



Invited feature article

Enhanced visible-light driven photocatalytic activity of hybrid ZnO/g-C₃N₄ by high performance ball millingQin Chen¹, Huijie Hou¹, Danwei Zhang, Shaogang Hu, Tao Min, Bingchuan Liu, Changzhu Yang, Wenhong Pu, Jingping Hu*, Jiakuan Yang*

School of Environmental Science and Engineering, Huazhong University of Science and Technology (HUST), Wuhan, 430074, PR China

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ABSTRACT

The ZnO/g-C₃N₄ photocatalyst was prepared by ball milling and demonstrated enhanced photocatalytic degradation of RhB under visible light irradiation. The photocatalyst is consisted of ZnO and g-C₃N₄ heterojunctions formed by the shear stress during the mechanical treatment. The addition of g-C₃N₄ can improve the photocatalytic performance of ZnO under visible light. The heterojunction formed between ZnO and g-C₃N₄ decreased the recombination rate of photogenerated carriers and improved the photocatalytic activity of the ZnO/g-C₃N₄ hybrids. The optimal ZnO/g-C₃N₄ photocatalyst with 10 wt% g-C₃N₄ showed a degradation efficiency of 51.3% for RhB under visible light, which is 2.1 times higher than that of pristine ZnO. The electron paramagnetic resonance (EPR) measurement showed that the •OH radicals are the dominant active species in photocatalytic process under both full solar spectrum and visible light irradiation. And the •OH radical generation is much more efficient under full solar spectrum than that under visible light due to the UV response of ZnO. This work provides a simple method for the design and preparation of novel heterojunction photocatalysts.

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1. Introduction

Semiconductor photocatalysis which utilizes renewable solar energy to initiate oxidation and reduction processes to drive highly efficient chemical reactions has become a promising strategy for industrial wastewater treatment, especially streams containing residual recalcitrant components that are not suitable for conventional wastewater treatment plant based on microbiology process. Photocatalysts including titanium oxide, zinc oxide and tungsten trioxide, etc. have been extensively studied in environmental waste treatment system owing to their less secondary pollution, high degradation efficiency and wide range of application possibilities [1–3]. Among them, ZnO is one of the most promising candidates because of its strong oxidation capability, non-toxicity, ready availability and good biocompatibility [4,5]. However, the wide band gap of 3.2–3.7 eV limited its response to the visible light region. In addition to the limited response spectrum, the long term stability was also restricted by the dissolution of active photocatalyst induced by photocorrosion [6–

8]. Efforts have been devoted to enhancing the photo-response of ZnO under visible light and reduce the recombination of photo-generated carriers by means of doping [9], coupling with other semiconductors [10] and deposition of metals [11]. It has been reported that doping with nonmetallic elements can reduce the band gap of metal oxides [12], and coupling with other semiconductors will form a heterojunction structure which is also beneficial for the separation of photo-generated carriers [13].

Graphite-like carbon nitride (g-C₃N₄) is a metal-free polymeric, stable, non-toxic and easily prepared semiconductor photocatalyst with a band gap of 2.7 eV, which is photo-responsive under visible light irradiation (~460 nm) [14]. However, the high recombination rate of the photo-generated carriers greatly hinders its practical applications [15]. Coupling semiconductors with different band structures, such as g-C₃N₄ with ZnO, could enhance the electron-hole charge separation and increase the charge carrier lifetime, which consequently enhances the photodegradation efficiency [16]. Strategies of the synthesis of ZnO composite will generate significant impact on the photocatalyst structure, mechanical and optical properties, luminescence and lattice dynamics properties, thereby influencing the photocatalytic efficiency [17]. The hybrid structure of ZnO and g-C₃N₄ prepared by different strategies including monolayer dispersion, calcination and

* Corresponding authors.

E-mail addresses: hujp@hust.edu.cn (J. Hu), jkyang@hust.edu.cn (J. Yang).¹ These two authors contributed equally to this paper.

mechanochemistry have been reported [18–20]. Zhu et al. [19] found that the nanocomposite photocatalysts of $g\text{-C}_3\text{N}_4/\text{ZnO}$ prepared by calcination of evaporation-dried mixture of dicyandiamide and zinc nitrate showed higher photocatalytic activities than pristine ZnO under visible light. The $g\text{-C}_3\text{N}_4/\text{ZnO}$ composite exhibits sponge-like porous structure decorated with nanoparticles. Wang et al. [6] reported a hybrid $g\text{-C}_3\text{N}_4/\text{ZnO}$ structure prepared by a monolayer-dispersed method. The photocatalytic activity of $g\text{-C}_3\text{N}_4/\text{ZnO}$ composite under UV irradiation was increased by 3.5 times than that of pristine ZnO. Ball milling process utilizing high energy collision from the balls has shown great advantages in material synthesis by initiating or changing chemical reaction conditions, and inducing phase transformation of target materials [21–23]. $\text{C}_3\text{N}_4/\text{TiO}_2$ hybrid photocatalysts synthesized from the facile ball milling method promoted the dispersion of a layered structure of $g\text{-C}_3\text{N}_4$ on the surface of TiO_2 particles, forming a single layer hybrid structure and a multi-layer core-shell structure [20]. Zhang et al. [24] prepared a direct Z-scheme photocatalyst $\text{Bi}_2\text{O}_3/g\text{-C}_3\text{N}_4$ by ball milling and heat treatment methods, and the interface between $g\text{-C}_3\text{N}_4$ and Bi_2O_3 was found to be favorable for the transport of photo-excited carriers. Considering that the application of ball milling on the preparation of $\text{ZnO}/g\text{-C}_3\text{N}_4$ hybrids has been rarely studied, the unique structure manipulation technique would provide a novel feasible strategy for the fabrication of $\text{ZnO}/g\text{-C}_3\text{N}_4$ hybrids to enhance the separation of photo-excited carrier as well as visible light response.

In this work, the $\text{ZnO}/g\text{-C}_3\text{N}_4$ hybrid photocatalyst was prepared by high performance ball milling with the use of commercially

available nano-sized zinc oxide and urea. Composites with different mass ratios of $g\text{-C}_3\text{N}_4$ to ZnO were synthesized and the photocatalytic efficiency was studied by using Rhodamine B (RhB) as the target contaminant under visible light irradiation. The mechanisms of the enhanced photocatalytic performance of the hybrid structure were also proposed.

2. Material and methods

2.1. Chemicals

Nano-sized zinc oxide (ZnO) was purchased from Chemical Reagent Research Institute Co., Ltd (Tianjin, China). Urea (CON_2H_4), isopropanol ($\text{C}_3\text{H}_8\text{O}$), triethanolamine ($\text{C}_6\text{H}_{15}\text{NO}_3$) and Rhodamine B ($\text{C}_{28}\text{H}_{31}\text{ClN}_2\text{O}_3$) were purchased from Sinopharm Chemical Reagent Co. Ltd (Shanghai, China). Dimethyl pyridine N-oxide (DMPO) was purchased from Aladdin Industrial Corporation (Shanghai, China). All chemicals were used without further purification in this research. Deionized water with a resistivity $\geq 18 \text{ M}\Omega \text{ cm}^{-1}$ was used throughout the experiments.

2.2. Synthesis of the hybrid structure

Graphitic carbon nitride ($g\text{-C}_3\text{N}_4$) in yellow bulk form was synthesized by pyrolyzing urea in a muffle furnace at 550°C for 2 h with a heating rate of 5°C min^{-1} [15]. Then it was manually grinded in an agate mortar to obtain the $g\text{-C}_3\text{N}_4$ powder in light yellow color for further use. Zinc oxide (ZnO) of 1.0 g was then mixed with 0.05 g, 0.10 g, 0.15 g, and 0.20 g of $g\text{-C}_3\text{N}_4$ respectively to

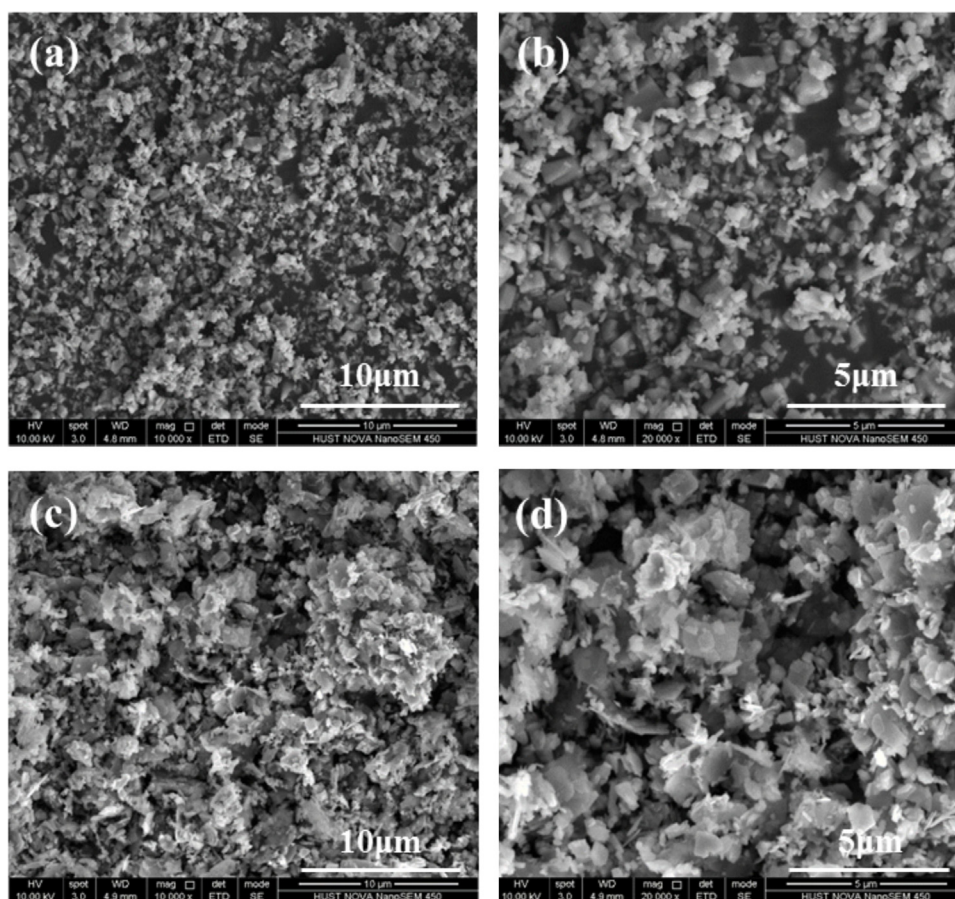


Fig. 1. SEM images of (a, b) pristine ZnO, (c, d) ZnO-C10.

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