



In situ fabrication of Bi₂O₂CO₃/MoS₂ on carbon nanofibers for efficient photocatalytic removal of NO under visible-light irradiation



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ARTICLE INFO

Article history:

Received 23 February 2017

Received in revised form 24 April 2017

Accepted 30 May 2017

Available online 3 June 2017

Keywords:

Novel photocatalyst

NO removal

Visible-light irradiation

Recyclable

ABSTRACT

A novel nanocomposite photocatalyst for NO removal, Bi₂O₂CO₃-MoS₂-CNFs, was fabricated by an efficient method. This new photocatalyst performed impressively in the removal of NO at low concentration (600 ppb), with a maximum efficiency of 68% under visible-light irradiation, superior to most other visible-light photocatalysts. Its high performance was ascribed to the introduction of carbon nanofibers as carriers, and MoS₂, which enhanced the absorption of visible light and accelerated the separation and transfer of electrons and holes. Photocurrent tests and electrochemical impedance spectroscopy also demonstrated that Bi₂O₂CO₃-MoS₂-CNFs had a high efficiency of interfacial charge separation, which is critical to improving the photocatalytic activity. Moreover, the membrane of the photocatalyst was stable and recyclable after multiple runs. All of these factors demonstrate its potential application in the removal of NO from air.

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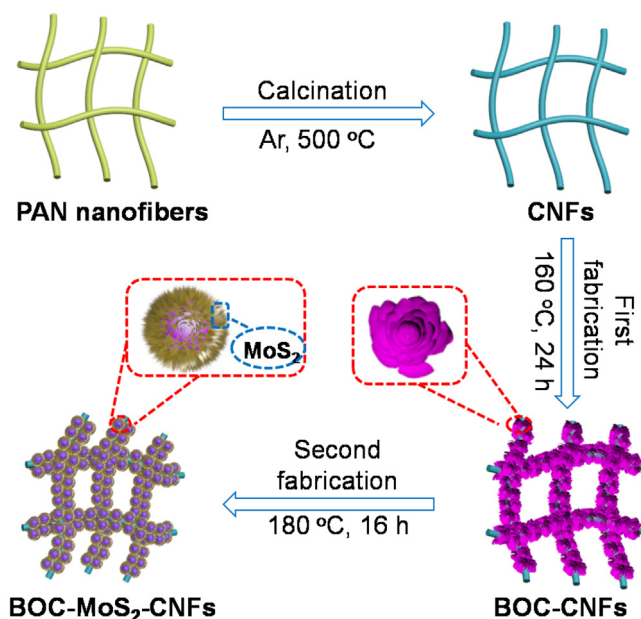
1. Introduction

With the rapid economic and industrial development of China, the number of automobiles in the country is increasing. The growing discharge of automobile exhaust gases into the air is a major cause of environmental pollution. Consequently, the concentration level of PM 2.5 (PM \leq 2.5 μ m in aerodynamic diameter, PM is particulate matter) in major cities is well above the National Ambient Air Quality Standard [1,2]. Recently, China has experienced severe haze events caused by secondary aerosol precursors [3,4]. Nitrogen oxides (NO_x, mainly comprising NO and NO₂) are important contributors to these pollution incidents. Therefore, the development of efficient and economical technologies to address the NO pollution problem is an urgent matter. There are many established techniques and methods for NO purification under ambient conditions, for example physical adsorption [5–7], biofiltration [8–10], thermal catalytic reduction [11,12] and selected catalytic reduction [13–15], but these methods are uneconomical and inefficient for the removal of NO at the parts per billion (ppb) level. However, semiconductor photocatalysis [16,17] is considered a promising technology for NO removal at ppb levels, with potential value in addressing the worldwide energy shortage, because of its high efficiency and eco-friendly characteristics.

Research into photocatalysts for NO removal initially focused on TiO₂ because of its low cost, non-toxicity and high activity, but TiO₂ can only be activated by UV light ($\lambda < 400$ nm), which limits its practical application [18–26]. In subsequent research, several novel photocatalysts, including g-C₃N₄-based materials [27–31], Bi-based materials [32–38], hematite (Fe₂O₃) [39] and Mn⁴⁺-doped anatase [40], have been investigated, and exhibited high performance in the removal of NO at low concentration. Among these, the Bi₂O₂CO₃ photocatalyst [32–35] has attracted particular attention because of its promising applications as a supercapacitor, low mammalian toxicity and good photocatalytic ability. Pure Bi₂O₂CO₃ nanoparticles were shown to remove NO with a percentage removal of 25% [41], while hierarchical Bi₂O₂CO₃ structures achieved a removal of 46% [42], illustrating that the morphology of the photocatalyst plays a significant role in its catalytic effectiveness. However, acceptable results cannot be achieved with the use of a single semiconductor. Therefore, a series of strategies have been developed to improve the photocatalytic efficiency of Bi₂O₂CO₃, including element doping [43], surface modification [44] and the construction of heterojunctions [32,35]. Compared with single semiconductors, heterojunctions inhibit the recombination of photoinduced electron–hole (e[−]–h⁺) pairs and contribute to electron transport, thereby improving the photocatalytic efficiency. Lee and coworkers fabricated a Bi₂O₂CO₃/g-C₃N₄ heterojunction for photocatalytic NO removal, and the NO removal ratio of this heterojunction was 29.4% higher than that of pure Bi₂O₂CO₃ [35]. Zang and coworkers constructed a three-dimensional Z-scheme

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Scheme 1. Schematic illustration of the fabrication of BOC-MoS₂-CNFs.

(BiO)₂CO₃/MoS₂, which dramatically enhanced the photocatalytic NO removal by a factor of 2.2 times compared with pure Bi₂O₂CO₃ under visible light [32], because of the fast separation and transfer of e⁻ and h⁺ enabled by the introduction of MoS₂. However, powder photocatalysts are difficult to recycle because they can be easily blown away during use. Thus, it is important to immobilize the photocatalyst on a suitable carrier.

In this work, Bi₂O₂CO₃-MoS₂-CNFs nanocomposites were fabricated and successfully used to remove NO at the ppb level under visible-light irradiation. Carbon nanofibers (CNFs) were obtained by the calcinations of PAN (polyacrylonitrile) nanofibers [45,46]. Rose-like Bi₂O₂CO₃ microflowers were modified on the surface of the CNFs, and then wrapped by thin MoS₂ nanosheets, as shown in Scheme 1. Here, the CNFs not only acted as a carrier to bind the powder photocatalyst, thus facilitating the transportation of e⁻, but also played the role of an adsorbent for NO. The experimental results showed that the Bi₂O₂CO₃-MoS₂-CNFs photocatalyst achieved an NO removal ratio of 68%, which is superior to most other visible-light photocatalysts. Therefore, the Bi₂O₂CO₃-MoS₂-CNFs membranes have a potential application value in the removal of NO from air in the future.

2. Experimental

2.1. Materials

Polyacrylonitrile (PAN, MW = 150,000), bismuth citrate, sodium molybdate (Na₂MoO₄) were purchased from Sigma Aldrich. Sodium carbonate (Na₂CO₃) were purchased from Adamas. *N,N*-dimethylformamide (DMF), thioacetamide (TAA) and ethylene glycol were purchased from Sinopharm Chemical ReagentCo., Ltd (China) and deionized water was used throughout the experiments. All the chemicals were used without further purification.

2.2. Synthesis of CNFs

In a typical process, CNFs were prepared using an electrospinning machine. 1.0 g of PAN was dissolved into 9.0 mL of DMF in a 20 mL reaction flask, then the mixture under magnetic stirring at room temperature for about 12 h. The above clarified solution

was placed in a 5 mL syringe with a flattened metal needle. The electrospinning was carried out at a positive voltage of 15 kV and negative voltage of -5 kV, the dosing speed was 0.2 mm min⁻¹ and then the PAN nanofibers were obtained. Subsequently, the white polymer nanofibers were heated from room temperature to 500 °C under flowing Ar for 4 h with a ramp rate of 2 °C min⁻¹, then black products were obtained.

2.3. Preparation of Bi₂O₂CO₃-CNFs

Bi₂O₂CO₃-CNFs were prepared by a simple hydrothermal method [32] according to previously described process with some modifications as follows: 230 mg Na₂CO₃ was dissolved in a mixed solution of deionized water (30 mL) and ethylene glycol (6 mL) and stirred for 30 min, then 800 mg bismuth citrate was added into the above suspension and stirred for another 30 min, transparent solution was obtained. Subsequently, the precursor suspension and 50 mg CNFs were transferred into a 50 mL Teflon-lined stainless autoclave and heated at 160 °C for 24 h. After reaction, the reacted mixture was collected with a tweezers and washed with deionized water and ethanol for three times respectively, then dried in an vacuum oven at 60 °C for 6 h. The product Bi₂O₂CO₃-CNFs was obtained and labeled as BOC-CNFs. The pure Bi₂O₂CO₃ microflowers were synthesized under the same conditions without adding CNFs, and labeled as BOC.

2.4. Preparation of Bi₂O₂CO₃-MoS₂-CNFs

Firstly, 24.2 mg Na₂MoO₄ and 19.4 mg thioacetamide were dissolved in 30 mL of deionized water and ultrasound for 30 min, then the mixture and the above BOC-CNFs were transferred to a 50 mL Teflon-lined stainless autoclave and heated at 200 °C for 16 h. Finally, the product was collected and washed with deionized water and ethanol for three times respectively, then dried in an vacuum oven at 60 °C for 6 h. The obtained product Bi₂O₂CO₃-MoS₂-CNFs was labeled as BOC-MoS₂-CNFs. The pure MoS₂ was synthesized under the same conditions without adding BOC-CNFs [47].

2.5. Characterization

Scanning electron microscopy (SEM) (Hitachi S-4800) coupled with X-ray energy dispersive spectroscopy (EDS), and transmission electron microscopy (TEM) (Hitachi H600, 200 kV) were used to measure the morphology, element and crystal lattice of the samples. X-ray diffraction (XRD) (X'Pert-Pro MPD) was used to investigate the crystallographic structure of the products. X-ray photoelectron spectroscopy (XPS) analysis was performed in an X-ray photoelectron spectrometer (ESCALAB MK II) using Al-K α radiation as the exciting source. Using the UV-vis spectrophotometer (CARY50) and fourier transform infrared (FTIR) spectrometer (Nicolet 4700) to measure the light absorbance spectra of samples. The photoluminescence (PL) spectra of the samples were recorded by using a fluorescence spectrophotometer (FLS920) with an excitation wavelength of 370 nm. Electron spin resonance (ESR) spectra of paramagnetic species spin-trapped with 5,5-dimethyl-L-pyrroline-*N*-oxide (DMPO) were recorded with a Bruker A300 EPR spectrometer. Electrochemical measurements were conducted with a CHI 660 B electrochemical system (Shanghai, China) with a standard three-electrode cell, Pt plate was used as the counter electrode and Ag/AgCl electrode was used as the reference electrode, the samples were made as the working electrode, Na₂SO₄ (0.1 M) was used as the electrode solution. 10 mg sample was ground into powder and dispersed in 10 mL of deionized water by sonication to get a slurry. The slurry was spread onto surface of indium-tin oxide (ITO) glass, the working electrode was obtained after the ITO glass was dried at 393 K for 3 h. Visible-light irradiation was provided

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