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Enhancement of Perovskite Solar Cells Efficiency using N-Doped TiO₂ Nanorod Arrays as Electron Transfer Layer

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Abstract

In this paper, N-doped TiO₂ (N-TiO₂) nanorod arrays were synthesized with hydrothermal method, and perovskite solar cells were fabricated using them as electron transfer layer. The solar cell performance was optimized by changing the N doping contents. The power conversion efficiency of solar cells based on N-TiO₂ with the N doping content of 1% (N/Ti, atomic ratio) has been achieved 11.1%, which was 14.7% higher than that of solar cells based on un-doped TiO₂. To get an insight into the improvement, some investigations were performed. The structure was examined with X-ray powder diffraction (XRD), and morphology was examined by scanning electron microscopy (SEM). Energy dispersive spectrometer (EDS) and Tauc plot spectra indicated the incorporation of N in TiO₂ nanorods. Absorption spectra showed higher absorption of visible light for N-TiO₂ than un-doped TiO₂. The N doping reduced the energy band gap from 3.03 to 2.74 eV. The photoluminescence (PL) and time-resolved photoluminescence (TRPL) spectra displayed the faster electron transfer from perovskite layer to N-TiO₂ than to un-doped TiO₂. Electrochemical impedance spectroscopy (EIS) showed the smaller resistance of device based on N-TiO₂ than that on un-doped TiO₂.

Keywords: Enhancement of efficiency, N-doped TiO₂ nanorod arrays, Electron transfer layer

Background

In recent years, extensive studies are focused on perovskite solar cells (PSCs) due to their outstanding properties, such as large absorption coefficient, electron-hole diffusion length, and high charge carrier mobility [1–5]. The power conversion efficiency (PCE) of perovskite solar cells has been over 22% [6]. Conventionally, perovskite solar cells consist of a perovskite layer sandwiched between an electron transfer material (ETM) layer and a hole transfer material (HTM) layer. Mesoporous TiO₂ has been used as the ETM in most of the perovskite solar cells [7, 8]. Compared with the mesoporous structure, one dimensional (1D) nanostructure has some advantages, such as easy pore filling of active layer or HTM, better electron transfer, and lower charge recombination [9, 10]. Therefore, TiO₂ nanorods (NRs) have

been widely applied to perovskite solar cells [11, 12]. However, there are a mass of oxygen vacancies defects exist in pristine TiO₂ nanorods, which reduces the efficiency and stability of the perovskite solar cell [13].

In order to solve the issues, some methods have been adopted, such as metal doping [14, 15] and nonmetal doping [16]. It has been reported that N-doped TiO₂ as a photoanode of dye-sensitized solar cells (DSSCs) can improve the energy conversion efficiency due to the change of properties of TiO₂, such as electron lifetime prolongation, charge transfer resistance reduction, and visible light absorption extension [17, 18].

We wondered about the effect of N-doped TiO₂ on the performance of perovskite solar cells. Hence, in the present study, we synthesized N-doped TiO₂ (N-TiO₂) nanorod arrays with hydrothermal method and fabricated perovskite solar cells using them as electron transfer layer. The solar cell performance was optimized by changing the N doping contents. The PCE of solar cells based on N-TiO₂ with the N

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doping content of 1% (N/Ti, atomic ratio) has been achieved 11.1%, which was 14.7% higher than that of solar cells based on un-doped TiO_2 . The possible mechanisms of enhancement were discussed based on some investigations.

Methods

Growth of TiO_2 Nanorod Arrays

Patterned fluorine-doped tin oxide (FTO)-coated glass substrate was cleaned by sonication for 20 min in detergent, acetone, 2-propanol, and ethanol, respectively. A TiO_2 compact layer was deposited by dipping the substrate in a 0.2 M TiCl_4 aqueous solution at 70 °C for 30 min. TiO_2 NRs were grown on the treated FTO substrate by a hydrothermal method in our previous report [19]. A 0.7 mL of titanium(IV) n-butoxide was added to a mixture of hydrochloric acid and deionized water. Subsequently, the pre-calculated amount of $\text{CO}(\text{NH}_2)_2$ was added to the solution (the nominal N/Ti atomic ratio, 0.5, 1, 2, and 3%) and stirred until it was completely dissolved. The FTO substrate was put into the solution and sealed in an autoclave. The autoclave was heated to 170 °C for several hours. The obtained TiO_2 nanorods film was rinsed and annealed at 500 °C for 60 min.

Materials Preparation

Methylammonium iodide ($\text{CH}_3\text{NH}_3\text{I}$) was synthesized with a reported method [20]. The precursor solution for perovskite film formation was obtained by mixing PbCl_2 and $\text{CH}_3\text{NH}_3\text{I}$ in anhydrous *N,N*-dimethylformamide (DMF) at a 1:3 molar ratio at 60 °C overnight.

Solar Cell Fabrication

The perovskite film was formed by spin coating at 2000 rpm for 60 s in a glove box, and drying on a hot-plate at 110 °C for 60 min. The HTM layer was obtained by spin coating a spiro-OMeTAD solution at 2000 rpm

for 60 s. Finally, a gold layer was deposited on the top of the device by thermal evaporation.

Characterization

X-ray diffraction (XRD) patterns were measured using a diffractometer (DX-2700). Photocurrent–voltage (I – V) curves were carried out with a Keithley 2440 Source meter under AM 1.5 G illumination from a Newport Oriol Solar Simulator with an intensity of 100 mW/cm^2 . A shadow mask was used to determine the active area of 0.1 cm^2 . Morphologies and microstructures were performed using a scanning electron microscope (SEM, JEM-7001F, JEOL) equipped with an energy dispersive spectrometer (EDS). Absorption spectra were obtained with a UV–Vis spectrophotometer (Varian Cary 5000). Steady-state photoluminescence (PL) and time-resolved photoluminescence (TRPL) spectra were collected with a fluorometer (FLS 980E, Edinburgh Photonics). An electrochemical workstation (CHI660e, Shanghai CHI Co., Ltd.) was used to collect the electrochemical impedance spectroscopy (EIS) with a bias of 0.6 V.

Results and Discussion

TiO_2 nanorod arrays with different N doping contents were prepared, and perovskite solar cells based on them were fabricated. The I – V measurements were performed by reverse scan (RS) and forward scan (FS). The photovoltaic parameters were obtained by the average of reverse scan and forward scan for each device. The I_{sc} , V_{oc} , FF, and PCE of the solar cells in the study were obtained by an average of the data from 20 pieces of devices. Figure 1a shows the power conversion efficiency of solar cells dependence on nominal N doping contents. The PCE changes with the increase of N doping content, which reaches the maximum at the doping content of 1% (N/Ti, atomic ratio). Table 1 shows the photovoltaic parameters of the solar cells based on un-doped and 1% N- TiO_2 NRs. The I_{sc} , V_{oc} , FF, and PCE of the solar cells

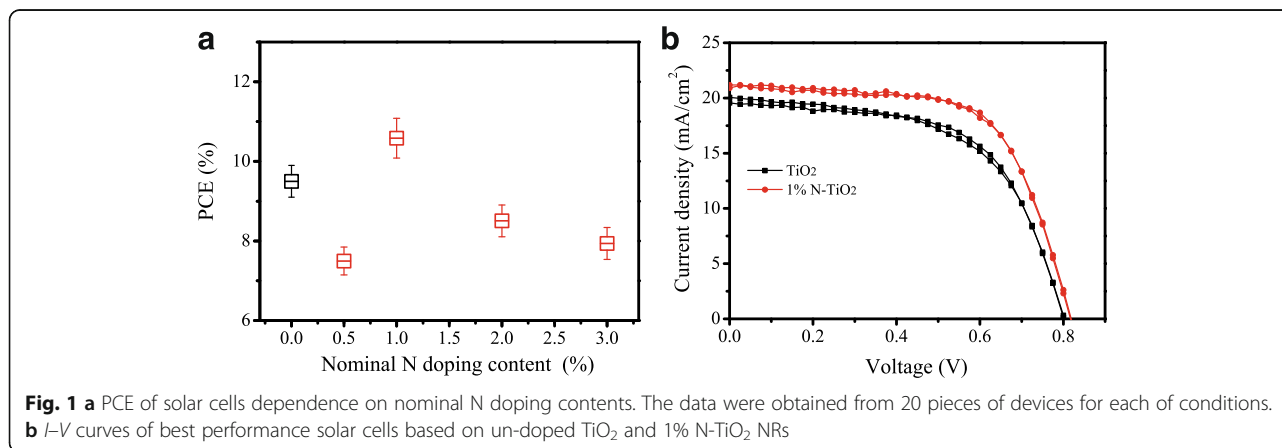


Fig. 1 a PCE of solar cells dependence on nominal N doping contents. The data were obtained from 20 pieces of devices for each of conditions. b I – V curves of best performance solar cells based on un-doped TiO_2 and 1% N- TiO_2 NRs

Table 1 Photovoltaic parameters of best performance solar cells based on the TiO₂ and N-TiO₂ NRs

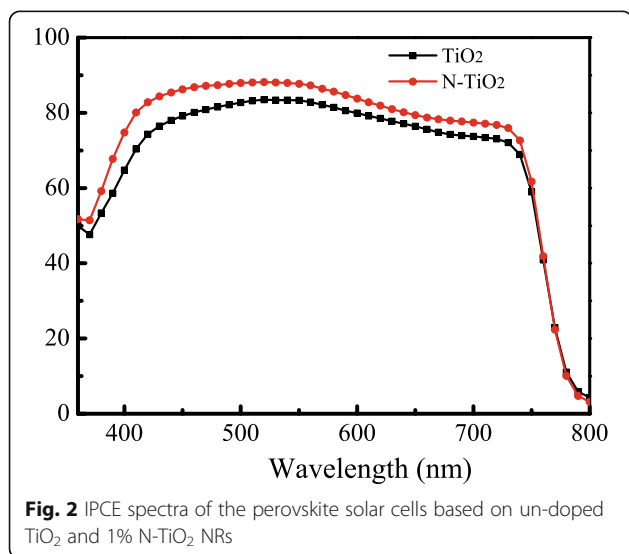
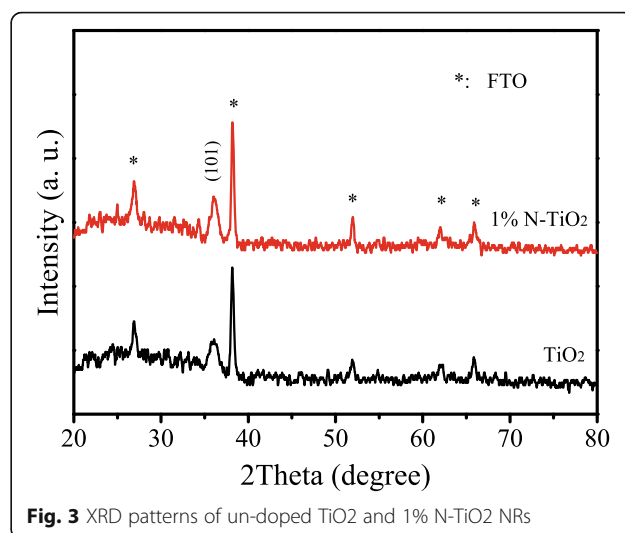
Sample	V _{oc} (V)	I _{sc} (mA/cm ²)	FF	PCE (%)
TiO ₂	0.80 ± 0.02	19.2 ± 0.06	0.62 ± 0.03	9.5 ± 0.3
1% N-TiO ₂	0.82 ± 0.01	20.5 ± 0.7	0.65 ± 0.02	10.9 ± 0.2

based on N-TiO₂ NRs are enhanced compared with those based on un-doped TiO₂ NRs. The PCE of solar cells based on N-TiO₂ NRs is 14.7% higher than that of solar cells based on un-doped TiO₂ NRs. Figure 1b shows the *I*-*V* curves of best performance solar cells based on un-doped TiO₂ and 1% N-TiO₂ NRs.

Figure 2 shows the IPCE of the perovskite solar cells based on un-doped TiO₂ NRs and 1% N-TiO₂ NRs. The IPCE at the entire wavelength range of the cells based on N-TiO₂ NRs are larger than those on the un-doped TiO₂ NRs, which agree with the *I*-*V* measurements.

To get an insight into the enhancement, some investigations were performed. Figure 3 shows the XRD patterns of un-doped TiO₂ and 1% N-TiO₂ NRs. The peaks labeled with stars were assigned to SnO₂ (JCPDS card, 41-1445) on FTO substrate. The peak at 36.1° was assigned to the (101) planes of rutile TiO₂ (JCPDS card, 21-1276) [21]. For the XRD patterns of N-TiO₂ NRs, the peaks on N element were not observed. This could be due to the homogeneous distribution of N with Ti in the samples [22] and small amount of doping contents.

Figure 4 shows the XPS spectra of 1% N-TiO₂ NRs. Figure 4a shows the survey XPS spectrum. The peaks at 458.8 and 464.8 eV in Fig. 4b are attributed to the binding energy of Ti 2p_{3/2} and Ti 2p_{1/2}, respectively. The peak located at 400.1 eV in Fig. 4c is attributed to N 1s [23]. The peak at 530.1 eV in Fig. 4d could be from O 1s. The XPS spectra demonstrated the successful doping of N in the

**Fig. 2** IPCE spectra of the perovskite solar cells based on un-doped TiO₂ and 1% N-TiO₂ NRs**Fig. 3** XRD patterns of un-doped TiO₂ and 1% N-TiO₂ NRs

TiO₂ film, which was further confirmed by the EDS spectrum as shown in Additional file 1: Figure S1 (Supplementary Material).

Figure 5a, c shows the plane-view and cross-sectional SEM images of un-doped TiO₂ NRs, and Fig. 5b, d shows the plane-view and cross-sectional SEM images of 1% N-TiO₂ NRs. The diameter and length of the un-doped TiO₂ NRs were determined to be 48 ± 5 nm and 490 ± 15 nm, respectively. The diameter and length of the 1% N-TiO₂ NRs were determined to be 42 ± 6 nm and 480 ± 25 nm, respectively. The diameter of N-TiO₂ NRs is slightly decreased compared with that of un-doped TiO₂ NRs. The length distribution of N-TiO₂ NRs is more ununiform than that of un-doped TiO₂ NRs. From a view of large area (Additional file 1: Figure S2, Supplementary Material), there are some bluges on the sample surfaces due to the inhomogenous oritiation. There are more bluges on the surfaces of N-TiO₂ NRs compared with those of un-doped TiO₂ NRs. This could be attributed to the effect of N doping. Additional file 1: Figure S3 (Supplementary Material) shows the cross-sectional SEM images of the whole solar cells based on TiO₂ NRs. The bottom is the FTO glass. Pores of nanorods are filled by MAPbI_{3-x}Cl_x, on which a capping layer of perovskite was formed. The Spiro-OMeTAD layer is separated by the capping layer from nanorod films. The top layer is a thin Au film.

Figure 6a shows the UV-Vis absorption spectra of un-doped TiO₂ and 1% N-TiO₂ NRs. The absorption intensity of N-TiO₂ is stronger than that of un-doped TiO₂. The energy band gap (E_g) can be calculated based on the absorption spectra using the Tauc equation [24]. The Tauc curve is shown in Fig. 6b, in which E_g can be estimated to be 3.03 and 2.74 eV for un-doped TiO₂ and N-TiO₂ NRs, respectively. Compared with that of un-doped TiO₂, the energy band gap of N-TiO₂ becomes smaller.

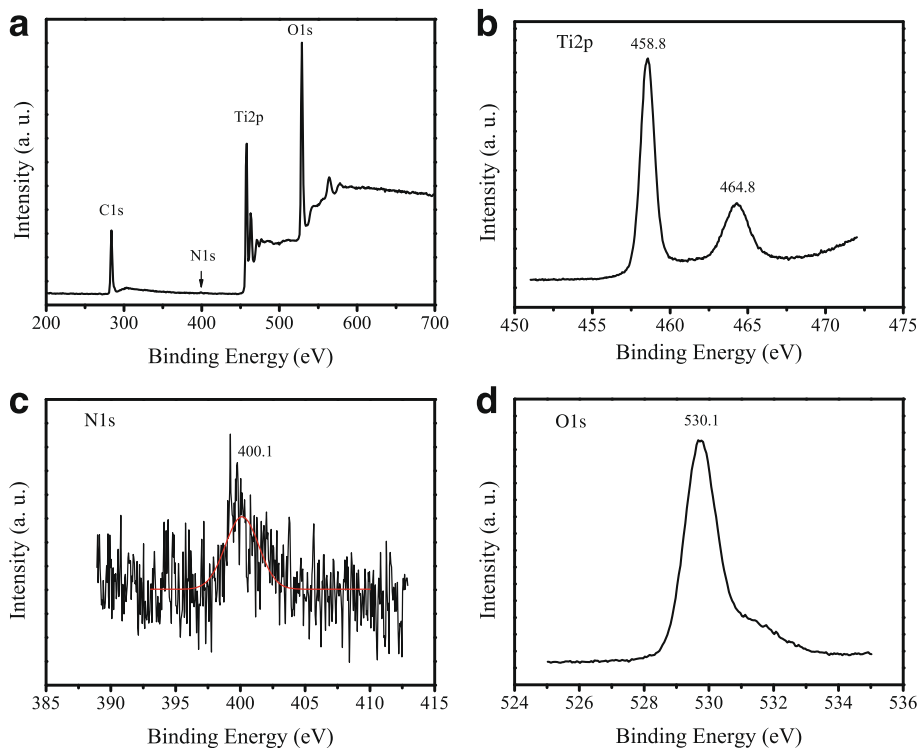


Fig. 4 XPS spectra of 1% N-TiO₂ NRs. **a** Survey XPS, **b** Ti 2p, **c** N 1s, and **d** O 1s

This could be attributed to the substitution location of N in the TiO₂ lattice, in which an O (Ti) atom is replaced by an N atom [25]. The conduction band offset between N-TiO₂ and MAPbI_{3-x}Cl_x is larger than that between un-doped TiO₂ and MAPbI_{3-x}Cl_x due to its narrow energy

band gap, which might be one of the reasons to present a higher voltage for N-TiO₂-based solar cells [26].

PL is a suitable tool to study the efficiency of charge carrier trapping, migration, and transfer [27, 28]. Figure 7a shows the PL spectra of un-doped TiO₂/

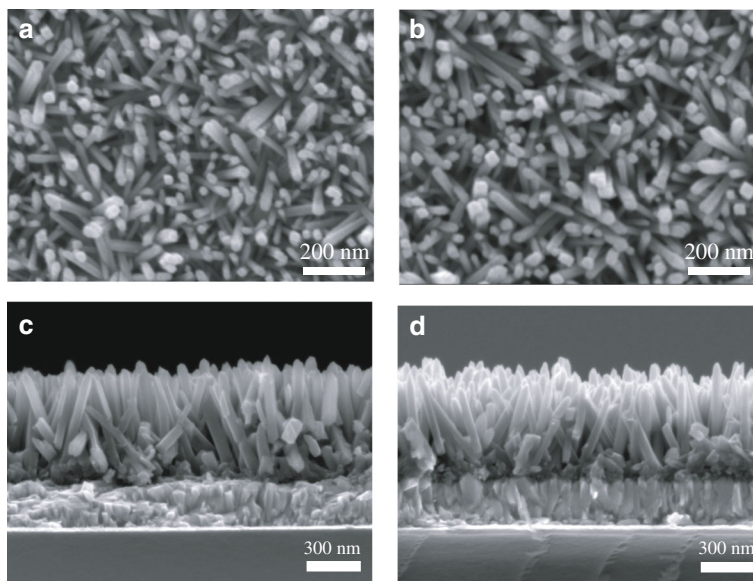
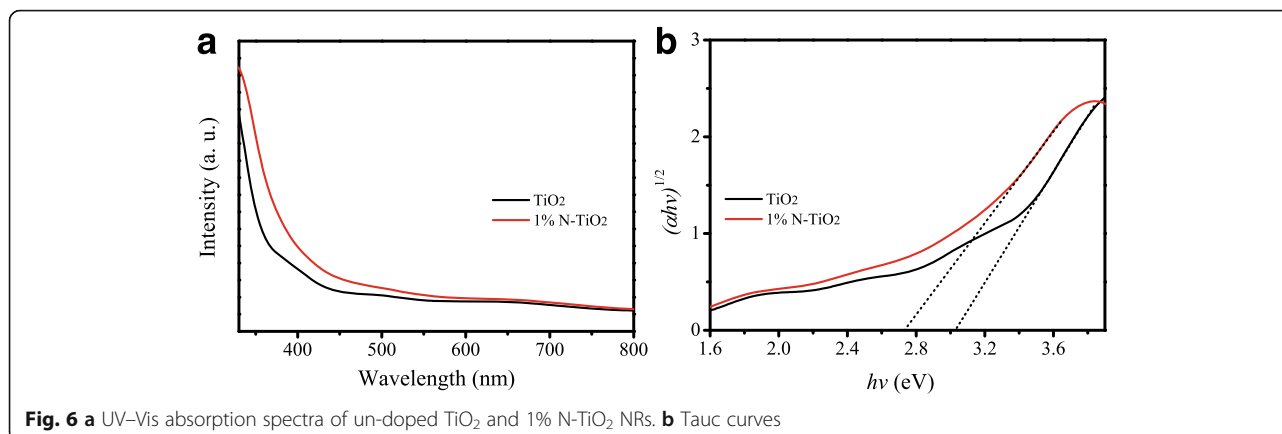


Fig. 5 Plane-view SEM images of un-doped TiO₂ (**a**) and 1% N-TiO₂ NRs (**b**). Cross-sectional SEM images of un-doped TiO₂ (**c**) and 1% N-TiO₂ NRs (**d**)

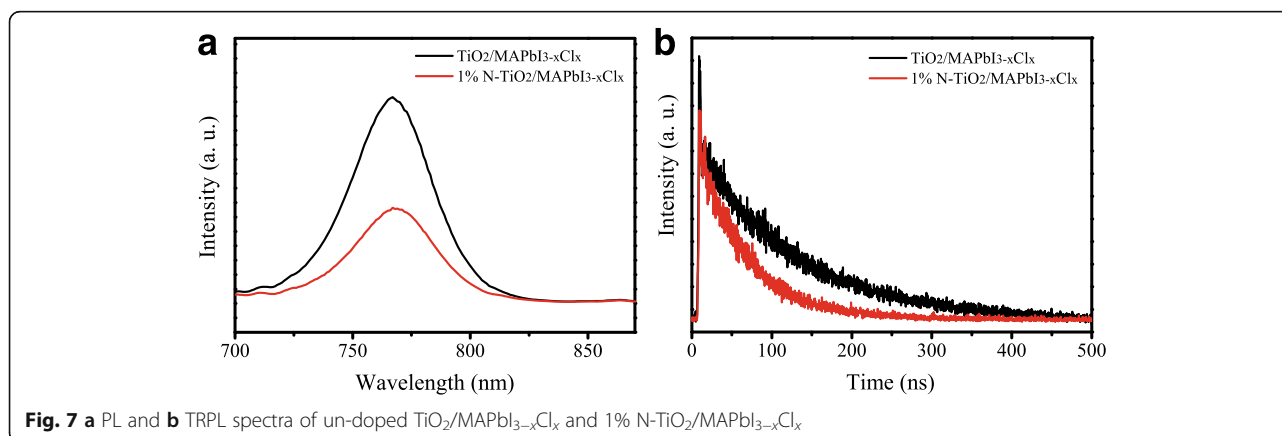


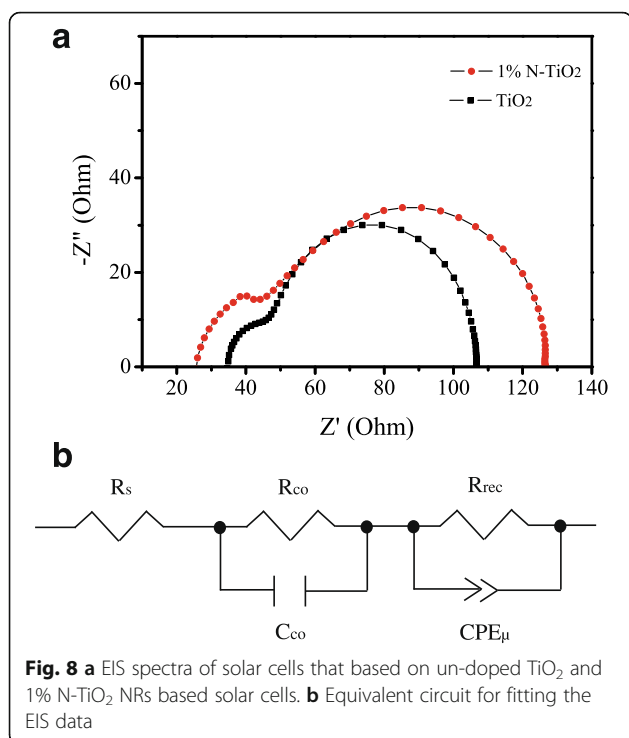
MAPbI_{3-x}Cl_x and 1% N-TiO₂/MAPbI_{3-x}Cl_x. The intensity of PL spectrum was decreased due to the electron transfer when the MAPbI_{3-x}Cl_x film contacts with TiO₂ nanorod layer. The intensity decrease for N-TiO₂ is more significant than that for un-doped TiO₂, which indicates the electron transfer from MAPbI_{3-x}Cl_x layer to N-TiO₂ is more effective than to un-doped TiO₂.

Figure 7b shows the TRPL spectra of un-doped TiO₂/MAPbI_{3-x}Cl_x and 1% N-TiO₂/MAPbI_{3-x}Cl_x. The TRPL curves were analyzed with a bi-exponential decay function containing a fast decay (τ_1) component and a slow decay component (τ_2), and the detailed parameters are listed in Table 2. The fast decay component would be the result of free carrier transportation from MAPbI_{3-x}Cl_x to TiO₂, and the slow decay component could be the result of radiative decay [29, 30]. In the case of un-doped TiO₂/MAPbI_{3-x}Cl_x, the fast decay time is 55.1 ns, and the slow decay time is 121.4 ns, while their weight fractions are 31.3 and 67.7%, respectively. Compared with those of un-doped TiO₂/MAPbI_{3-x}Cl_x, the fast decay lifetime of N-TiO₂/MAPbI_{3-x}Cl_x is decreased to 36.4 from 55.1 ns, and the slow decay life time to 109.5 from 121.4 ns, while the weight fraction of fast decay is increased to 35.1 from

31.3%. This suggests that N-TiO₂/MAPbI_{3-x}Cl_x interface presents a faster charge transfer and induced charge recombination than the un-doped TiO₂/MAPbI_{3-x}Cl_x interface. The performance difference between the solar cells based on un-doped TiO₂ and N-doped TiO₂ NRs could be due to the property change of ETM, which affects the charge behavior in the interfaces [31, 32].

To understand the charge transfer behavior of the solar cells, electrochemical impedance spectrum (EIS) was measured. Figure 8a shows the Nyquist plots of solar cells that based on un-doped TiO₂ and 1% N-TiO₂ NRs. The EIS contains two RC arcs. The arc at high frequency is originated from the contact resistance of the interfaces and that at low frequency is attributed to recombination resistance (R_{rec}) and chemical capacitance (C_{μ}) of the device [33, 34]. Figure 8b shows an equivalent circuit which was applied to fit the EIS. Table 3 lists the fitting parameters. The solar cells based on N-TiO₂ present smaller series resistance and larger recombination resistance than those on un-doped TiO₂. This demonstrates that the charge transport ability was enhanced and the carrier recombination rate was induced for the device on N-TiO₂. There are many surface and





bulk trap states due to oxygen vacancies for TiO₂. It demonstrated that the incorporation of N atom into the lattice of TiO₂ can decrease the traps density probably due to oxygen vacancy reduction [18, 35]. The smaller series resistance of N-TiO₂ NRs could be due to the decreased traps density. The larger recombination resistance of N-TiO₂ NRs may be contributed to the inactive N element leading to the increase of surface resistance [36, 37].

Conclusions

In the present study, N-TiO₂ NRs were synthesized with hydrothermal method, and perovskite solar cells based on them were fabricated. Compared with the solar cells based on un-doped TiO₂, solar cells based on N-TiO₂ present an enhanced performance. The solar cell performance was optimized by changing the N doping contents. The PCE of solar cells based on N-TiO₂ with the N doping content of 1% (N/Ti, atomic ratio) has been achieved 11.1%, which was 14.7% higher than that of un-doped TiO₂-based solar cells. To explain this phenomenon, some investigations were performed. The results indicate that the larger V_{oc} could be due to the larger conduction band offset resulting from the smaller

Table 2 Parameters of the TRPL spectra

Sample	τ_1 /ns	% of τ_1	τ_2 /ns	% of τ_2
TiO ₂ /MAPbI _{3-x} Cl _x	55.1	31.3	121.4	67.7
1% N-TiO ₂ /MAPbI _{3-x} Cl _x	36.4	35.1	109.5	64.9

Table 3 Fitting parameters of EIS data

Sample	R_s/Ω	R_{co}/Ω	R_{rec}/Ω	CPE-T/F
TiO ₂	34.8	59.3	12.7	6.3×10^{-6}
1% N-TiO ₂	27.5	23.7	75.1	5.9×10^{-6}

energy band gap for N-TiO₂, and the enlarged I_{sc} could be attributed to the faster electron transfer and reduced recombination rate for N-TiO₂ NRs. These induce the enhanced performance of the solar cells based on N-TiO₂ NRs.

Additional file

Additional file 1: Supplementary Material. **Figure S1.** EDS spectrum of 1% N-TiO₂ NRs. **Figure S2.** Plane-view SEM images of un-doped TiO₂ (A), and 1% N-TiO₂ NRs(B). **Figure S3.** Cross-sectional SEM image of perovskite solar cells. (DOCX 826 kb)

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Authors' Contributions

Z-LZ, J-FL, and Y-LM carried out the main part of experiment and drafted the manuscript. The other authors provided assistance with the 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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