



# A simple post-treatment with urea solution to enhance the photoelectric conversion efficiency for TiO<sub>2</sub> dye-sensitized solar cells



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## ARTICLE INFO

### Keywords:

C<sub>3</sub>N<sub>4</sub>

TiO<sub>2</sub>

Post-treatment

Dye-sensitized solar cells (DSSCs)

## ABSTRACT

Dye-sensitized solar cells (DSSCs) are performed using C<sub>3</sub>N<sub>4</sub> post-treated TiO<sub>2</sub> particles as photoanode materials. The photoanode materials were synthesized via a simple method by which a thin layer of C<sub>3</sub>N<sub>4</sub> was coated onto the surface of TiO<sub>2</sub> particles after the TiO<sub>2</sub> was dipped in urea solution followed by calcination properly. The experimental results show that the photoelectric conversion efficiency of the TiO<sub>2</sub> DSSCs is remarkably improved. The measurements of *I*-*V* characteristic along with the analysis of electrochemical impedance spectroscopy indicate that the C<sub>3</sub>N<sub>4</sub> layer can expand the visible light absorption, suppress the recombination reaction of the e<sup>-</sup>-h<sup>+</sup> pair and increase both the open circuit voltage and the short-circuit photocurrent density. The mechanism of the improved performance of TiO<sub>2</sub> DSSCs by C<sub>3</sub>N<sub>4</sub> coating coating is proposed. The post-treatment method recorded in this work can be easily operated and achieve an enhanced photoelectric performance of DSSCs.

## 1. Introduction

Dye-sensitized solar cells (DSSCs) have attracted much attention because of the notable advantages including low cost, plentiful raw materials, simple process and low pollution [1–7]. The real research for DSSC began on 1991, when Gratzel et al. successfully fabricated the original DSSCs with porous TiO<sub>2</sub> film being used as the photoanode, which achieved a photoelectric efficiency of 7% [8]. Recently, perovskite sensitized solid-state DSSCs have achieved a photoelectric efficiency of 15%, bringing DSSCs back in the spotlight [9]. However, compared with solid-state DSSCs, photoelectric efficiency of the original DSSCs does not make a great breakthrough during the recent twenty years [10–16].

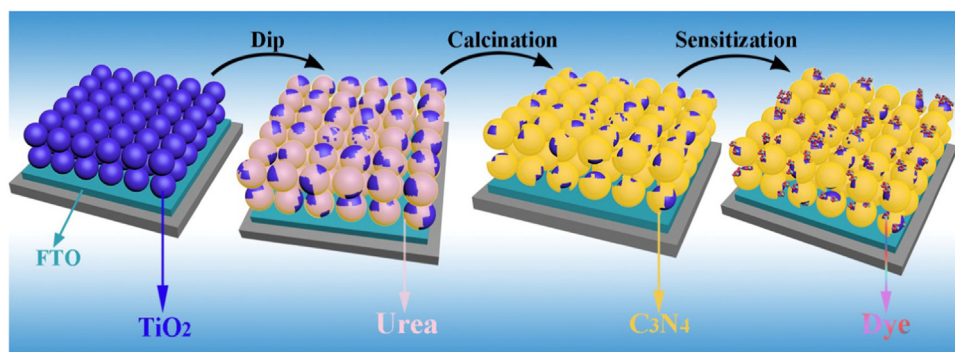
The principle of original DSSCs is that [17], dye molecules are adsorbed on TiO<sub>2</sub> surface, and absorb sunlight, the electrons on the dye molecules are excited and transfer into the conduction band (CB) of TiO<sub>2</sub>, the electrons are transmitted to outer circular across the TiO<sub>2</sub> porous film. At the same time, the oxidized dye molecule is reduced by I<sup>-</sup> in electrolyte and thereby rebirths. In addition to the above reaction, there are also several undesired reactions which affect the photoelectric

efficiency therein, such as the recombination reaction of photo induced electron-hole pairs which causes great loss of the photo induced electrons, and greatly suppresses the photoelectric conversion efficiency of DSSCs.

In order to improve the conversion efficiency of the original DSSCs, many efforts have been conducted, such as: (1) Using larger TiO<sub>2</sub> particles as the scattering centers, to increase the light scatter and the optical path for better utilization of light. Alternatively, creating pores to increase the specific surface area so as to increase the dye absorption, and thereby increase the absorption of light. In addition to the control of the morphology, other methods for increasing the absorption of light, such as ion doping or noble metal loading, are also adopted. (2) Combining with high conductivity material, such as carbon nano tube (CNT) or graphene, to improve the electron transmission and the separation of photogenerated pair of charge carrier. (3) Performing pre- or post- treatment for photoanode, such as treating with TiCl<sub>4</sub> or TiCl<sub>3</sub> solution, so as to repair the surface defect of TiO<sub>2</sub>, decrease electron recombination. (4) Before sensitized by dye, coating the TiO<sub>2</sub> film with solid-state semiconductor materials which have a higher conduction band than TiO<sub>2</sub> to form an energy level barrier, so as to prevent

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**Scheme 1.** Schematic illustration of preparation of the  $\text{TiO}_2\text{-C}_3\text{N}_4$  composites and their application as multifunctional cover layer on the photoanode for DSSCs.

recombination of photogenerated pair of charges. Common coating materials include  $\text{MgO}$ ,  $\text{ZnO}$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{SnO}_2$  and so on [18–23].

Particularly, the CB of  $\text{C}_3\text{N}_4$  ( $-1.12$  eV) is higher than CB of  $\text{TiO}_2$  ( $-0.29$  eV), thus  $\text{C}_3\text{N}_4$  can be used as the blocking layer to form an energy barrier, which can effectively suppress the recombination reaction and indicate very promising future [24–26]. Therefore, in this work, a simple post-treatment method has been developed to synthesize  $\text{C}_3\text{N}_4$  coated  $\text{TiO}_2$  composites, as shown in Scheme 1, to improve the photoelectric conversion efficiency of the original DSSCs.

## 2. Experimental

### 2.1. Chemicals

All chemicals in this work were used analytical grade and without any further purification. Poly (ethylene glycol) (PEG) was bought from Sinopharm Chemical Reagent Co. Ltd and its average molecular weight (Mw) was ca. 2000. Other chemicals were purchased from Shanghai Chemical Reagent Company. Deionized water was used in all experiments.

### 2.2. Preparation of $\text{TiO}_2$ photoanode films

$\text{TiO}_2$  photoanode films are prepared by doctor-blade method [8], and kept with the same nominal area and same thickness by using the adhesive tape. The F-doped  $\text{SnO}_2$ -modified conductive glass (FTO glass) (Nippon,  $14 - 20 \Omega \text{sq}^{-1}$ ) is treated as follows: firstly processed for 15 min by ultrasound in the ethanol and distilled water respectively, and then dried in the air. Secondly, commercial P25 is used as the raw material of photoanode film. The photoanode paste is prepared by mixing 0.5 g P25 powers, 0.2 g polyethylene glycol (PEG, MW2000) and 2 ml water into the mortar with subsequent grind for 30 min, and smeared on FTO glass. At last, the prepared films are formed after the paste-FTO plate is calcinated in a muffle at  $450^\circ\text{C}$  for 30 min.

### 2.3. Post-treatment of the $\text{TiO}_2$ photoanode films

The post-treatment of the  $\text{TiO}_2$  photoanode films is performed by dipping the bare photoanode films into the urea solution with different concentrations, including 1, 2 and 3 g/ml, at  $50^\circ\text{C}$  for 30 s. The photoanode films after post-treatment are calcinated in a crucible in muffle at  $500^\circ\text{C}$  for 1 h immediately after being pulled out from the urea solution. The corresponding samples are labeled as CP-1, CP-2 and CP-3, accordingly. The bare photoanode film is denoted with CP-0. Clearly, the  $\text{C}_3\text{N}_4$  layer covering the photoanode is obtained via calcinating the urea adsorbed on the  $\text{TiO}_2$  particle surface.

### 2.4. Assembly of the new DSSC

Dye sensitization was carried out by immersing the as-prepared

photoanode films into 0.3 mM N719 ruthenium dye (Solaronix S. A., Lausanne, Switzerland) ethanol solution for 12 h at  $50^\circ\text{C}$  in a sealed beaker. Then, the sensitized films were washed by absolute ethanol, and then dried in an oven at  $80^\circ\text{C}$  for 1 h. The new DSSCs were assembled in a typical sandwich-type cell by placing a platinum counter electrode side-by-side with the dye-sensitized  $\text{C}_3\text{N}_4\text{-TiO}_2$  electrode separated by a  $\sim 50 \mu\text{m}$  polymer spacer. The active area of the cell was  $4 \times 4 \text{mm}^2$ . The electrolyte was prepared with 0.3 mol/L LiI (Sigma-Aldrich Corp., St. Louis, MO, USA), 0.05 mol/L  $\text{I}_2$  (Sigma-Aldrich Corp., St. Louis, MO, USA), 0.6 mol/L 1-propyl-3-methylimidazolium iodide (Suzhou Zhongsheng Chemical Co., Ltd., Suzhou, China), and 0.5 mol/L tert-butylpyridine (Sigma-Aldrich Corp., St. Louis, MO, USA) in anhydrous acetonitrile (Shanghai Chemical Reagent Factory of China, Shanghai, China). In order to ensure the same batch of electrolyte used for each sample, a large amount of electrolyte solution was prepared and then divided into several smaller glass vials. All vials were sealed and stored in a vacuum desiccator. The electrolyte was injected into the middle part of the cell from the edge and spread into the inner space of the cell by capillary force, and the cell was tested immediately.

### 2.5. Characterization and measurements

The phases of as-prepared samples were studied by X-ray diffraction (XRD) using a HZG41B-PC X-ray diffractometer (Rigaku, Japan) with  $\text{Cu K}\alpha$  radiation ( $\lambda = 0.15418 \text{nm}$ ). The structure and morphology of the particles scraped from the photoanode film were examined by transmission electron microscopy (TEM) and high resolution transmission electron microscopy (HRTEM), which were conducted on a JEM-2100F electron microscope (JEOL, Japan). The absorption spectra of the as-prepared samples were recorded using a UV-vis diffuse reflectance spectroscopy (DRS) (UV-2550, Shimadzu, Japan), with  $\text{BaSO}_4$  as the reference. The dye adsorption was analyzed by the UV-vis spectrophotometer (UV-2550, Shimadzu, Japan), with water as the reference. The thermal gravimetric analysis (TGA) of the as-prepared samples was performed using a DTG-60H analyzer (Shimadzu Corp., Tokyo, Japan). About 5 mg of each sample was heated to  $900^\circ\text{C}$  in air at a rate of  $10^\circ\text{C min}^{-1}$ . The Fourier transform infrared spectra (FTIR) of the samples were recorded using an IRAffinity-1 FTIR spectrometer (Shimadzu Corp., Tokyo, Japan).

The electrochemical impedance spectroscopy (EIS) measurements were performed by a computer-controlled electrochemical workstation with impedance analyzer (CHI660C Instruments, Shanghai Chenhua Instrument Corp., Shanghai, China) in a two-electrode configuration. The photoanode was used as a working electrode and the Pt electrode was used as a counter electrode. The measurements were carried out by applying bias on the open circuit voltage ( $V_{\text{OC}}$ ) and recorded over a frequency range of  $0.005 - 10^5 \text{Hz}$  with ac amplitude of 10 mV.

The photocurrent-voltage ( $I$ - $V$ ) characteristic curves were measured using the electrochemical analyzer (CHI660C Instruments), under the irradiation of a solar simulator (Newport 91,160) with a light intensity

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