



Photoelectrocatalytic oxidation of ascorbate promoted by glucose and tris-(hydroxymethyl)-amino methane on cadmium sulfide/titanium dioxide electrodes for efficient visible light-enhanced fuel cells

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ABSTRACT

Glucose (GLU) and tris-(hydroxymethyl)-amino methane (Tris) have been introduced to promote the photoelectrocatalytic oxidation of ascorbate (AA) on binary CdS-TiO₂ nanoparticle-modified electrodes for efficient photoelectrochemical fuel cells. The oxidative peak height of AA shows an increase with the rise of AA, GLU, OH⁻ or Tris concentration under dark or visible light irradiation conditions. The photoelectrocatalytic activities of TiO₂ nanoparticles are dependent on the calcination temperature ranged between 250 and 850 °C. The TiO₂-450 generated from the calcination treatment at 450 °C is combined with CdS nanoparticles to achieve a CdS/TiO₂-450 electrode with high photoelectrocatalytic activity towards the oxidation of AA and GLU, for which the oxidation mechanism is discussed. While employing CdS/TiO₂-450 as anode, carbon felt (CF) as cathode, 0.1 mol L⁻¹ AA-0.1 mol L⁻¹ GLU as fuels, Na₂SO₃-Na₂S as sulfur-containing sacrificial agents, and 60 mL min⁻¹ O₂ as oxidant, the visible light-assisted fuel cell shows synergistically enhanced performances. The open-circuit photovoltage (*V*_{OC}) and short-circuit photocurrent density (*I*_{SC}) are 0.813 V and 0.568 mA cm⁻², and the maximum power density (*P*_{max}) is 35.56 μW cm⁻² upon visible light irradiation of 0.18 mW cm⁻². The present results provide an interesting platform for the effective utilization of renewable energy sources.

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1. Introduction

There is an increasing focus on the conversion and utilization of renewable energy sources [1]. In nature, plants harvest solar energy for the photosynthesis of glucose (GLU), ascorbic acid (AA) and other chemical fuels [2], and humans convert chemical energy from the photosynthesized fuels through a series of chemical reactions with oxygen or other oxidizing agents [3,4]. Inspired by nature, the photoelectrochemical fuel cell has been considered as a promising and low cost technology to convert solar energy and biomass energy into electric energy [5–8]. Among various components used to assemble the photoelectrochemical fuel cell, the semiconductor-based photosensitizers play an important role in photocatalytic processes [9–12]. Cadmium sulfide (CdS) with a band gap of 2.42 eV that matches the visible spectral range of solar irradiation is one of the most promising *n*-type semiconductor photosensitizers

for photovoltaic and photocatalytic applications [13–15]. Unfortunately, the pure CdS is easily photocorroded in aqueous media due to its oxidized sulfide ion by photo-generated holes [16]. It is therefore vital to develop suitable methods to improve the anti-photocorrosion performance of CdS-based photoanodes. (i) An integration of CdS with other photosensitizers is favorable for overcoming its intrinsic defects [17,18]. (ii) The sulfur-containing electrolytes like Na₂S and Na₂SO₃ act as sacrificial agents to sustain the integrity of CdS [19]. (iii) The presence of organic pollutants or fuels facilitates to increase the stability of photo-excited CdS layers [20].

Since nano-TiO₂ was used to promote the photoelectrochemical generation of hydrogen [21], it has been regarded as a kind of distinguished photocatalysts widely used for solar energy conversion and environmental remediation because of its low cost, strong oxidizing ability and long-term stability against photochemical corruptions [22–24]. However, the maximum utilization of the visible spectrum and efficient charge transport is always limited by wide band gap energy, fast electron-hole recombination and slow hole scavenging rate [25,26]. To inhibit the photocorrosion of CdS

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and to enlarge simultaneously photo absorption range of TiO₂ to visible light, they are often coupled each other because of their matched band structures, and complementary spectral and photocatalytic properties [27–29]. Nevertheless, there are rare reports on the fabrication and evaluation of photoelectrochemical fuel cells employing visible light-excited CdS/TiO₂ as anode and GLU-AA as fuels.

GLU and AA usually act as primary energy sources and strong anti-oxidative agents of living cells, respectively [30,31], therefore, many efforts have been made to fabricate a GLU or AA fuel cell in the absence and presence of light irradiation [32,33]. The electrocatalytic or photocatalytic oxidation of GLU and/or AA has been extensively studied by using enzyme, coenzyme, redox mediator, metal and/or metal oxide, and photosensitizer as catalysts [34–36]. Despite the fact that the electrocatalytic and/or photocatalytic oxidation of AA on a CdS or [Ru(tatp)₃]²⁺/TiO₂ (tatp = 1,4,8,9-tetraaza-triphenylene) electrode enhanced by GLU has been reported in our previous studies [37,38], the investigations into the fabrication of high-performance photoelectrochemical GLU-AA fuel cells behind these phenomena are still ongoing.

Tris is an important organic compound extensively used in chemical, biochemical and biomedical applications to stabilize pH of a solution (pK_a = 8.07, 25 °C) [39]. It contains amino and hydroxyl functional groups, which have potential to bind biomolecules or involve in some of biochemical reactions [40–42]. Therefore, it is interesting to know whether Tris could be used to improve the photoelectrocatalytic oxidation of AA and GLU coexisting in biological fluids.

Herein, we aim to design an effective visible light-enhanced fuel cell by evaluating the effects of anode materials (CdS and TiO₂ calcined between 250 and 850 °C) and electrolyte compositions (Tris, OH⁻ and Cl⁻ ions, Na₂S and Na₂SO₃) on the photoelectrocatalytic oxidation of AA-GLU. Fortunately, the CdS/TiO₂-450 binary electrode shows photoelectrocatalytic activity towards the synergistically enhanced oxidation of AA and GLU. On the other hand, the OH⁻ and Cl⁻ ions, Tris, Na₂S and Na₂SO₃ added in the anode compartment can improve the visible-light-excited AA-GLU fuel cell performances. To our knowledge, this work presents the first example of photoelectrochemical fuel cells employing AA-GLU fuels and binary semiconductor photoanodes dependent on calcination temperature.

2. Experimental section

2.1. Instruments and methods

All the electrochemical measurements were performed using a CHI660a electrochemical working station (Shanghai, China). Unless otherwise noted, the modified electrode was employed as working electrode, a titanium sheet as counter electrode and a saturated calomel electrode (SCE) as the reference electrode. Linear sweep voltammetry was performed at 0.01 V s⁻¹ scan rate. Electrochemical impedance spectroscopy (EIS) was measured on an Autolab PGSTAT-30 electrochemical system (Ecochemie, The Netherlands), controlled in a frequency range from 100 kHz to 0.1 Hz with an amplitude of 10 mV. Ascorbic acid (AA) from Qiyun Biotechnology Co. (Guangzhou, China) at an approximately physiological condition was verified to dominantly be ascorbate (pK₁ = 4.2 and pK₂ = 11.6) [43]. GLU, Tris, and other reagents were used as received. The electrolyte solution (I) was 0.01 mol L⁻¹ Tris/0.05 mol L⁻¹ NaCl of pH 7.2 prepared with doubly distilled water. The Zeiss Ultra55 field-emission scanning electron microscope (SEM, Germany), JEM-2100 high-resolution transmission electron microscope (HR-TEM, Japan) and Bruker D8 Advance X-ray powder diffractometer (XRD, Germany) were employed to analyze the

appearance and crystal structure of the as-prepared samples.

2.2. Preparation of TiO₂-T electrodes

The commercial Degussa P25 TiO₂ powder was added to a crucible, following by calcination treatment in a muffle furnace for 1 h at temperatures (*T*) between 250 and 850 °C, and then cooled to room temperature, yielding the TiO₂ powders labeled as “TiO₂-T”. The un-calcined P25 sample was denoted as TiO₂-no. Subsequently, the as-calcined TiO₂ powders of 0.015 g were added to 1.0 mL of ethanol to prepare a colloidal solution with the aid of ultrasonic agitation for 1 h. An indium-tin oxide (ITO, 10 Ω sq⁻¹) sheet (Shenzhen Nanbo Co., China) was served as the substrate to support the TiO₂ coating (0.56 cm²), which was formed by placing drops of TiO₂ colloidal solutions for 10 s. The resulting TiO₂-T electrode was kept at a constant temperature of 40 °C for 2 h to evaporate the solvent.

2.3. Preparation of CdS/TiO₂-450 electrode

The TiO₂-450 electrode was used as the substrate to prepare a CdS/TiO₂-450 electrode using previously published methods [44]. Briefly, a CdS coating was deposited on TiO₂-450 electrode by 100-cycle potential scanning between -0.3 and -0.9 V at 0.05 V s⁻¹ scan rate in 0.01 mol L⁻¹ CdCl₂/0.1 mol L⁻¹ Na₂S₂O₃ (pH 2.0) solutions using a titanium counter electrode and a SCE reference electrode. After cleaning the resultant electrode with doubly distilled water for 10 min, it was kept at a constant temperature of 40 °C for 2 h. The average sizes of CdS and TiO₂-450 particles were about 5 nm and 30 nm as revealed by TEM images in Fig. 1.

2.4. Assembly of visible light-enhanced fuel cell

The photoelectrochemical AA-GLU/O₂ fuel cell was schematically shown in Fig. 1. The CdS/TiO₂ and carbon felt (CF) electrodes were employed as photoanode and cathode, both of which were placed in two quartz compartments separated by a Nafion 117 proton exchange membrane (DuPont). The anodic and cathodic compartments were filled with 2.0 mL of solution I containing fuels/sacrificial agents and injecting O₂ at different flow rates via a gas-flow controller, respectively. The non-conductive backside of the photoanode faced a tungsten halogen lamp mostly located between 400 and 1100 nm with power density of 0.18 mW cm⁻².

All the measurements were performed at room temperature (25–27 °C).

3. Results and discussion

3.1. Photoelectrocatalytic oxidation of AA and GLU on CdS/TiO₂-450 electrode

Linear sweep voltammogram (LSV) of 0.25 mmol L⁻¹ AA and 5.0 mmol L⁻¹ GLU on CdS/TiO₂-450 electrode in a dark box shows an oxidative peak (curve 1 in Fig. 2a), for which the onset potential and peak potential are -0.032 V and 0.276 V, respectively. Compared with the LSVs of AA or Glu alone on CdS/TiO₂-450 electrode in curves 2 and 3 of Fig. 2a, no detectable oxidation response of GLU alone is observed. However, the presence of GLU increases the oxidative peak height of AA, indicating that AA not only is electrochemically oxidized on CdS/TiO₂-450 electrode, but also its oxidation can be enhanced by GLU.

To throw the light on the catalytic oxidation of AA and GLU on CdS/TiO₂ electrode, various major influence factors are further discussed.

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