Enhanced Power Conversion Efficiency of Perovskite Solar Cells with an Up-Conversion Material of \(\text{Er}^{3+}\)-\(\text{Yb}^{3+}\)-\(\text{Li}^{+}\) Tri-doped \(\text{TiO}_2\)

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Abstract

In this paper, \(\text{Er}^{3+}\)-\(\text{Yb}^{3+}\)-\(\text{Li}^{+}\) tri-doped \(\text{TiO}_2\) (UC-\(\text{TiO}_2\)) was prepared by an addition of \(\text{Li}^{+}\) to \(\text{Er}^{3+}\)-\(\text{Yb}^{3+}\) co-doped \(\text{TiO}_2\). The UC-\(\text{TiO}_2\) presented an enhanced up-conversion emission compared with \(\text{Er}^{3+}\)-\(\text{Yb}^{3+}\) co-doped \(\text{TiO}_2\). The UC-\(\text{TiO}_2\) was applied to the perovskite solar cells. The power conversion efficiency (PCE) of the solar cells without UC-\(\text{TiO}_2\) was 14.0%, while the PCE of the solar cells with UC-\(\text{TiO}_2\) was increased to 16.5%, which presented an increase of 19%. The results suggested that UC-\(\text{TiO}_2\) is an effective up-conversion material. And this study provided a route to expand the spectral absorption of perovskite solar cells from visible light to near-infrared using up-conversion materials.

Keywords: Enhanced power conversion efficiency, Perovskite solar cells, Up-conversion material

Background

Organolead halide perovskite solar cells (PSCs) have become attractive in the solar cell field, which is due to their advantages, such as high efficiency, lost cost, and simple fabrication [1–4]. In a few years, the power conversion efficiency (PCE) of PSCs has been improved to 22.1% [5]. However, perovskite solar cells only absorb a small fraction of incident light in UV and visible ranges due to the narrow energy band gap of perovskite sensitizer; thus, a large portion of incident light is lost due to its non-absorption of near-infrared (NIR) [6, 7].

One promising method to solve the NIR energy loss issue is to apply up-conversion materials to PSCs, which can convert NIR to visible light. Some authors have reported the applications of up-conversion materials to perovskite solar cells [8–10], in which the up-conversion materials adopted were mainly based on beta-phase sodium yttrium fluoride (\(\beta\)-\(\text{NaYF}_4\)). While the \(\beta\)-\(\text{NaYF}_4\) up-conversion materials can reduce charge transport ability of electron transfer layer [11]. It has been reported that \(\text{Er}^{3+}\)-\(\text{Yb}^{3+}\)-\(\text{F}^{-}\) tri-doped \(\text{TiO}_2\) can improve the PCE of dye-sensitized solar cells (DSSCs) due to its enhanced up-conversion emission compared with \(\text{Er}^{3+}\)-\(\text{Yb}^{3+}\) co-doped \(\text{TiO}_2\) [12]. In our previous publication [13], we reported the application of \(\text{Er}^{3+}\)-\(\text{Yb}^{3+}\) co-doped \(\text{TiO}_2\) nanorods to PSCs. Some researchers have proved that the addition of \(\text{Li}^{+}\) into \(\text{Er}^{3+}\)-\(\text{Yb}^{3+}\) co-doped \(\text{TiO}_2\) could increase the up-conversion emission [14, 15]. And it has been reported that the perovskite solar cells based on Li-doped \(\text{TiO}_2\) produce higher performances compared to the device based on un-doped \(\text{TiO}_2\) [16]. Thus, we wonder if the up-conversion materials of \(\text{Er}^{3+}\)-\(\text{Yb}^{3+}\)-\(\text{Li}^{+}\) tri-doped \(\text{TiO}_2\) can be applied to PSCs to further improve the performance.

Therefore, in the present study, we prepared \(\text{Er}^{3+}\)-\(\text{Yb}^{3+}\)-\(\text{Li}^{+}\) tri-doped \(\text{TiO}_2\) (UC-\(\text{TiO}_2\)) by addition of \(\text{Li}^{+}\) into \(\text{Er}^{3+}\)-\(\text{Yb}^{3+}\) co-doped \(\text{TiO}_2\), which presented an enhanced up-conversion emission compared with \(\text{Er}^{3+}\)-\(\text{Yb}^{3+}\) co-doped \(\text{TiO}_2\). The UC-\(\text{TiO}_2\) was applied to perovskite solar cells. The PCE of the solar cells with UC-\(\text{TiO}_2\) is...
increased to 16.5 from 14.0% for the solar cells without UC-TiO₂, which presents an increase of 19%.

Methods

Synthesis of Er³⁺-Yb³⁺-Li⁺ Tri-Doped TiO₂

The nanocrystals of Er³⁺-Yb³⁺-Li⁺ tri-doped TiO₂ (UC-TiO₂) were synthesized by a modified method [15]. A titanium (IV) n-butoxide was prepared by mixing n-butyl titanate (Ti(OBu)₄) with acetylacetone (AcAc) at room temperature for 1 h under agitation. Then, the iso-propyl (i-PrOH) was put in the titanium (IV) n-butoxide. Next, i-PrOH, deionized water, and concentrated nitric acid (HNO₃) was mixed and dropped into the solution. A light yellow TiO₂ sol was obtained after stirring for 6 h. The molar ratios of AcAc, H₂O, and HNO₃ to Ti(OBu)₄ were 1:1, 2:1, and 0.3:1, respectively. Then, Er(NO₃)₃·5H₂O, Yb(NO₃)₃·5H₂O, and LiNO₃ were added into the TiO₂ sol to make the molar ratios of Er:Yb:Li:Ti = 0.5:10:x:100 (x = 0, 10, 15, 20, and 25). The solvent in the Er³⁺-Yb³⁺-Li⁺ tri-doped TiO₂ sol (UC-TiO₂ sol) was removed by drying for 8 h at 100 °C. Then, the UC-TiO₂ was calcined at 500 °C for 30 min.

Fabrication of Perovskite Solar Cells

Patterned FTO glass substrate was cleaned in acetone, 2-propanol, and ethanol by sonication for 20 min, respectively. Then, UV-O₃ was used to treat the FTO for 15 min. A compact layer was formed by spin-coating a precursor solution on FTO and annealed at 500 °C for 30 min. The precursor solution is 0.1 M titanium diisopropoxide bis (acetylacetone) (75 wt% in isopropanol, Aldrich) solution in 1-butanol. A mesoporous TiO₂ film was obtained by spin-coating TiO₂ solution on the compact layer at 4000 rpm for 30 s, followed by annealing at 100 °C for 10 min and 500 °C for 30 min, respectively. The TiO₂ solution was prepared by diluting TiO₂ paste (30NR-D, Dyesol) with ethanol (1:6, weight ratio) or by mixing the UC-TiO₂ sol and the diluted TiO₂ solution (UC-TiO₂: TiO₂ = x:100, v/v, x = 10, 20, 30, and 40). A perovskite layer was formed on UC-TiO₂ layer by spin-coating perovskite precursor solution in two steps at 1000 rpm for 10 s and 4000 rpm for 30 s, and 200 µL chlorobenzene was poured on the substrate during the second step before the end of 20 s. Then, the sample was heated on the hotplate at 100 °C for 1 h. The perovskite precursor solution was obtained by mixing PbI₂ (1.1 M), FAI (1 M), PbBr₂ (0.22 M), and MABr (0.2 M) in the mixed solvent of anhydrous DMF/DMSO (4:1 v:v). Then, a 1.5 M stock solution of CsI pre-dissolved in DMSO was dropped into the
mixed perovskite precursor solution [17]. A hole-transfer layer was formed by spin-coating a solution of spiro-MeOTAD at 4000 rpm 30 s. Finally, an 80-nm-thick gold layer was thermally evaporated on the top of the device.

**Characterization**

Up-conversion fluorescence, steady-state photoluminescence (PL), and time-resolved photoluminescence (TRPL) spectra were measured with a FLS 980 E fluorometer (Edinburgh Photonics). X-ray diffraction (XRD) spectra were acquired on a diffractometer (DX-2700). X-ray photoelectron spectroscopy (XPS THS-103) with Al Ka as X-ray source was applied to measure the chemical state of the samples. Ultraviolet visible near-infrared (UV-vis-NIR) absorption spectra were collected on a Varian Cary 5000 spectrophotometer. Microstructure and morphologies were observed with a scanning electron microscope (SEM; JEM-7001F, JEOL). Photocurrent-voltage (J-V) curves were measured with a Keithley 2440 Sourcemeter under AM 1.5 G illumination. The electrochemical impedance spectroscopy

![Fig. 3 XPS spectra of UC-TiO2. a Ti 2p, b Er 4d, c Yb 4d, and d Li 1s](image)

![Fig. 4 a UV-vis-NIR absorption spectra of commercial TiO2 (30NR-D) and UC-TiO2. b Tauc plots](image)
(EIS) was obtained with an electrochemical workstation (CHI660e, Shanghai CHI Co., Ltd.).

Results and Discussion

Up-conversion emissions were measured with an excitation of a 980-nm laser. Figure 1a shows the up-conversion emissions spectra of Er\(^{3+}\)-Yb\(^{3+}\)-Li\(^{+}\) tri-doped TiO\(_2\) (Er:Yb:Li:Ti = 0.5:10:x:100, x = 0, 10, 15, 20, and 25, molar ratio). Figure 1b shows the schematic energy-transfer mechanisms of the Er\(^{3+}\) ions. The green emission bands located at about 525 and 545 nm can be attributed to \(2H_{11/2} \rightarrow 4I_{15/2}\) and \(4S_{3/2} \rightarrow 4I_{15/2}\) transitions of Er\(^{3+}\) ions, respectively. And the red emission bands centered at about 658 nm could correspond to \(4F_{9/2} \rightarrow 4I_{15/2}\) transitions of Er\(^{3+}\) ions [15, 16]. With the increase of Li\(^+\) doping content, the intensity of the spectrum increases firstly, and then decreases, which is the maximum when the doping content of Li\(^+\) is \(x = 20\). Hereinafter, the up-conversion material of Er\(^{3+}\)-Yb\(^{3+}\)-Li\(^+\) tri-doped TiO\(_2\) (Er:Yb:Li:Ti = 0.5:10:20:100, molar ratio) was applied.

Figure 2 displays the XRD patterns of commercial TiO\(_2\) (30NR-D) and UC-TiO\(_2\). The XRD pattern of UC-TiO\(_2\) is similar to that of 30NR-D. The peaks at 25.3°, 37.8°, 48.0°, and 53.8° in the XRD patterns are assigned to the (101), (004), (200), and (105) planes (JCPDS card no.21-1272), respectively, which indicates that UC-TiO\(_2\) and 30NR-D belong to the anatase phase of TiO\(_2\).

To confirm the doping of Er, Yb, and Li into TiO\(_2\), XPS spectra of UC-TiO\(_2\) were recorded and shown in Fig. 3. The peaks at 458.1 and 463.9 eV in Fig. 3a could belong to Ti 2p\(_{3/2}\) and Ti 2p\(_{1/2}\), respectively, and the peaks at 168.8 eV in Fig. 3b and at 192.7 eV in Fig. 3c could be attributed to Er 4d and Yb 4d, respectively [18]. The peak at 55.5 eV in Fig. 3d can correspond to Li 1s [19]. The survey XPS spectrum of UC-TiO\(_2\) and O1s peak were also presented in Additional file 1: Figure S1. The results demonstrated that Er, Yb, and Li atoms were doped into TiO\(_2\).

Figure 4a displays the UV-vis-NIR absorption spectra of TiO\(_2\) (30NR-D) and UC-TiO\(_2\). Compared with 30NR-D, UC-TiO\(_2\) presents a stronger absorption, especially at the range from 900 to 1000 nm. The energy band gap...
\( E_g \) could be estimated with the Tauc plot [20]. The Tauc plots are shown in Fig. 4b, from which the values of \( E_g \) could be obtained to be 3.20 and 3.10 eV for 30NR-D and UC-TiO\(_2\), respectively. The \( E_g \) of UC-TiO\(_2\) is smaller than that of un-doped TiO\(_2\).

Figure 5a shows the SEM image of 30NR-D film formed on the compact layer. The nanoparticle size is about 30 nm, and the size distribution is uniform. Figure 5b shows the SEM image of the film containing 30NR-D and UC-TiO\(_2\) deposited on the compact layer by spin-coating method. There is no obvious difference between the two films, which displays that the particle size and morphology of UC-TiO\(_2\) are similar to those of 30NR-D.

In the present work, the perovskite film was formed with the method previously reported [17]. According to report, the composition of the perovskite film is \( \text{Cs}_5(\text{MA}_{0.17}\text{FA}_{0.83})_9\text{Pb}(\text{I}_{0.83}\text{Br}_{0.17})_3 \), and the role of the CsI is to make the perovskite solar cells thermally more stable, with less phase impurities, and less sensitive to processing conditions [17]. The scheme of the device is presented in Additional file 1: Figure S2.

The perovskite solar cells based on mesoporous layer formed with the mixture of UC-TiO\(_2\) sol and diluted TiO\(_2\) solution (UC-TiO\(_2\)/TiO\(_2\) = \( x:100, v/v \), \( x = 0, 10, 20, 30, \) and 40) were fabricated and their I-V curves were measured. The photovoltaic parameters were obtained from the I-V measurements. Figure 6a shows the PCE dependence of solar cells on the content of UC-TiO\(_2\) (UC-TiO\(_2\)/TiO\(_2\) = \( x:100, v/v \)) in the mixture. With the increase of UC-TiO\(_2\) content, the power conversion efficiency (PCE) of solar cells increases firstly, and then decreases, which is the maximum at the content of \( x = 20 \) for UC-TiO\(_2\). The detailed photovoltaic parameters of solar cells with 20% UC-TiO\(_2\) and without UC-TiO\(_2\) were listed in Table 1. Compared with those of the devices without UC-TiO\(_2\), the photovoltaic parameters of the solar cells with UC-TiO\(_2\) present an improvement. The PCE of the solar cells with 20% UC-TiO\(_2\) is increased to 16.5 from 14.0% for the solar cells without UC-TiO\(_2\), which presents an increase of 19%.

To understand the enhancement, some investigations were carried out. Steady-state photoluminescence (PL) and time-resolved photoluminescence (TRPL) can be applied to investigate the electron extration and transport process. The PL of perovskite layer on the mesoporous layers formed by 30NR-D and 30NR-D with UC-TiO\(_2\) were measured and shown in Fig. 7a. Compared with that of 30NR-D/perovskite, the PL intensity of 30NR-D with UC-TiO\(_2\)/perovskite becomes reduced, which indicates that the electron extration and transport efficiency across the interface between 30NR-D with UC-TiO\(_2\) and perovskite is better than that between 30NR-D and perovskite [21]. Figure 7b shows the TRPL spectra of perovskite layer on the mesoporous layers formed by 30NR-D and 30NR-D with UC-TiO\(_2\). The TRPL spectrum was fitted to a biexponential function, in which the fast decay \( (\tau_1) \) may be resulted from transportation of free carriers, and the slow decay \( (\tau_2) \) can be originated from radiative recombination of free carriers [22–24]. The obtained parameters are listed in Table 2. Compared with that of 30NR-D/perovskite, the fast decay time (2.8 ns) of 30NR-D with UC-TiO\(_2\)/perovskite becomes

**Table 1** Photovoltaic parameters of the solar cells with UC-TiO\(_2\) (20%) and without UC-TiO\(_2\)

| Samples          | \( V_{oc} \) (V) | \( I_{sc} \) (mA/cm\(^2\)) | FF (%) | PCE (%) |
|------------------|------------------|----------------------------|--------|---------|
| Control          | 1.01 ± 0.01      | 21.0 ± 0.2                 | 66.0 ± 0.3 | 14.0 ± 0.3 |
| With UC-TiO\(_2\) | 1.05 ± 0.01      | 22.2 ± 0.3                 | 70.8 ± 0.02 | 16.5 ± 0.2 |

![Fig. 7](image-url) a PL and b TRPL of perovskite layers on the mesoporous layers formed by 30NR-D without UC-TiO\(_2\) and 30NR-D with UC-TiO\(_2\)
smaller, while the fraction of fast decay process (98.2\%) becomes larger. This implies that the charge transfer between perovskite and 30NR-D with UC-TiO₂ is faster than that between perovskite and 30NR-D.

Eletrochemical impedance spectroscopy (EIS) is an effective method to get some information on carrier transfer behavior. Figure 8a displays the Nyquist plots of the devices based on mesoporous layers formed by 30NR-D and 30NR-D with UC-TiO₂ in which two arcs were observed. The arc at high-frequency could be resulted from the contact resistance between the interfaces, and the arc at low-frequency could come from the recombination resistance ($R_{\text{rec}}$) and chemical capacitance ($C_\mu$) of the device \[25, 26\]. The EIS was fitted with an equivalent circuit shown in Fig. 8b, and the obtained parameters are listed in Table 3. The series resistance of the devices based on 30NR-D with UC-TiO₂ becomes smaller than that of the devices on based on 30NR-D, while the recombination resistance of the former becomes larger than that of the later. This indicates the charge recombination was decreased and the charge transport was improved for the device based on 30NR-D with UC-TiO₂.

To further prove the effect of UC-TiO₂ on photocurrents of the devices, the I-V curves of the devices based on the mesoporous layers without UC-TiO₂ and with UC-TiO₂ were measured under the simulated solar radiation in the wavelength range of $\lambda \geq 980$ nm with a NIR filter, which are shown in Additional file 1: Figure S3. Compared with that of the device without UC-TiO₂, the photocurrent of the device with UC-TiO₂ was obviously enhanced, which demonstrates that the incorporation of UC-TiO₂ in the device can transform the NIR light into visible light, which can be absorbed by the devices to generate photocurrent.

To explain the increased open circuit voltage ($V_{\text{oc}}$) of the solar cells, the energy band arrangements of UC-TiO₂, TiO₂, perovskite, and Spiro-OMeTAD were shown in Fig. 9 based on the absorption spectra (Fig. 4) and the literatures \[27, 28\]. The conduction band edge of the UC-TiO₂ is lower than that of TiO₂ (30NR-D) due to its smaller energy band gap; thus, the conduction band offset between UC-TiO₂ and perovskite is larger than that between TiO₂ and perovskite. This could be one of the reasons to have a higher open circuit voltage for UC-TiO₂ based solar cells \[29, 30\].

In summary, the PCE increase of the solar cells based on the mesoporous layer with UC-TiO₂ is due to the enlarged $I_{\text{sc}}$ and increased $V_{\text{oc}}$. The enlarged $I_{\text{sc}}$ could be due to expansion of spectral absorption to near-infrared (NIR) range with up-conversion material, reduced recombination, and fast charge transfer of photo-generated carriers. The increased $V_{\text{oc}}$ may be attributed to the enlarged conduction band offset between UC-TiO₂ and perovskite.

### Conclusions

$\text{Er}^{3+}$-$\text{Yb}^{3+}$-$\text{Li}^+$ tri-doped TiO₂ (UC-TiO₂) was prepared by addition of Li⁺ into Er³⁺-Yb³⁺ co-doped TiO₂, which presented an enhanced up-conversion emission. The UC-TiO₂ was applied to the perovskite solar cells. The performance of solar cells with UC-TiO₂ was improved compared with that of control device. The $I_{\text{sc}}$, $V_{\text{oc}}$, and FF of solar cells with UC-TiO₂ were increased to 22.2 mA/cm², 1.05 V, and 70.8% from 21.0 mA/cm², 1.01 V,
and 66.0% for the control devices, respectively. Thus, the PCE with UC-TiO$_2$ was increased to 16.5 from 14.0% for the solar cells without UC-TiO$_2$, which presents an increase of 19%. Based on some experimental results, this PCE increase was explained.

**Additional file**

Additional file 1: Figure S1. a Survey XPS spectrum of UC-TiO$_2$. b O 1s peak. Figure S2 Scheme of the perovskite solar cells. Figure S3 I-V curves of the devices based on the mesoporous layers without UC-TiO$_2$ and with UC-TiO$_2$ measured under the simulated solar radiation in the wavelength range of λ ≥ 980 nm with a NIR filter. (DOCX 81 kb)

**Abbreviations**

EIS: Electrochemical impedance spectroscopy; NIR: Near-infrared; PCE: Power conversion efficiency; PL: Photoluminescence; PSCs: Perovskite solar cells; TRPL: Time-resolved photoluminescence

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**Availability of Data and Materials**

All data are fully available without restriction.

**Authors’ Contributions**

ZZ and Y-L Mao carried out the main part of the experiment and drafted the manuscript. The other authors provided assistance with experimental measurements, data analysis and the manuscript writing. All authors read and approved the final manuscript.

**Competing Interests**

The authors declare that they have no competing interests.

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