



Synthesis of recyclable magnetic mesoporous RH-FSBA photoelectrocatalyst with double cavity structure

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ABSTRACT

In this paper, a series of BiOCl-Ag/AgBr heterojunctions supported by magnetic mesoporous silica microspheres with double cavity structure (RH-FSBA_x) were successfully synthesized. For the combination of double cavity structure and orderly mesoporous opening structure of magnetic mesoporous RH-FSBA_x photoelectrocatalysts, they exhibited ultra-strong enriching property to targeted pollutants. For the formation of BiOCl-AgBr heterojunctions, superior photosensitization of AgBr and existence of metallic Ag plasmas with surface plasmon resonance (SPR) effect, the photoelectrocatalysts exhibited enhanced visible light photoelectrocatalytic performance. Furthermore, the photoelectrocatalyst can be recycled easily by magnetic separation for its superior magnetic response. Particularly, the magnetic mesoporous RH-FSBA_{0.03} photoelectrocatalyst exhibited the highest photoelectrocatalytic activity ($k = 1.3 \times 10^{-2} \text{ min}^{-1}$) in degradation of acid fuchsin (AF) under visible light irradiation, and it was about 4 times of RH-FSB photoelectrocatalyst. Therefore, this novel photoelectrocatalyst can effectively decompose organic pollutants in water system under visible light irradiation by rapid enrichment and efficient photodegradation.

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1. Introduction

The development of leather industry brings human lives great convenience for the emergence of numerous new products. However, severe environment problem arises in the course of quick development of leather industry. Tannery wastewater, produced during the leather making process, consists of various pollutants and toxic compounds, such as greases, collagens, plant and animal fibers, organic-inorganic solids, sulfides, hexavalent chromium, salts, surfactants and dyes [1–3]. Considering the low cost and no secondary pollution of photocatalysis technology, it undoubtedly is an effective method to resolve the global environment problems. Up to now, plenty of photocatalysts had been synthesized by researchers [4–8]. However, traditional photocatalysts mainly exhibit

superior photoelectrocatalytic performance under UV light irradiation, and most of them are not sensitive to visible light. As known to all of us, UV light energy only takes up about 4%–6% of solar energy, but visible light energy takes up about 43% of solar energy [9,10]. Furthermore, the photoexcited electron and hole pairs easily recombine after traditional photocatalyst being excited, and it would severely influence its photoelectrocatalytic performance [11,12]. Therefore, design and synthesis of novel photocatalysts with favourable visible light response and fast charge migration ability have attracted many researcher's interests. To improve the performance of traditional photocatalysts, many strategies had been reported. For instance, preparation of heterojunction [13,14], metal/nonmetal doping [15,16], surface modification [17,18], plasmonic effect [19,20], crystal-controlling [21,22], and so on. Therefore, many novel photocatalysts with superior visible light photoactivity had been synthesized, such as metal/multi-metal oxide [23,24], carbonitride [25,26], sulfide [27], quantum dots [28,29] and other metal compounds [30,31]. After reasonable modification, the interaction between different components occurs, and it is possible for the migration of photoexcited carriers in different components, which effectively improves the utilization rate of

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photoexcited carriers in photocatalysis process. Simultaneously, For the formation of heterojunctions, the light response of photocatalysts was enhanced significantly.

To most of photocatalysts, superior visible light photoactivity and high enrichment capacity are hard to be exhibited in a same photocatalyst, thus further study should be carried out. Research shows that irradiated light can be multi-reflected by hollow silica microspheres for the unique inner cavity structure, then the action time of irradiated light would be effectively prolonged, and the temperature of the inner cavity would rise to some extent [32,33]. Furthermore, mesoporous silica exhibits superior material transfer ability for its orderly mesoporous opening structure and high specific surface area [34,35]. More importantly, to reduce the operation cost of catalyst, the recycle ability is also an very important role to be considered [36,37]. Based on above background, the magnetic mesoporous silica microspheres with double cavity structure (RH-FS) were designed and synthesized, then the BiOCl-Ag/AgBr photoelectrocatalyst was assembled on the surface of magnetic RH-FS microspheres, and a series of novel RH-FSBA_x photoelectrocatalysts were obtained. For the combination of double cavity structure and orderly mesoporous opening structure, the photoelectrocatalysts exhibited ultra-strong enriching property to targeted pollutants. For the formation of BiOCl-AgBr heterojunctions, superior photosensitization of AgBr and existence of Ag SPR effect, the photoelectrocatalysts exhibited enhanced visible light photoelectrocatalytic performance. Furthermore, the photoelectrocatalyst can be recycled easily by magnetic separation for its superior magnetic response. Therefore, it has great significance to synthesize the magnetic mesoporous RH-FSBA photoelectrocatalyst with double cavity structure.

2. Experimental section

2.1. Reagents and materials

All chemicals were purchased from Aladdin Chemical and were used without further treatment.

2.2. Synthesis of magnetic RH-FSBA photoelectrocatalyst

The synthesis and amino-functionalization process of magnetic mesoporous RH-FS microspheres with double-cavity structure were shown in Supplementary Material. The BiOCl-Ag/AgBr heterojunctions were grown/assembled on the surface of magnetic mesoporous RH-FS-NH₂ microspheres under the guiding role of the chelation between metal ions and amino groups. 0.5 g RH-FS-NH₂ was ultrasonically dispersed into 140 mL of EG, then 3 g Bi(NO₃)₃·5H₂O and 0.9 g NaCl were added into this mixture under mechanical agitation. 0.01 g AgNO₃ and 0.007 g KBr were added into 100 mL of EG, and this mixed system was stirred thoroughly with glass rod under ultrasonic. Then the homogeneous mixture was added into above dispersion, and the mixed system was constantly stirred for 30 min at room temperature. Subsequently, above mixture was transferred to three teflon-lined stainless steel autoclaves with a capacity of 100 mL, then it was kept for 10 h at 180 °C. After the reaction finished, the product was separated and washed by above method. At last, the sample was dried for 12 h by vacuum freeze-drying technology and 8 h by vacuum oven, respectively. In addition, the samples were also synthesized when the addition of AgNO₃ was 0.02 g, 0.03 g and 0.04 g, and the addition of KBr was 0.014 g, 0.021 g and 0.028 g, respectively. Here, the sample was expressed as RH-FSBA_x (x = 0.01, 0.02, 0.03 and 0.04).

2.3. Characterization

Fourier transform-infrared (FT-IR) spectra of the samples were recorded on a PerkinElmer 580BIR spectrophotometer using the KBr pellet technique. X-ray power diffraction (XRD) analysis was performed on a Bruker AXS D8-advance X-ray diffractometer with Cu K α radiation. The morphologies and sizes of the samples were characterized using the field-emitting scanning electron microscope (FEI, Verios 460, America) and field emission transmission electron microscopy (FEI, Tecnai G2 F20 S-TWIN, America). X-ray photoelectron spectroscopy (XPS) datas were collected to examine the surface chemical states of the samples on an AXIS SUPRA instrument (U.K.). N₂ adsorption/desorption isotherms were obtained on a TriStar II 20 apparatus. The Brunauer-Emmett-Teller (BET) method was used to calculate the specific surface areas based on the adsorption branches. UV–vis diffuse reflectance spectra (DRS) were obtained using a Agilent Technologies Cary Series UV–vis–NIR Spectrometer by using BaSO₄ as background at room temperature. The photoluminescence (PL) spectra were measured on a fluorescence spectrophotometer (EDINBURGH INSTRUMENTS, FS5) using the excitation wavelength of 413 nm. Electrochemical measurements were performed in a electrochemical workstation (PARSTAT 4000).

2.4. Photochemical measurements

The photochemical measurements were finished according to the method in our previous research [38]. The band gaps of the semiconductors were obtained by calculating according to the formula:

$$\alpha h\nu = A(h\nu - E_g)^{n/2} \quad (1)$$

Where h , ν , A and E_g is the Planck constant, frequency of light, absorbance value and band gap, respectively. The parameter n depends on the characteristics of the semiconductor, and $n = 1$ in indirect transition. Thus, E_g of the semiconductors can be estimated from the intercept of the tangent to the xaxis from the plots of $(\alpha h\nu)^{1/2}$ vs $h\nu$.

2.5. Electrochemical measurements

The electrochemical measurements were operated according to the method in other research [39].

2.6. Test of photoelectrocatalytic performance

The AF was chosen as the targeted pollutant, and the photoelectrocatalytic performance of the samples were evaluated under visible-light irradiation. Detailedly, 0.05 g photoelectrocatalyst was ultrasonically dispersed into 100 mL targeted pollutant solution (The concentration of AF solution was 20 mg L⁻¹), then the reaction vessel was placed in dark for 1 h with magnetic stirring to reach the adsorption equilibrium of the targeted pollutant molecules. 3 mL of above mixture was transferred into a sample vial with a capacity of 10 mL and was separated by magnet in dark for the subsequent UV–vis detection. Subsequently, the reaction vessel was illuminated by visible-light source (xenon lamp, 600 W), and 3 mL of reaction solution was transferred into the sample vial in a given time period for the UV–vis detection after magnetic separation. To make sure that the photocatalytic reaction was really driven by visible-light, all the UV lights with the wavelength less than 420 nm were removed by a glass filter (JB-420). After the photoelectrocatalytic reaction finished, the photoelectrocatalyst was

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