Materials Chemistry and Physics xxx (2016) 1–8

Contents lists available at ScienceDirect

Materials Chemistry and Physics

journal homepage: www.elsevier.com/locate/matchemphys

TiO_2 nanotube-carbon macroscopic monoliths with multimodal porosity as efficient recyclable photocatalytic adsorbents for water purification

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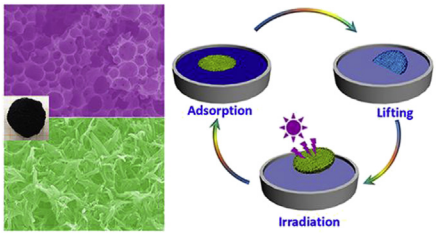
HIGHLIGHTS

- TiO_2 nanotube-carbon macroscopic monoliths with multimodal porosity were prepared.
- The TiO_2-carbon monoliths have adsorption function and photocatalytic function.
- The TiO_2-carbon macroscopic monoliths greatly facilitate organic wastewater purification.

GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:
Received 14 July 2015
Received in revised form
1 February 2016
Accepted 10 February 2016
Available online xxx

Keywords:
Microporous materials
Chemical synthesis
Nanostructures
Oxidation

ABSTRACT

In this paper, we have shown a facile strategy for the construction of porous TiO_2 nanotube-carbon macroscopic monoliths (TNCMs). The resulting macroscopic materials not only possess multimodal porosity, large surface area, and good optical properties, but also combine the advantages of both adsorption materials and photocatalytic materials. Furthermore, the TNCMs can be easily recovered and the robustness of the TNCMs allows them to be reused. Therefore, the as-prepared TNCMs greatly facilitate the purification of organic wastewater, which should have potential for real-world water purification.

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1. Introduction

Due to the rapid population growth and increasing environmental degradation, water crisis is becoming one of the top issues and is probably expected to be the most baffling problem in the future [1–3]. Therefore, increasing attention has been paid to the development of efficient water purification materials. In order to meet the demand of real world water purification, the essential features of advanced water purification materials should be high efficiency, easy recovery, high durability, low cost, and environment beneath [4,5].

To date, various kinds of functional materials have been applied to remove the pollutants from contaminated water, including photocatalytic materials, adsorption materials, and separation materials, etc [6–11]. Among all the functional materials proposed, adsorption materials is one of the most popular candidates, because adsorption is currently considered as an effective, efficient, and easy technique for water purification [9]. However, traditional powder adsorbents need to be dispersed into the polluted water and therefore it is impractical to use them to purify rivers or lakes

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http://dx.doi.org/10.1016/j.matchemphys.2016.02.037
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Please cite this article in press as: Q. Zhang, et al., TiO_2 nanotube-carbon macroscopic monoliths with multimodal porosity as efficient recyclable photocatalytic adsorbents for water purification, Materials Chemistry and Physics (2016), http://dx.doi.org/10.1016/j.matchemphys.2016.02.037

because recovery is complicated and expensive. Furthermore, adsorption process only transfers pollutants from water phase to adsorbent rather than eliminating them from the environment. In addition, the regeneration of exhausted adsorbents by chemical and thermal procedure is required, which is expensive and high-energy consuming [9].

It is widely known that photocatalysts can effectively oxidize a wide range of organic contamination into non-toxic byproducts such as H_2O, CO_2, and mineral acids at ambient conditions [8]. Therefore, it is anticipated that the incorporation of adsorption materials with photocatalytic materials will give rise to a novel photocatalytic adsorbents, which would combine the advantages of both techniques. On one hand, photocatalysts can easily decompose the pollutants anchored on adsorbents under light illumination, therefore the problem about the high cost regeneration of exhausted adsorbents can be effectively addressed; on the other hand, large surface area adsorbents would concentrate the pollutants around the photocatalysts, leading to an enhancement of the photodecomposition rate [12].

Benefiting from their high surface area, porous structure, and special surface reactivity, carbon adsorbents (activated carbon, graphene, carbon nanotubes, carbon nanofibers, etc) have been the most popular candidate and are expected to be widely used in water treatment area [13–17]. Furthermore, comparing with other kind of adsorbents, carbon units can be easily assembled into macroscopic materials, including monoliths, membranes, sponge, aerogel and hydrogel, leading to the formation of easy recovery adsorption materials. TiO_2 is one of the most popular photocatalysts with distinct photochemical activities, which for many years have been intensively exploited for the purification of contaminated water [18,19]. Therefore, combination of TiO_2 with carbon macroscopic materials would give rise to a kind of advanced water purification materials with both adsorption and cleaning functions.

To date, several macroscopic TiO_2-carbon composites have been achieved, where TiO_2 is composed of irregular nanoparticles [20,21]. Compared with nanoparticles, TiO_2 nanotubes possess higher photocatalytic performance because of their superior charge transport properties [22], larger surface area, and multiple pathways for the diffusion of molecules and ions [23]. Furthermore, the pore structure of the reported TiO_2-carbon composites is monotonous. Compared with irregular pore structures, multimodal porous structure permit faster and efficient diffusion of molecules and ions, leading to an obvious enhancement of the photocatalytic activity and adsorption performance [24–27]. Kazuya Nakata and coworkers reported that TiO_2 monoliths with macropores and mesopores possess superior water purification performance [28]. Highly porous TiO_2-based filters and membranes were used to fabricate effective photocatalytic reactor for environmental purification [29,30]. In addition, expensive CNTs or graphene oxide and organic titanium compounds were used as carbon source and titanium source in many previous reported macroscopic TiO_2-carbon structure system, which increase the production cost [21,31]. Therefore, macroscopic TiO_2 nanotube-carbon composites with multimodal pore structures are anticipated to be a kind of advanced water purification materials due to their several obvious advantages. (i) Easy recovery owing to the macroscopic size; (ii) Self-regeneration ability because of the photocatalytic feature of TiO_2 nanotubes; (iii) Outstanding purification performance owing to the multimodal pore structures. However, as far as we know, the construction of multimodal porous TiO_2 nanotube-carbon macroscopic composites is still a big challenge.

In this work, a scalable approach is reported for the construction of multimodal porous TiO_2 nanotube-carbon macroscopic monoliths, in which economic polyurethane (PU) sponge was employed as carbon source, P25 and SiO_2 as pore-forming agents, phenol-formaldehyde resin as adhesive materials to maintain the macroscopic shape. The as-prepared TiO_2 nanotube-carbon macroscopic monoliths (TNCMs) possess both photocatalytic function and adsorption function, which should have potential in water purification area.

2. Experimental

2.1. Materials and methods

PU sponges were commercially available (purchased from Jiaxing Longteng Sponge Co., Ltd.). The other reagents were all purchased from Shanghai Chemical Co. and used as received. The morphology observations of the monoliths and the nanotubes were carried out on a HITACHI S-4800 scanning electron microscope (SEM). Transmission electron microscopy (TEM) measurement was performed using a FEI Tecnai G2 instrument operating at 200 kV. To prepare TEM samples, TiO_2 nanotube was detached from carbon backbone by ultrasonic method, dispersed in ethanol, and deposited onto a carbon-coated copper grid. X-ray diffraction (XRD) patterns of the monoliths was recorded on an X'Pert PRO SUPER rA rotation anode X-ray diffractometer with Ni-filtered Cu-Ka radiation ($\lambda = 1.5418$ Å). Diffuse reflectance spectra (DRS) and UV–vis absorption spectra were recorded on a Cary 5000 UV–vis–near-infrared spectrophotometer fitted with an integrating sphere. The samples for SEM, XRD, and DRS characterizations were the monolith directly.

2.2. Preparation of TNCMs

Before use, PU sponge was cleaned with 1 M NaOH solution and distilled water and was dried in an oven at 90 °C. The clean PU was immersed into mixed solution containing P25, SiO_2, and water soluble phenol-formaldehyde resin in a weight ratio of 4:2:1 for 5 min, and thus the interior of PU was filled by mixed solution. When PU was taken out and kept in hot oven, water would volatilize rapidly and P25, SiO_2, and phenol-formaldehyde resin would be left on the sponge. This wetting-dry process was repeated twenty times in order to increase the loading capacity of P25, SiO_2, and phenol-formaldehyde resin. After that, the sponge coated by P25, SiO_2, and phenol-formaldehyde resin was calcined at 400 °C under nitrogen atmosphere for 2 h and then placed into an autoclave filled with 10 M NaOH solution. After heated at 160 °C for 48 h, the autoclave was cooled down to room temperature and the sponge was taken out, washed alternatively with 0.1 M HCl and distilled water for five times, and calcined at 600 °C under nitrogen atmosphere for 2 h. Consequently, the multimodal porous TNCMs are successfully formed.

2.3. Photocatalytic activity test

A piece of TNCMs (0.5 g) was immersed into 100 mL MB, RhB and MO solution (MB: 10 mg/L; RhB: 10 mg/L; MO: 5 mg/L) under irradiation by a 10 W UV lamp (wavelength: 254 nm, light intensity: 0.08 mW/cm^{-1}). The concentration of the dye solution was monitored by Cary 5000 UV–vis–near-infrared spectrophotometer per 15 min. The absorbance measurement of the sample solution was repeated for three times and no obvious absorbance change was found during the repeated test.

2.4. Adsorption test

A piece of TNCMs (0.5 g) was immersed into 100 mL MB solution with various concentration (8 mg/L; 12 mg/L; 24 mg/L; 60 mg/L) in the dark. The concentration of the dye solution was monitored by Cary 5000 UV–vis–near-infrared spectrophotometer per 2 h. The

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