25 is about 25 nm. For UCNPs/TiO2 photoelectrode (8 wt% UCNPs), the film is similar porous structure with that of TiO2 electrode while it is more uniform as shown in Figure 1(d), and the thickness is ∼8 μm which is the same with that of TiO2 electrode. The combination of UCNPs and TiO2 particles are observed from Figure 1(f). Due to the doping of UCNPs, the sintered UCNPs/TiO2 shows bigger size than pure TiO2 and UCNPs. Figure 1(g) shows the TEM image of highly uniform UCNPs with ∼20 nm in diameter, and inset is the well-indexed SAED patterns, which is consistent with the aforementioned XRD data. The HRTEM image of UCNPs particles and UCNPs/TiO2 combined particles are shown in Figure 1(h) and (i), respectively. For UCNPs particles, the interlayer spacing of d = 0.305 nm and d = 0.518 nm are consistent well with the (311) and (440) lattice planes of NaYF4:Yb/Er. It could also be observed that an interlayer spacing of d = 0.336 nm corresponds to the (101) lattice plane of NaYF4. For UCNPs/TiO2 combined particles, Figure 1(i) exhibits TiO2 nanoparticles with lattice spacing of d = 0.352 nm corresponding to (101) plane of hexagonal TiO2, and UCNPs particles with spacing of d = 0.305 nm and d = 0.518 nm corresponding to the (311) and (440) lattice planes of NaYF4:Yb/Er.

This confirms the existence of UCNPs particles in TiO2 electrode and elemental mapping further supports the result as shown in Figure 1(j). For a typical combined particle, the elemental mapping of 8 wt% UCNPs/TiO2/QD electrodes shows a uniform distribution of O, Ti, Na, Y, Er, Yb and Mn elements, indicating the successful preparation of UCNPs/TiO2 combined particles. Supplementary Figure 1 shows the XRD patterns of TiO2/QD and 8 wt% UCNPs/TiO2/QD electrodes deposited on FTO substrates.

The upconversion luminescence spectrum of NaYF4: Yb3+, Er3+, Mn2+ nanophosphors was measured under the excitation of a CW diode laser of 980 nm. As shown in Figure 2(a), the tri-doped NaYF4 nanophosphors exhibit a green emission along with a more intense red emission at around 660 nm. Due to the large absorption cross-section for the excitation wavelength at 980 nm, Yb3+ ion is usually used as a sensitizer in upconverting system. The ground-state ^2F_7/2 of Yb^{3+} could be excited to a higher energy level ^2F_{5/2} as shown in Figure 2(b), and then drops back to the ground state with energy transfer to the adjacent Er^{3+} simultaneously. As a result that the ground state Er^{3+}^4I_{15/2} would be excited to ^4I_{11/2} level. Then Er^{3+} might suffer from nonradiative decay to the lower energy level (^4I_{13/2}). Subsequently, the two states of Er^{3+}: ^4I_{11/2} and ^4I_{13/2} was excited to the higher energy levels of Er^{3+}: ^4F_{7/2} and ^4F_{9/2}, respectively. Then Er^{3+} in the ^4F_{7/2} level would relax nonradiatively to the ^2H_{11/2} and ^4S_{3/2} levels.
Figure 2. (a) Upconversion luminescence spectra of NaYF₄:Yb³⁺, Er³⁺, Mn²⁺, TiO₂, UC/TiO₂, all sample under 980 nm excitation; (b) energy level diagram of UCNPs/TiO₂ showing two energy transfer process in QDSSCs.

and might further relax to a lower level (4F₉/₂), followed by the upconversion transitions of ²H₁₁/₂ → ⁴I₅/₂ (520 nm), ⁴S₃/₂ → ⁴I₅/₂ (540 nm) and ⁴F₉/₂ → ⁴I₅/₂ (650 nm), respectively [23]. Mn²⁺ ions were codoped into the lattices to enhance the red emission of Er³⁺ by creating an alternative energy transfer channel for the excited electrons populated on ²H₁₁/₂ and ⁴F₇/₂ levels migrating back to ⁴F₉/₂ state via ⁴T₁ levels of Mn²⁺ [25].

Figure 3(a) shows the J–V curves of solar cells prepared with photoelectrodes including different UCNPs loading amount (0%, 2%, 4%, 6%, 8%, 10%) with photoactive area of 0.16 cm². The detailed photovoltaic parameters such as short-circuit current density (Jsc), open circuit voltage (Voc), fill factor (FF) and PCE (η) are summarized in Table 2.

Compared with undoped TiO₂ electrodes, the PCE of UCNPs/TiO₂ electrodes exhibits remarkable enhancement. The dramatically improved efficiency of 4.75% is achieved by 8 wt% UCNPs/TiO₂ electrode, which exhibits a 42.6% improvement in PCE compared with pure TiO₂ electrodes (3.33%). From Table 2 and Figure 3(a), it can be observed that the performance of cells is improved with the increasing doping amount of UCNPs till to 8 wt%, with Jsc = 21.30 mA m⁻², Voc = 0.54 V, FF = 0.41, and η = 4.75%. The improved PCE is due to the both increase in open circuit voltage and short-circuit current. The further increase of UCNPs loading amount to 10 wt% induces decrement in cell performance, probably because the excess addition of UCNPs would hinder the contact of QDs and TiO₂. Meanwhile, the porosity and defects may probably be formed which could capture or absorb electrons injected from QDs, thus limiting Jsc at higher doping concentration. If the UCNPs were incorporated in QDSSCs electrode structure, they would increase absorbance and slightly extend light absorption range resulting in red shift of the QD sensitizer absorption. This would be further confirmed by UV–VIS absorption spectrum. The increase in light absorption and the effective separation of charge are crucial to improve the cell photovoltaic performance.

IPCE is measured for further investigation of the effect of UCNPs on the performance and electron generation characteristics of QDSSCs. It is well known that quantum efficiency and short-circuit current are determined not only by the light absorption efficiency of the QD sensitizers, but also by the charge injection efficiency from the QDs to TiO₂. Figure 3(b) is the IPCE results. As expected, the doping of UC particles results in the overall enhancement in IPCE, which has the similar trend with J–V curves in Figure 3(a). The best IPCE is achieved by 8 wt% UCNPs/TiO₂ electrode. The absorption intensity of IPCE was improved from 550 nm to 600 nm obviously, and the absorption range of 8% UCNPs/TiO₂ is the largest, which is consistent with the Jsc parameters as shown in Table 2. Figure 3(c,d) shows the variation of short-circuit current and PCE versus doping amount of UC particles, which clearly indicates that the performance of cells is improved due to the UCNPs incorporation in TiO₂ photoanode.

Nyquist curve, as shown in Figure 4(a), consists of two semicircles: the first represents the recombination resistance at the counter-electrode/electrolyte interface (R₁), and the second indicates the charge transfer resistance at the photoanode/electrolyte interfaces (R₂). The series resistance (Rs) consisting of transport resistance of conductive glass substrate and external circuit resistance
Figure 3. Photocurrent–voltage curves and incident photon-to-current conversion efficiency (IPCE) of the QDSSCs based on different ratios UCNPs doped into photoelectrodes (a) and (b); (c) and (d) the distribution of short-circuit current and efficiency, respectively.

Table 2. Photovoltaic performance parameters of different ratios (with 0%, 2%, 4%, 6%, 8%, 10%) UCNPs doped into electrodes.

| UCNPs/TiO2 (wt%)/QD photoelectrode | V<sub>oc</sub> (V) | J<sub>sc</sub> (mA cm<sup>–2</sup>) | FF | η (%) |
|-----------------------------------|------------------|-------------------------------|----|------|
| 0%                                | 0.484            | 14.673                        | 0.469 | 3.33  |
| 2%                                | 0.468            | 15.805                        | 0.477 | 3.52  |
| 4%                                | 0.506            | 16.98                         | 0.424 | 3.64  |
| 6%                                | 0.491            | 20.037                        | 0.45  | 4.42  |
| 8%                                | 0.539            | 21.298                        | 0.414 | 4.75  |
| 10%                               | 0.527            | 19.705                        | 0.442 | 4.59  |

Moreover, UV–VIS absorption spectrum has been measured in order to clarify the underlying mechanism of improved J<sub>sc</sub> and the data were shown in Figure 4(c,d). From Figure 4(c), it could be observed that TiO<sub>2</sub> and UC/TiO<sub>2</sub> films show obvious elevated absorbance in the range of 300–400 nm without QD loading. With the increasing loading amount of UCNPs, the absorption intensity increases obviously. After loading with QDs as shown in Figure 4(d), the photoanode shows increased absorption capacity in the range of 300–650 nm, indicating expanded absorption range due to the addition of QDs. Absorption spectra further evidenced the enhanced light harvesting capability of the UCNPs incorporated CdS/CdSe/ZnS sensitized TiO<sub>2</sub> electrodes. The mechanism of the performance improvement of QDSSCs in this work is suggested as Scheme 1.

In summary, we developed a Mn<sup>2+</sup>, Yb<sup>3+</sup>, Er<sup>3+</sup> tri-doped NaYF<sub>4</sub> upconverting nanophosphors incorporated TiO<sub>2</sub> (UCNPs/TiO<sub>2</sub>) nanostructures as photoelectrode material in QDSSCs. This kind of UCNPs/TiO<sub>2</sub> nanostructures not only enhanced photocurrent due to increased electron injection from QD to TiO<sub>2</sub> using near-infrared light, but also improved the existing advantages.

can also be acquired from Nyquist curve. The 8 wt% UCNPs/TiO<sub>2</sub> electrode shows the smallest values of R<sub>1</sub>, R<sub>2</sub>, and R<sub>s</sub>. With the addition of UCNPs, the impedance is decreased, and the lowest values achieved by 8 wt% UCNPs/TiO<sub>2</sub> with R<sub>1</sub> (7.94 Ω), R<sub>2</sub> (48.42 Ω), and R<sub>s</sub> (5.12 Ω). Electron lifetime (τ<sub>r</sub>) can be estimated by the following formula: τ<sub>r</sub> = 1/2π · f<sub>max</sub>, through the Bode plots listed in the inset of Figure 4(b), where f<sub>max</sub> is the characteristic frequency at the peak of the low-frequency range. These results are consistent well with the J–V curve and IPCE measurement, indicating that the optimal efficiency is achieved by 8 wt% UCNPs/TiO<sub>2</sub>.
of TiO$_2$ through effectively suppressed interfacial recombination, facilitating the charge separation and prolonging the lifetime of excited electrons. There were 42.6% enhancement in the PCE and 45.1% improvement in the photocurrent.

**Disclosure statement**

No potential conflict of interest was reported by the authors.

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