



Short communication

A microfluidic all-vanadium photoelectrochemical cell with multi-nanostructured TiO₂ photoanodeHao Feng^{a,b}, Xiaohong Jiao^{a,b}, Rong Chen^{a,b,*}, Xun Zhu^{a,b}, Qiang Liao^{a,b}, Dingding Ye^{a,b}, Biao Zhang^{a,b}^a Key Laboratory of Low-grade Energy Utilization Technologies and Systems (Chongqing University), Ministry of Education, Chongqing, 400030, China^b Institute of Engineering Thermophysics, Chongqing University, Chongqing, 400030, China

HIGHLIGHTS

- A multi-nanostructured TiO₂ photoanode is developed for μVPEC.
- The developed photoanode ensures large specific surface area and plentiful pore structure.
- The photon and mass transport is enhanced and the light scattering is promoted.
- Photoelectrochemical activity and vanadium ion conversion rate are improved.

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ABSTRACT

All-vanadium photoelectrochemical cell possesses the inherent merits of both the all-vanadium redox flow battery and the solar energy utilization, which can directly store the solar energy in the form of the chemical energy. A promising design of the photoelectrochemical cell should provide large active surface area and ensure efficient transport of reactants, photons and charge carriers. To meet the above-mentioned requirements, a microfluidic all-vanadium photoelectrochemical cell with a multi-nanostructured TiO₂ photoanode consisting of the interwoven nanotube bundles and the interspersed nanorods and nanoparticles, is proposed in this work for enhancing the solar energy storage. The miniaturization design can intensify the photon and mass transport because of the intrinsic large specific surface area, while the multi-nanostructured photoanode can offer more active surface area and plentiful pore structure, which can also enhance the photon and mass transport and avail the light scattering to deplete the photons. Such synergistic effect is able to promote the photoelectrochemical reaction efficiency. Experimental results show that the proposed photoelectrochemical cell exhibits excellent photoresponse and operation stability. Under each operation conditions, compared to conventional TiO₂ nanoparticles stacked photoanode, the proposed photoanode can remarkably improve the vanadium ion conversion rate, demonstrating its promising potential in real applications.

1. Introduction

Photoelectrochemical cell (PEC) is now a promising candidate to provide a practical technology of directly converting the chemical energy stored in the wastewater into electricity by using solar energy [1–5]. However, the intrinsic defect of intermittence for sunlight is one of the barriers to inhibit the practical applications of this technology. Under this circumstance, the solar energy storage in the form of chemical energy, such as hydrogen, redox couples, etc., through the PEC technology for subsequent usage becomes one of the most important

means for solar energy applications, receiving plenty of attention [6–9]. Among the chemicals for storing the solar energy by the PEC technology, the reversible redox pairs of VO₂⁺/VO²⁺ and V³⁺/V²⁺ have been considered as a competitive candidate for solar energy storage due to its salient merits of large storage capacity and the avoidance of cross contamination in the electrolyte [10–12]. In principle, photosensitive semiconductors are usually utilized at the photoanode of PECs. Upon the illumination, the semiconductors can capture photons to generate the electron-hole pairs. Due to the strong oxidation ability, the VO²⁺ can be oxidized to VO₂⁺, while the electrons flow to the cathode to

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drive the reduction reaction of $V^{3+} + e^- \rightarrow V^{2+}$. In this way, the solar energy is stored by the redox couples [9,13].

Benefited from the intrinsic advantages of chemical stability, abundance in nature and low-cost, semiconductor TiO_2 has become one of the most widely utilized photocatalyst materials and been applied in various photoelectrochemical systems, such as water splitting [6,14], wastewater treatment [1], CO_2 photoreduction [7], etc. In conventional, the photoanode was commonly prepared by stacking the TiO_2 nanoparticles on conductive glass to form a TiO_2 film, which could be realized through the wet-spraying, screen-printing, doctor-blade and brushing [9,15–18], etc. In this case, the inherent defects like low active surface area and easy recombination of the photo-induced electron-hole pairs as well as the poor photon and mass transfer greatly inhibit the photocatalytic quantum efficiency [19]. As a result, the synthesis of TiO_2 nanomaterials with sufficiently high active surface area and the construction of the spatial photoanode structure for furnishing adequate active sites and promoting the transport of both the reactants and photons as well as charger carriers become valid strategies for the photocatalytic performance improvement [20,21].

Recently, TiO_2 preparation from various methods in the form of nanotubes [21,22] has been demonstrated to have larger specific surface area and pore volume compared with the TiO_2 nanoparticles [19]. Such vigorous pore structure accompanied with high active surface area can be considered as a favorable nanostructured photocatalyst. On the other hand, regarding the PEC structure, the conventional design is the double-compartment separated by a proton exchange membrane [9,23–26]. Such design leads to, not only the increase of both the photon, ion and mass transfer resistances, but also the large light attenuation and poor light distribution, which greatly restrains the photoelectrochemical reaction efficiency. While the microfluidics can overcome the above mentioned issues when it is adapted to the PEC design due to its intrinsic advantage of the tremendous specific surface area [13,27–29]. As a result, the integration of the TiO_2 nanotubes and the microfluidics into the PECs is expected to be able to intensify the photoelectrochemical reaction and solar energy conversion efficiency.

In the present work, a microfluidic all-vanadium photoelectrochemical cell (μ VPEC) with a multi-nanostructured TiO_2 photoanode, consisting of the interwoven nanotube bundles and the interspersed nanorods and nanoparticles, was proposed for storing the solar energy. Various analyses were performed to obtain the morphology and material properties of the photoanode. The performance of the developed all-vanadium photoelectrochemical cell was also investigated under various operating conditions.

2. Materials and methods

2.1. Photoanode preparation

The preparation of the photoanode can be divided into two steps: the synthesis of the TiO_2 nanotube photocatalyst and the construction of the spatial TiO_2 layer on the Fluorine-doped SnO_2 (FTO) glass. In the first step, the commercial TiO_2 powders (P25) underwent an alkaline hydrothermal process at 130 °C for 24 h for the nanotubes preparation [21]. For the spatial TiO_2 layer construction, to intensify the conductivity, the bottom densely packed nanocrystalline TiO_2 layer was pre-prepared onto the FTO glass by the spin-coating method [13]. Thereafter, the synthesized TiO_2 nanotubes were dispersed in an aqueous solution and sprayed on the top of the bottom densely packed nanocrystalline TiO_2 layer. After that, to ensure a strong adhesion of the porous nanostructured TiO_2 layer to the FTO glass, the sample was placed in a tube furnace and calcined at 550 °C for 2 h. The final loading of the nanostructured TiO_2 photocatalyst was about 1.0 mg/cm² with the surface area of about 1.0 cm × 1.0 cm. In addition, the commercial P25 TiO_2 nanoparticles were also used to prepare the conventional TiO_2 photoanode with the same loading for fair comparison.

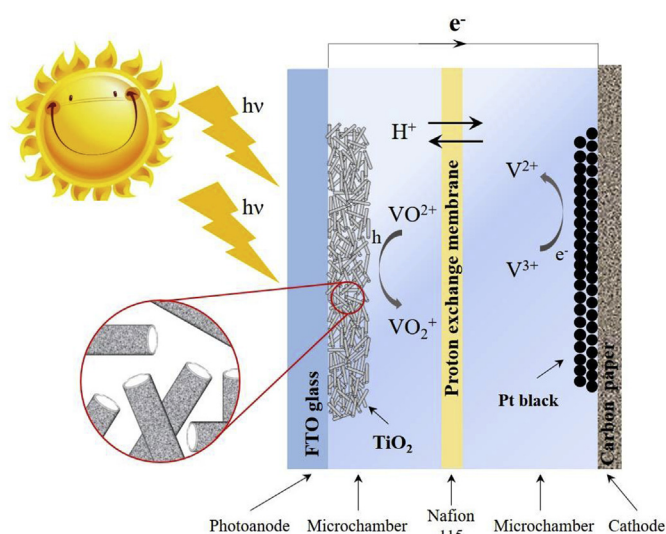


Fig. 1. Schematic illustration of the μ VPEC.

2.2. Materials characterization

The external feature and surface morphology of the synthesized TiO_2 nanotubes were characterized by using a high-resolution transmission electron microscope (HR-TEM, Tecnai G2 F20, FEI) and field emission scanning electron microscope (FESEM, S4800, Hitachi), respectively. The crystalline phase and structure analysis of the TiO_2 samples were conducted with X-ray diffraction (XRD) using a D8 Advance powder X-ray diffractometer (Bruker) with $Cu K\alpha$ radiation. Pore structure of the photocatalysts was characterized by the N_2 adsorption-desorption isotherm at 77.35 K using an ASAP 2460 surface area and porosity analyzer (Micromeritics, USA). BET specific surface area was calculated according to the Brunauer-Emmett-Teller (BET) model. Pore size distribution was evaluated from the adsorption branch of the isotherm using the Barrett-Joyner-Halenda (BJH) method. Total volume was calculated based on the N_2 amount adsorbed at a relative pressure of 0.99.

2.3. Experimental setup

The schematic of the μ VPEC is shown in Fig. 1. The cell consisted of a TiO_2 photoanode, two microchambers with the depth of 500 μ m, a Nafion[®] 115 membrane to separate the microchambers, and a Pt coated carbon paper with the loading of 1 mg/cm² and surface area of 1.0 cm × 1.0 cm as the cathode. A four-channel syringe pump (Longer-Pump, China) was used to simultaneously inject both the photoanode and cathode electrolyte solutions into the cell. A 300 W Xe lamp (Aulight, China) was utilized to simulate the sunlight and illuminate the photoanode. The photocurrent was recorded by a data collecting instrument (Agilent, USA) connected with a computer.

3. Results and discussion

3.1. Photocatalyst characterization

The calcination is an essential process to ensure a strong adhesion of the porous nanostructured TiO_2 layer to the FTO glass and obtain the appropriate crystalline phase, whereas the increased temperature to 550 °C may result in the sintering of the nanotubes [21]. Thus, the morphology, pore structure and crystalline phase of both the synthesized TiO_2 nanotubes dried at 100 °C (TNT) and the TiO_2 nanotubes calcined at 550 °C (TNT-550) as well as the conventional P25 TiO_2 nanoparticles were first discussed. As shown in Fig. 2a, the TEM image

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