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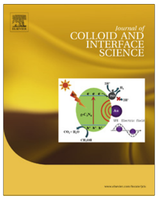

Metal-free hybrids of graphitic carbon nitride and nanodiamonds for photoelectrochemical and photocatalytic applications

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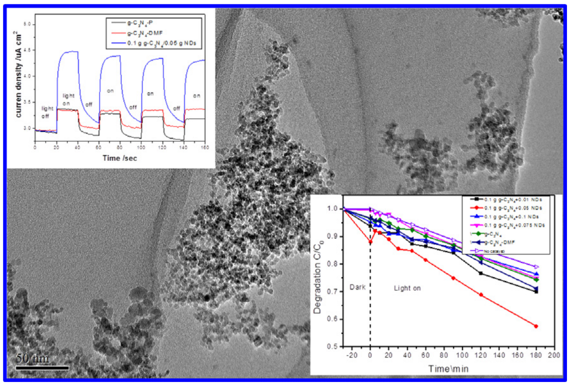
HIGHLIGHTS

- Metal-free hybrids of graphitic carbon nitride and nanodiamonds were synthesized.
- The absorption, separation and transportation rate of carriers were improved.
- Enhanced photocurrent and photocatalysis were obtained on the hybrid photocatalysts.

GRAPHICAL ABSTRACT



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ABSTRACT

Graphitic carbon nitride (g-C_3N_4) has been considered as a metal-free, cost-effective, eco-friendly and efficient catalyst for various photoelectrochemical applications. However, compared to conventional metal-based photocatalysts, its photocatalytic activity is still low because of the low mobility of carriers restricted by the polymer nature. Herein, a series of hybrids of g-C_3N_4 (GCN) and nanodiamonds (NDs) were synthesized using a solvothermal method. The photoelectrochemical performance and photocatalytic efficiency of the GCN/NDs were investigated by means of the generation of photocurrent and photodegradation of methylene blue (MB) solutions under UV–visible light irradiations. In this study, the sample of GCN/ND-33% derived from 0.1 g GCN and 0.05 g NDs displayed the highest photocatalytic activity and the strongest photocurrent density. The mechanism of enhanced photoelectrochemical and photocatalytic performances was also discussed.

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1. Introduction

In modern society, energy crisis and environmental pollution that caused by the rapid industrialization and civilization have attracted worldwide concerns [1,2]. A variety of strategies have been employed to explore sustainable energy resources and to develop sustainable remediation technologies. Photocatalysis, as a green technology, has demonstrated great potentials for both renewable energy and water purification because it is cost-effective, clean and sustainable [3–6]. The application of metal-based photocatalysts has resulted in secondary contamination from metal leaching into water [7–9]. More recently, the breakthrough of development in metal-free photocatalysts has been promising to overcome the drawback of metal leaching [10,11].

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Graphitic carbon nitride (g-C_3N_4), a typical green, metal-free, polymeric photocatalyst, has been extensively investigated because of its unique properties and versatile applications [6,12–14]. It has been well established that g-C_3N_4 can be widely employed for hydrogen production, CO_2 reduction and degradation of organic pollutants [5,15–17]. However, the photocatalytic efficiency of g-C_3N_4 still remains at a low level because of the low specific surface e area (SSA, usually below 10 $m^2\ g^{-1}$), high recombination rate, slow charge mobility and relatively short absorption range [12,18]. Therefore, great efforts have been made to modify g-C_3N_4 to enhance the photocatalytic and photoelectrochemical activity [19–21]. It was found that metal-based hybrids of g-C_3N_4 could significantly improve the photocatalytic efficiency of g-C_3N_4, while coupling with other metal-free species/compounds attracts more attention because the metal-free nature can be still remained [3,11,22].

Apart from the employed metal-free heteroatoms [23,24], the hybrids of g-C_3N_4 and nanocarbons, such as graphene oxide [25,26], graphene [27], carbon nanospheres [3,28], carbon nanotubes [29], melem [30] and carbon quantum dots [31] were fabricated to achieve enhanced performances. In recent years, nanodiamonds (NDs), as non-toxic and carbonaceous materials, have attracted more scientific interests because they have large surface areas, biocompatibility, unique optical and chemical properties [32]. NDs also have a promising perspective in the field of biomedicines [33]. For better performances, NDs have been modified by the fabrication of hybrids, for example, nanodiamonds-TiO_2 [34–36] and nanodiamonds-graphene oxide [37]. Jang et al. [38] reported that a composite of nanodiamonds-rGO had a high and stable photoelectrochemical activity under visible light irradiations. Wang et al. [39] also reported that surface-tailored NDs had an excellent catalytic activity for degradation of organic pollutants in wastewater.

In this study, melamine was used to synthesize pristine g-C_3N_4 because it is cheap, non-toxic, eco-friendly and has a high productivity of pristine g-C_3N_4. Commercial detonation nanodiamonds were employed to fabricate the hybrids. For the first time, the novel hybrid photocatalysts, g-C_3N_4/nanodiamonds (GCN/ND), were prepared by a solvothermal route. The photoelectrochemical activity of the hybrids was evaluated in an electrochemical cell under irradiations. The photocatalytic efficiency was investigated by the photodegradation of aqueous MB solutions under UV–vis irradiations.

2. Experimental section

2.1. Materials and chemicals

Nanodiamonds (with a particle size below 10 nm) and melamine (99.9%) were purchased from Sigma-Aldrich. Methylene blue (99.9%) and N, N-dimethylformamide (DMF) were obtained from Sigma. Ethanol (99.9%) was received from Chem Supply. All the chemicals and materials were used as received without any further purification.

2.2. Synthesis of g-C_3N_4

Graphitic carbon nitride was prepared by a thermal condensation method using melamine as the precursor. In a typical run, 5 g melamine was put into a crucible with a loose cover and then heated at 550 °C for 2 h in a muffle furnace with a heating rate of 10 °C/min [3]. When the temperature was cooled down to room temperature, the solid pristine g-C_3N_4 was obtained and then grinded into powder for further use.

2.3. Synthesis of GCN/ND

For the synthesis of the hybrids, 0.1 g g-C_3N_4 and 0.05 g NDs were mixed into 80 mL N, N-dimethylformamide (DMF). The mixed solution was kept magnetically stirring for 30 min and then underwent ultrasonication for 30 min to ensure that g-C_3N_4 and NDs were dispersed homogeneously. After that, the mixture was transferred into a stainless steel autoclave and heated in an oven at 150 °C for 24 h. When the autoclave was cooled down to room temperature, the mixture was separated by a centrifuge at 7500 rpm for 20 min. Then the solid was washed with ethanol and pure water each for twice. After washed thoroughly, it was dried in an oven at 60 °C for over 24 h to obtain the hybrid photocatalyst of GCN/ND-33%. By the same procedure, GCN-DMF and the composites of GCN/ND-9%, GCN/ND-43% and GCN/ND-50% were also prepared.

2.4. Characterization of the materials

Powder X-ray diffraction (XRD) was used to analyze the crystalline structure of the samples on a Germany Bruker D8-X-ray diffractometer with Cu Kα radiation (λ = 1.5418 Å). The XRD results were obtained from 2 theta range of 10–70°. A Micromeritics 3000 was employed to evaluate the specific surface area (SSA) and the pore size distribution by liquid nitrogen sorption at −196 °C. The thermal analysis of these photocatalysts was performed on a Mettler-Toledo-Star equipment under an air flow with a heating rate of 10 °C/min. The morphology of the samples was investigated by a scanning electron microscopy (SEM). Transmission electron microscopy (TEM) images were received on a JEOL 2100 TEM microscope. A JASCO V670 UV–vis spectrophotometer was used to record the UV–visible diffuse spectra of these samples, and $BaSO_4$ was used as the reference material. A Varian Eclipse spectrometer (wavelength = 300 nm) was employed to obtain the photoluminescence (PL) spectra. X-ray photoelectron spectroscopy (XPS) survey was applied to investigate the chemical compositions and states of the hybrids.

2.5. Photoelectrochemical and photocatalytic performances

2.5.1. Photoelectrochemical performance tests

Firstly, 8 mg GCN/ND powders were mixed with 50 μL Nafion and 500 μL ethanol in a vial. Then the vial was ultrasonicated for 20 min to enable the samples to be dispersed thoroughly. After that, the mixed paste was smeared on a 1 cm^2 square FTO glass and then the smeared glass was dried for a few minutes in air. The dried glass was used as the working electrode. The photoelectrochemical activity of the sample was investigated on an electrochemical workstation (Zahner Zennium) and 0 V voltage was applied. A solar simulator (TriSOL, OAI) provided the light irradiations and a three-electrode photoelectrochemical cell was employed, including a counter electrode (a platinum wire), a reference electrode (Ag/AgCl) and a working electrode (the prepared smeared FTO glasses). Na_2SO_4 solution (0.2 mol/L) was used as the electrolyte. The light irradiation was switched on and off in each 20 s respectively during the measurements.

2.5.2. Photodegradation of MB solutions

The photodegradation efficiency of GCN/ND was tested in degradation of MB solutions under UV–vis light irradiations. A 300 W Newport Oriel Universal Xenon arc lamp was used as the light source. In details, 50 mg photocatalyst was added into the 200 mL MB solution. A two-jacket cylindrical reactor and a water bath were used to control the reaction temperature at 25 °C. A magnetic stirrer was used to ensure the photocatalyst dispersed homogeneously during the whole process of reaction. Prior to the

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