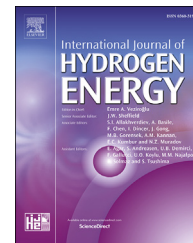




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Meso-g-C₃N₄/g-C₃N₄ nanosheets laminated homojunctions as efficient visible-light-driven photocatalysts

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ABSTRACT

Mesoporous g-C₃N₄/g-C₃N₄ (Meso-g-C₃N₄/g-C₃N₄) nanosheets laminated homojunctions have been fabricated via template-calcination strategy using melamine and amino cyanamide as co-precursors. The prepared Meso-g-C₃N₄/g-C₃N₄ nanosheets laminated homojunctions possess relative high surface area of 34 m² g⁻¹, large pore size of 15.0 nm and narrow band gap of 2.75 eV. The visible-light-driven photocatalytic reaction rate constant of methyl orange and hydrogen production rate (~115.6 μmol h⁻¹ g⁻¹) for Meso-g-C₃N₄/g-C₃N₄ nanosheets laminated homojunctions is about 12.5 and 6.5 times higher than that of the pristine g-C₃N₄, respectively. This may be attributed to the synergetic effect of the close-contact laminated structure contributing to the separation of photogenerated charge carriers and mesoporous structure facilitating the diffusion of reactants and products, and offering more surface active sites. This novel laminated homojunction may open up a new avenue for designing other high-efficient photocatalysts.

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Introduction

Semiconductor photocatalysts have attracted unprecedented attention in mitigate the environmental issues and energy crisis, and mainly concentrated in photocatalytic H₂ evolution and photocatalytic degradation of contaminants [1–3]. As a representative photocatalyst, titanium dioxide (TiO₂) has emerged as one of the most interesting themes because of its

outstanding properties in nature [4–6]. Nevertheless, the relatively wide band gap of ~3.2 eV for anatase TiO₂ seriously restricts its application [7,8].

In 2009, Wang et al. published the significant work on the polymeric graphitic carbon nitride (g-C₃N₄), which could split water into hydrogen [9]. Since then, g-C₃N₄ has drawn ever booming attention as a noble metal-free semiconductor photocatalyst owing to its low toxicity [10], easy availability

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[11], high thermal [12], chemical stability, and benign photocatalytic activity [13,14]. However, its photocatalytic efficiency also faces the invalidation because of the bulk $g\text{-C}_3\text{N}_4$ with low specific surface area ($<10\text{ m}^2\text{ g}^{-1}$), poor quantum efficiency, and easy recombination of the photoinduced charge carriers, which restricts the photoinduced redox reaction [15–17]. Therefore, it is imperative to propose reasonable approaches to facilitate the separation efficiency of photo-generated electron-hole pairs, meanwhile, enhances the performance of photocatalysts.

To date, extensive efforts have been committed to enhance the photoresponse horizon and limited the recombination of photogenerated charge carriers, such as mesostructure, the construction of heterojunction, noble metal deposition, and metal-doped/non-metal-doped semiconductor [18–21]. Among them, the methods, regulating its surface area and constructing the nanojunction structure in the composites, could enhance the photocatalytic activity of $g\text{-C}_3\text{N}_4$ by restricting the photogenerated charge carriers recombination. And it is highly appreciated, such as, fabricating advanced mesostructure and coupling with an array of materials. Wang et al. prepared the mesoporous $g\text{-C}_3\text{N}_4$ by the template method and studied on the photocatalytic hydrogen evolution, resulted in the photocatalytic H_2 evolution is enhanced nearly 10-folds [22]. Besides, in some work, allotypic $g\text{-C}_3\text{N}_4$ -based heterojunction such as $g\text{-C}_3\text{N}_4/\text{graphene}$ [23], $\text{TiO}_2/g\text{-C}_3\text{N}_4$ [24], $\text{MoS}_2/g\text{-C}_3\text{N}_4$ [25], $\text{ZnO}/g\text{-C}_3\text{N}_4$ [26], $\text{WO}_3/g\text{-C}_3\text{N}_4$ [27], $\text{NiMoO}_4/g\text{-C}_3\text{N}_4$ [28], $g\text{-C}_3\text{N}_4/\text{Ag}_2\text{CrO}_4$ [29], and $g\text{-C}_3\text{N}_4/\text{BiVO}_4$ [30], have been actively studied. However, coupling with extra semiconductors will import the metal elements, causing pollution and energy penalty.

Meanwhile, the reported band gap energies of $g\text{-C}_3\text{N}_4$ range from 2.4 to 2.8 eV depending on the different preparation conditions [31–33]. Different co-precursors result in different band structures, which can distinctly facilitate the charge separation and then enhance the photocatalytic activity. Subsequently, $g\text{-C}_3\text{N}_4$ -based homojunctions have been found [34,35]. In consideration of that the band gap energies of $g\text{-C}_3\text{N}_4$ may cause a little bit of mediation using different precursors. Coupling different components of $g\text{-C}_3\text{N}_4$ with well-matched band structure to form a $g\text{-C}_3\text{N}_4/g\text{-C}_3\text{N}_4$ homojunction that exhibited the apparently photocatalytic activity [36]. Mesostructure $g\text{-C}_3\text{N}_4$ coupling with $g\text{-C}_3\text{N}_4$ to form homojunction may be good candidate for high-efficient visible light photocatalyst, in which the mesopores favor the diffusion for reactants and products, and could also provide sufficient surface active sites.

Here, a novel Meso- $g\text{-C}_3\text{N}_4/g\text{-C}_3\text{N}_4$ nanosheets laminated homojunction has been prepared using melamine and amino cyanamide as co-precursors via a facile template-calcination process. The prepared photocatalyst exhibits conspicuous photocatalytic activity for the degradation of methyl orange and hydrogen evolution under visible light illumination. Moreover, a possible photocatalytic mechanism of the Meso- $g\text{-C}_3\text{N}_4/g\text{-C}_3\text{N}_4$ nanosheets laminated homojunction is also proposed. Significantly, the synthetic method will provide an efficient and environmentally friend way for the design of other homojunction structure.

Materials and methods

Materials

The amino cyanide aqueous solution (50 wt%), melamine, absolute ethanol (EtOH), 40% hydrofluoric acid (HF), and the silica colloidal solution (LUDOX HS-40, 40 wt% suspension in H_2O) were all purchased from Sigma-Aldrich. All of the reagents used in the experiments were analytical grade and employed without further purification, and the deionized (DI) water was used throughout this study.

Synthesis of Meso- $g\text{-C}_3\text{N}_4/g\text{-C}_3\text{N}_4$ nanosheets laminated homojunction

The Meso- $g\text{-C}_3\text{N}_4/g\text{-C}_3\text{N}_4$ was prepared based on template-calcination process. Typically, amino cyanide solution was dissolved in a dispersion of SiO_2 nanoparticles of 12 nm in water with vigorous magnetic stirring in a fume hood for 9 h, then 5 g of melamine was put into the semitransparent mixture with magnetic stirring for 24 h. The well-mixed solution was placed into oven dried at $50\text{ }^\circ\text{C}$ until most of the water was evaporated. Then, the resulting white powder was directly heated in a ceramic combustion boat, which was annealed at $550\text{ }^\circ\text{C}$ for 4 h with a ramp rate of $2.3\text{ }^\circ\text{C min}^{-1}$ and tempered for an additional 2.5 h. The resulting light yellow powder was treated with 200 mL of 1 M HF for 24 h, followed by centrifugation at 4000 rpm and washed with DI and ethanol for several times respectively to guarantee the complete removal of silica template. Finally, the resultant powders were dried at $80\text{ }^\circ\text{C}$ overnight. The ultimate yellow powder was denoted as Meso- $g\text{-C}_3\text{N}_4/g\text{-C}_3\text{N}_4$ (the mass ratio of Meso- $g\text{-C}_3\text{N}_4$ and $g\text{-C}_3\text{N}_4$ is 2:1). For comparison, the Meso- $g\text{-C}_3\text{N}_4$ was also synthesized under the same conditions without mixing melamine. In addition, the bulk $g\text{-C}_3\text{N}_4$ has been prepared by heating melamine from the room temperature to $550\text{ }^\circ\text{C}$ with a ramp rate of $2.3\text{ }^\circ\text{C min}^{-1}$ for 4 h in a muffle furnace, and then was kept at $550\text{ }^\circ\text{C}$ for 2.5 h (see Scheme 1).

Characterization

The crystallinity of the Meso- $g\text{-C}_3\text{N}_4/g\text{-C}_3\text{N}_4$ was characterized by X-ray Diffraction (XRD) Patterns with a Bruker D8 advance under $\text{Cu K}\alpha$ radiation ($\lambda = 1.5406\text{ \AA}$). Fourier transformed infrared (FT-IR) spectra were recorded on a PerkinElmer spectrum one system using KBr as diluents. Scanning electron microscopy (SEM) was performed with a Philips XL-30-ESEM-FEG operated at an accelerating voltage of 20 kV. The structures and morphologies of all samples were characterized by transmission electron microscopy (JEM-2100 TEM). X-ray photoelectron spectroscopy (XPS) was measured by a PHI-5700 ESCA system. The specific surface area and pore size distribution of all samples were performed on Brunauer-Emmett-Teller (BET) method on nitrogen adsorption apparatus with an AUTOSORB-1 instrument. UV-vis diffuse reflectance spectroscopy (UV-DRS) was recorded with a UV-2550, Shimadzu UV-vis spectrophotometer, in which BaSO_4 was employed as the background. (The wavelength of UV lamp

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