



Preparation of fiber-based plasmonic photocatalyst and its photocatalytic performance under the visible light



Lin Chen^{a,c}, Sudong Yang^{a,b}, Bin Hao^{a,c}, Jiongming Ruan^d, Peng-Cheng Ma^{a,b,*}

^a Laboratory of Environmental Science and Technology, The Xinjiang Technical Institute of Physics and Chemistry, Chinese Academy of Sciences, Urumqi 830011, China

^b Key Laboratory of Functional Materials and Devices for Special Environments, Chinese Academy of Sciences, Urumqi 830011, China

^c University of Chinese Academy of Science, Beijing 100049, China

^d Department of Thermal Power Technology and Economic Operation, Huadian Electric Power Research Institute, Hangzhou 310030, China

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ABSTRACT

This paper reported the preparation of fiber-based plasmonic photocatalyst consisting of nano-scale Ag-AgBr, sub-micro TiO₂ and micro-scale glass fiber (GF). Various techniques were employed to study the morphology, structure and optical properties of the developed materials. The results showed that Ag-AgBr nanoparticles were highly dispersed on the surface of spherical TiO₂/GF, and the fibers exhibited a unique morphology of helical cracks along the fiber direction. The photocatalytic activities of obtained photocatalyst were evaluated by degrading methyl orange and phenol as model pollutants under the visible-light irradiation ($\lambda \geq 420$ nm). Inspiringly, the material exhibited wide absorption in the visible light and superior visible-light-driven photocatalytic activities compared with other TiO₂-based plasmonic photocatalysts. The improved photocatalytic performance was attributed to the synergistic effects arising from the hetero-junctions in the sample as well as the surface plasmon resonance of Ag nanoparticles. The integration of GF with plasmonic particles endowed the photocatalyst a high stability and reusability under the cyclic operations.

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1. Introduction

Photocatalysis is regarded as a promising technology to solve the problems associated with the air and water pollution due to its strong oxidation capacity and environmental friendliness [1,2]. Since the pioneering work by Fujishima and Honda in 1972, significant progress has been made on the photocatalysis using titanium dioxide (TiO₂) [3]. However, TiO₂, a typical semiconducting material, has a band gap around 3.2 eV, and the activation of its photocatalytic capability requires ultraviolet (UV) light that contributes less than 5% of total energy of solar spectrum [4]. In addition, the low efficiency on separating electron-hole pairs in the material limits the practice of TiO₂ for wide photocatalytic applications [5]. To overcome these problems, many efforts have been devoted to modify the structure and composition of TiO₂ to enhance its visible light utilization, including doping with non-metal materials [6,7], surface sensitization with organic dyes [8],

coupling with narrow band gap semiconductors [9,10], and so on. Among various dopants, noble-metal nanoparticles (such as gold, silver, platinum) attracted much interest in recent years due to their surface plasmon resonance (SPR) phenomenon [11,12]. This unique property not only endows TiO₂ with improved absorption to visible light, but also facilitates the formation of Schottky barrier at the metal-TiO₂ interface, which acts as an electron trapper to suppress the recombination of photogenerated electron-hole pairs [13,14], thus enhancing the overall photocatalytic performance of TiO₂.

Silver halide (AgX, X = Cl, Br, I) is an important photosensitive material, and has been introduced into TiO₂-based photocatalysts for photocatalytic reactions with varying degree of success [15,16]. However, the photodecomposition of pure AgX reduces its activity under the light irradiation. Hybrid structure consisting of Ag nanoparticles and AgX (Ag-AgX) was developed as an alternative to address this issue. In this system, the Ag nanoparticles were generated on the surface of AgX, functioning as a protecting layer to improve the stability of AgX. The strong SPR effect of Ag nanoparticles also contributes greatly on the photoactivity of TiO₂ in the visible light region. For example, Hu et al. [17] prepared Ag-AgBr/TiO₂ using deposition-precipitation method, and the material showed an enhanced photocatalytic activity for the destruction of azo dyes under visible light. In another study, Tian

* Corresponding author at: Laboratory of Environmental Science and Technology, The Xinjiang Technical Institute of Physics and Chemistry, Chinese Academy of Sciences, Urumqi 830011, China. Tel.: +86 991 6992225.

E-mail address: mapc@ms.xjb.ac.cn (P.-C. Ma).

et al. reported [18] the controlled deposition of Ag-AgBr on TiO₂ tubes, and the developed photocatalyst showed strong degradation for phenol under the simulated solar-light irradiation.

Although Ag-AgBr/TiO₂ photocatalysts have been successfully prepared using different techniques, there are still many issues to be considered to optimize their structural, morphological, and photocatalytic properties for specific application. The collection and reuse of TiO₂-based photocatalysts in a slurry system become another major concern for their practical application [19,20]. Anchoring of TiO₂ nanoparticles onto suitable support offers an alternative for this problem. In this context, glass fiber (GF) is a promising candidate due to its characteristics of lightweight, low cost, transparency and high stability against light [21]. The fiber material can be processed into a variety of shapes (yarn, roven, fabrics, etc.), offering an advantage of easy operation when combining with different photocatalytic reactors.

Motivated by the above efforts, this paper reported the preparation of photocatalytic fibers decorated with micro-nano TiO₂ spheres using a solution route. Then the as-prepared TiO₂/GF was used as a support for loading plasmon Ag-AgBr nanocrystals, aiming at enhancing the photocatalytic capability of material using the visible light and stabilizing this property under the cyclic runs. The morphology, surface information and composition of fiber-based plasmonic photocatalyst were studied, and the photocatalytic activity of material was evaluated and compared with other TiO₂-based plasmonic photocatalysts.

2. Experimental

2.1. Raw materials

E-glass fibers with an average diameter of 12 μm were used in this study. The fibers were cleaned using acetone and then dried for application. Titanium tetrachloride (TiCl₄, 98%), silver nitrate (AgNO₃), potassium bromide (KBr), ammonium sulfate ((NH₄)₂SO₄), sulfuric acid (96%, H₂SO₄), ammonia water (28%, NH₃·H₂O), polyvinylpyrrolidone (PVP, K30), ethylene glycol, and methyl orange (MO) were purchased from Tianjing Chemical Company, China. All chemicals were of analytical grade and used without further purification. Ultrapure water (Millipore, resistivity >18.5 MΩ cm) was used in all experiments.

2.2. Material preparation

2.2.1. Synthesis of TiO₂/GF

In a typical experiment, 1.5 g (NH₄)₂SO₄ was dissolved into 100 ml H₂SO₄ (0.01 mol/L). Then 1.0 mL of TiCl₄ was added dropwise into the solution under an ice bath. This was followed by the addition of 0.5 g GF into the mixture and heated at 95 °C for 3 h, yielding TiO₂/GF sample. The product was isolated from the solution by centrifugation and washed with water and ethanol for several times, and then dried in a vacuum oven at 60 °C for 3 h.

2.2.2. Synthesis of Ag-AgBr-TiO₂/GF photocatalyst

The preparation of Ag-AgBr-TiO₂/GF photocatalyst was achieved by mixing solutions consisting of precursors for AgBr and TiO₂/GF. Briefly, 0.12 g PVP and 0.035 g AgNO₃ were dissolved into 40 mL of ethylene glycol, then 0.55 g of TiO₂/GF was added into the mixture to obtain solution A. 0.024 g of KBr was added to another 20 mL of ethylene glycol to obtain solution B. Subsequently, solution B were added into solution A slowly and heated at 60 °C for 1.5 h. In order to avoid the photodecomposition of AgBr, the above experiments were conducted in a dark condition. After the reaction, the obtained product was collected by centrifugation, and washed with water. The sample was re-dispersed into 50 mL of water and irradiated under Xe lamp for 40 min with a change

of solution color from transparency to slight gray, indicating the formation of Ag-AgBr-TiO₂/GF photocatalyst.

2.3. Photocatalytic tests

The photocatalytic activity of sample was evaluated by degrading MO under the visible light using a xenon lamp (300 W, PLS-SXE 300) with a cutoff filter at 420 nm. MO is a stable dye, and its photodegradation has been widely used to evaluate the performance of photocatalysts. A colorless organic dye, phenol (10 mg/L), was employed as another representative pollutant to exclude the contribution of dye-sensitized degradation during the photocatalytic reaction. In the experiment, 50 mg of photocatalyst was suspended in 100 ml of MO solution (10 mg/L) in a quartz reactor, which was equipped with inlet and outlet of water so that the heating effect to the solution due to the light can be minimized during the photocatalytic reaction. Prior to the irradiation, the suspension was stirred in the dark for 30 min to reach the adsorption-desorption equilibrium. Irradiation was located in the center of reactor, and at a given time interval, 3 mL of suspension was collected and the concentration of pollutants was determined by measuring their maximum absorbance (MO: 465 nm; Phenol: 269 nm) using a UV-Vis spectrophotometer (Shimadzu UV-2450). The total organic carbon (TOC), a parameter describing the mineralization of MO in solution, was also monitored using a TOC analyzer (Aoplo 9000).

2.4. Characterization

The crystal structure of samples was characterized on an X-ray diffractometer (XRD, Bruker D8) with Cu K_α radiation (λ = 0.15 nm). The morphology of photocatalyst was observed using a field-emission scanning electron microscope (SEM, Zeiss Supra55VP) and transmission electron microscope (TEM, JEOL JSM-2010). The elemental composition of sample was analyzed using an energy-dispersive X-ray detection system (EDX, Bruker X-Flash-SDD-5010) equipped in SEM. The chemical states of sample were analyzed by an X-ray photoelectron spectroscopy (XPS, PerkinElmer PHI 5000C). Diffuse reflectance spectroscopy (DRS) was performed on a spectrophotometer (SOLID 3700, Shimadzu) over the wavelength range of 300–800 nm. The photoluminescence (PL) characteristics of sample were probed on a fluorescence spectrophotometer (Hitachi F-4500).

3. Results and discussion

3.1. Structure and morphology of photocatalysts

The crystal structures of photocatalysts and corresponding precursors were characterized using XRD. The diffraction peak of pristine GF had a broad peak at 2θ = 22° (Fig. 1A), representing

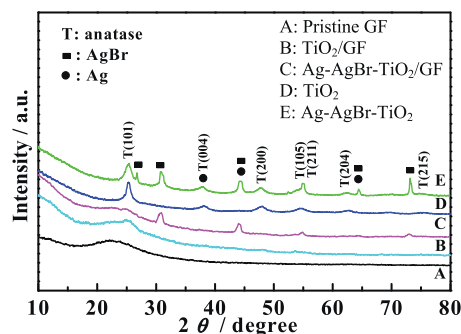


Fig. 1. XRD patterns of photocatalysts and corresponding precursors (A: Pristine GF; B: TiO₂/GF; C: Ag-AgBr-TiO₂/GF; D: TiO₂; E: Ag-AgBr-TiO₂).

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