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Dual template synthesis and photoelectrochemical performance of 3-D hierarchical porous zinc oxide

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ABSTRACT

In this work, a novel hierarchical porous ZnO is successfully synthesized through a sol–gel method, in which a kind of biological material is used as hard template, block copolymer Pluronic F127 as soft template. The phase and morphology of the products are characterized by X-ray diffraction analysis (XRD), scanning electron microscopy (SEM), and transmission electron microscope (TEM). The results show that the as-prepared ZnO with a hierarchical porous architecture is assembled by multiple-layered porous nanosheets, of which the pore structure is highly ordered. The photocatalytic activity of the as-prepared ZnO is evaluated by photodegradation reaction of methylene blue. The photoelectrochemical (PEC) property of the hierarchical porous ZnO film is also investigated in this work.

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Introduction

Zinc oxide (ZnO), a semiconductor with a bandgap of 3.37 eV and a large exciton binding energy of 60 meV [1], has attracted much more attention owing to its promising application in solar cells [2], sensors [3,4], photocatalysis [5], etc. One of the most important applications of ZnO is as a photocatalyst in environmental protection [6], under the irradiation of UV light, ZnO can produce electron–hole pair, and exhibit excellent catalytic properties. Hence, ZnO can be used to degrade many organic pollutants in mild conditions [7–10]. The key approach for improving its photocatalytic efficiency is to increase its specific surface area. Except reducing its particle size, creating different morphologies is not bad a choice. So far many morphologies of ZnO, such as ZnO nanowires [11,12], nanobelts [13], nanocombs [14], ZnO porous thin films have been synthesized by thermal evaporation–condensation method or vapor–liquid–solid method [15]. Liu et al. [16–18] prepared ZnO porous thin films by sol–gel method using polyethylene glycol (PEG) template, including nanoparticle films, ordered porous films, nanorod films, nanotube films and porous nanosheet films. However, the low dimensional nanoscaled building blocks have more chances to aggregate in the process of synthesis and photocatalysis, leading to a reduction of its specific surface and photocatalytic efficiency. While three-dimensional

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(3D) hierarchical nanostructure can avoid this problem, and significantly enhance the specific surface area as photocatalysts [1,19–21]. Yan et al. [22] synthesized a hierarchical porous ZnO using a facile hydrothermal method. Lei et al. [23] prepared a hierarchical porous ZnO microspheres with significantly enhanced photocatalytic activity than commercial ZnO and TiO_2 by taking advantage of a hydrothermal reaction of zinc nitrate hexahydrate and urea in the presence of trisodium citrate. Li et al. [21] hydrothermally synthesized 3D hierarchical flowerlike ZnO microspheres using two surfactants, which exhibits high catalytic activity for photocatalytic degradation of Rhodamin B. However, the high cost is a mainly defect of the hydrothermal preparation method.

Besides, it has been reported that photoactive nanostructured semiconductors can be applied in photoelectrochemical (PEC) cells as photoelectrode ever since the PEC splitting of water into hydrogen and oxygen on n-TiO_2 electrodes is reported [24,25]. Novel structured ZnO which could fulfill the demand of high electron mobility (10–100 fold higher than that of TiO_2), high electrochemical stability, and suitable conduction and valence band edges that straddle the redox potentials of water, has increasingly appealed scientists' attention [26–35].

In this work, a novel hierarchical porous ZnO was synthesized by a facile and low cost sol–gel method, in which surfactant Pluronic F127 was used as soft template and a biological material, the Artemia cyst shell as hard template. The pore walls of our as-prepared ZnO had good connectivity, which can provide efficient electrical pathways that ensure charge-transport property [36], and therefore contribute to the efficiency of PEC water splitting.

Experiment

Synthesis of hierarchical porous ZnO

All the reagents were of analytical grade and used without further purification. In an improved sol–gel approach, 1.5 g zinc nitrate hexahydrate ($Zn(NO_3)_2{\cdot}6H_2O$ Tianjin guangfu technology development co., LTD) and 1.5 g urea ($CO(NH_2)_2$ Tianjin kemiou chemical reagent co., LTD) were dissolved in 100 mL deionized water at 50 °C, then the system was kept for 1 h to promote the formation of ZnO nanocrystal precursor. Then 1 g Pluronic F127 (BASF) and 1.5 g Artemia cyst shell (after ball milling) were introduced into the above precursor system. The mixture was drastically stirred at 2250 r/min for 1 h and kept overnight at room temperature to allow the thorough adsorption of zinc oxide colloid particle into the Artemia cyst shell structure, and then vacuum filtered. The as-prepared product was gently heated in an oven at 80 °C overnight. Finally, hierarchical porous ZnO was obtained by calcinating the as-prepared product in a muffle furnace at 400 °C for 18 h in air.

Preparation of hierarchical porous ZnO film

As-prepared hierarchical porous ZnO powders were mixed with ethanol and stirred overnight, resulting in a colloidal suspension with ZnO content of 25 wt%. At last, the suspension was coated onto the indium tin oxide (ITO) glass substrate by scalpel method. After drying in air, the hierarchical porous ZnO film was calcined in air up to 500 °C.

Characterization

Powder X-ray diffraction (XRD) patterns of ZnO samples were recorded on a D-max-2500/PC X-ray diffractometer (Japan, Rigaku Corporation) operating at 40 kV and 100 mA, using Cu Ka (λ = 1.5418 Å) as the source. The morphology of ZnO samples were examined in a Hitachi-S4800 field emission scanning electron microscope (FESEM, Japan) operating at 15 kV and in a Hitachi–7650 transmission electron microscopy (TEM, Japan) operating at 80 kV.

Photocatalytic experiment

High pressure mercury lamp (GYZ 450 TYPE) was adopted as the light source (using UV irradiation wavelength of 365 nm), and methylene blue solution as the degradation target. A batch reactor was adopted in this work. A 250 mL beaker was used as the container, placing in a bigger beaker to kept at room temperature through water bath, during the photocatalytic experiment, the water in the big beaker would be replaced by fresh tap water at the same time of sampling. The above system was set on a magnetic stirring apparatus, and the methylene blue solution was kept stirred at 2250 r/min during the photocatalytic experiments. The initial concentration of methylene blue was 10 mg/L, and the mass concentration of photocatalyst as prepared was 0.3 g/L, after reaction in dark for 30 min, the photocatalytic experiment was carried out. Certain amount of the methylene blue solution was taken out every 10 min as samples, and ZnO particles suspended in the samples was separated via centrifugation. Then the obtained samples were left for UV–Vis measurements. The photocatalytic activity of as-prepared hierarchical porous ZnO was then evaluated according to the concentration changes of methylene blue.

Photocurrent and hydrogen generation test

Photocurrent of the samples was conducted in 1 mol/L Na_2S electrolyte under irradiation of a xenon lamp (100 mW/cm^2) with global AM1.5 condition. The efficiencies of hydrogen generation (η) for the ZnO/$SrTiO_3$ photocatalyst were calculated as below:

$$\eta = \left[I(1.23 - E_{bias})/J_{light}\right] \times 100\% \quad (1)$$

where I is the photocurrent density (mA/cm^2), 1.23 is the theoretical potential required for water splitting, E_{bias} is the applied external potential and J_{light} is the intensity of the solar simulated incident light, which is 100 mW/cm^2 in this study.

Results and discussion

Morphology and structure

The X-ray diffraction (XRD) pattern of the as-synthesized product is shown in Fig. 1. It indicates that the as-prepared

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