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Invited feature article

One-pot synthesis of magnetic graphitic carbon nitride photocatalyst with synergistic catalytic performance under visible-light irradiation

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ABSTRACT

In this study, a magnetically separable g-C_3N_4-Fe_3O_4 composite with the controllable Fe_3O_4 component was successfully synthesized via a one-step facile and economical hydrothermal method. The results showed strong interaction existed between Fe_3O_4 nanoparticles and g-C_3N_4 sheets, meanwhile, the prepared composites exhibited superior photocatalytic activity, and high magnetic response, which had a significant effect on recovery and reuse of g-C_3N_4-Fe_3O_4 composites. More importantly, the photocatalysis material could degrade various dyes efficiently under visible-light irradiation and still exhibited an excellent photocatalytic performance after five cycles. Therefore, the g-C_3N_4-Fe_3O_4 composites synthesized by the simple synthesis process, exhibited high magnetic response and excellent photocatalytic performance, and can show broad application prospects in the field of photodegradation.
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1. Introduction

In the past 20 years, environmental pollution has received a great deal of attention because there was significantly increasing trend of pollutant emissions to river and sea with the development of global economy. Due to prominent stability, low-cost, and easy separation, semiconductor photocatalysis has emerged as a most promising technology for the degradation of the organic contaminants in wastewater [1]. Recently, many researchers have focused on the research of an efficient semiconductor material with outstanding properties to photodegradation [2–4]. Nanostructured carbon nitride was found to be an intriguing semiconductor material, possessing superior properties comprising high hardness, cracking chemical stability, and good thermal tolerance. Consequently, carbon nitride shows a tremendous potential for energy conversion and environmental renovation [5–10].

Several phases of carbon nitride theoretically exist, containing α, β, cubic, pseudo-cubic, graphitic structure [11]. Among these phases, graphitic carbon nitride (g-C_3N_4) has a layer structure analogous to graphite and is regarded as the most stable one, i.e. it couldn't decompose thermally in air till 600 °C [12,13]. In addition, g-C_3N_4 has many advantages, including easy preparation, nontoxicity, chemical resistance and sensitive visible light catalytic performance [14,15]. However, there are also some defects for g-C_3N_4 materials, such as (a) low specific surface area, (b) large band gap, weak visible light response and (c) difficult collection [16]. Fortunately, the above drawbacks of g-C_3N_4 would be overcome readily through suitable adulteration, or chemical functionalization, according to the recent literatures. For instance, doped with some nonmetals like B, C, and S, or narrow band-gap semiconductors, absorption range of g-C_3N_4 could be extended significantly [17–20]. When g-C_3N_4 is combined with other semiconductors with narrow band gap, such as Bi_2WO_6/g-C_3N_4 [16], CdS/g-C_3N_4 [21] and CuO_x/g-C_3N_4 [22], heterojunction structures would be induced to dramatically enhance separation efficiency of photo-generated electron–hole pairs, migration rate of surface reactants, and resulting photocatalytic activities under visible light irradiation [23–27]. On the other side, Fe_3O_4 nanoparticles have become an active research area on account of their intrinsic magnetic properties and high conductivity (1.9×10^6 S m^{-1}) [28,29], which have facilitated their applications in lithium-ion battery [30], drug delivery [31], low-field magnetic separation [32], magnetic resonance imaging [33], and wastewater treatment [34]. Heterostructured catalysts consisting of Fe_3O_4 nanoparticles have exhibited improved properties due to high conductivity of Fe_3O_4 [35] and energy band structure matching (E_{CB} = 1.23 V vs NHE) [36].

According to the published literatures, g-C_3N_4-Fe_3O_4 composites were usually synthesized by in-situ growth method [37]. However, the Fe_3O_4 loaded on the g-C_3N_4 sheets reunited seriously, which couldn't effectively hinder the recombination of electron–

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hole pairs and limit the photocatalytic activity of g-C_3N_4-Fe_3O_4 composites. Herein, we have proposed a one-step facile and economical hydrothermal method for preparing magnetic g-C_3N_4-Fe_3O_4 composites with different Fe_3O_4 content. The photocatalytic performance was evaluated through the experiment of degradation of different dyes including methylene blue (MB), methyl orange, (MO) and rhodamine B (RhB) in the aqueous solution under visible light irradiation [38]. The results proved that Fe_3O_4 nanoparticles were successfully deposited onto the surface of g-C_3N_4-Fe_3O_4, and photocatalytic activity was dramatically enhanced. A possible mechanism of enhanced photocatalytic performance was also investigated. Furthermore, coupling Fe_3O_4 with g-C_3N_4 turned out to be a convenient manner to reclaim the as-obtained photocatalysts from the photocatalytic system using an external magnetic field, enabling reuse in manifold cycles. The proposed approach greatly restrained the gathering of Fe_3O_4 nanoparticles during preparation and recovery.

2. Experimental section

2.1. Materials

Melamine ($C_3N_3(NH_2)_3$) was purchased from Shanghai Lingfeng Chemical Reagent Co., Ltd., Shanghai, China. Ferric chloride hexahydrate ($FeCl_3{\cdot}6H_2O$), sodium acetate (NaAc), and 1,6-diaminohexane ($C_6H_{16}N_2$) were bought from Sinopharm Chemical Reagent Co. Absolute ethylene glycol ($(CH_2OH)_2$) and ethanol (C_2H_5OH) were purchased from Nanjing Chemical Reagent Co., Ltd., Nanjing, China. All chemicals used in this work were analytically pure and used without further treatment. Deionized water was used as detergent.

2.2. Preparation of g-C_3N_4 sheets

The g-C_3N_4 was synthesized via a facile method that melamine was just heated to 550 °C directly for 4 h in a box type furnace under air atmosphere.

2.3. Fabrication of g-C_3N_4-Fe_3O_4 magnetic composites

We prepared g-C_3N_4-Fe_3O_4 composites by a one-step solvent-thermal method, and the schematic was exhibited in Fig. 1. First, 5 g of 1, 6-diaminohexane, 1 g $FeCl_3{\cdot}6H_2O$, and 2 g NaAc were dissolved in 30 mL EG by ultrasound under 50 °C. Then, a certain amount of g-C_3N_4 was added to the above solution and had ultrasonic for 1 h to form a uniform suspension. After that, the resulting suspension was transferred to a Teflon-lined stainless-steel autoclave (50 mL) and heated to 190 °C for 6 h. The as-prepared g-C_3N_4-Fe_3O_4 was washed several times with deionized water and absolute alcohol after it was cooled to room temperature, and collected by means of external magnetism. Finally, the as-synthesized precipitation was dried in vacuum oven at 30 °C for further characterization. For comparison, sole Fe_3O_4 nanoparticles were synthesized by the same process without adding g-C_3N_4. The obtained g-C_3N_4-Fe_3O_4 photocatalysts with Fe_3O_4 mass percent of 10.0%, 19.7%, and 30.8% were entitled CNFO-10.0, CNFO-19.7, CNFO-30.8, respectively. Similarly, the sole g-C_3N_4 was entitled CN, and Fe_3O_4 was entitled FO.

2.4. Characterization

Power X-ray diffraction (XRD) measurement was carried out on a Bruker D8 Advanced diffractometer with Cu Kα radiation (λ = 0.154 nm, 40 kV) at 10–80°. The morphology of the products aforementioned was observed via scanning electron microscopy

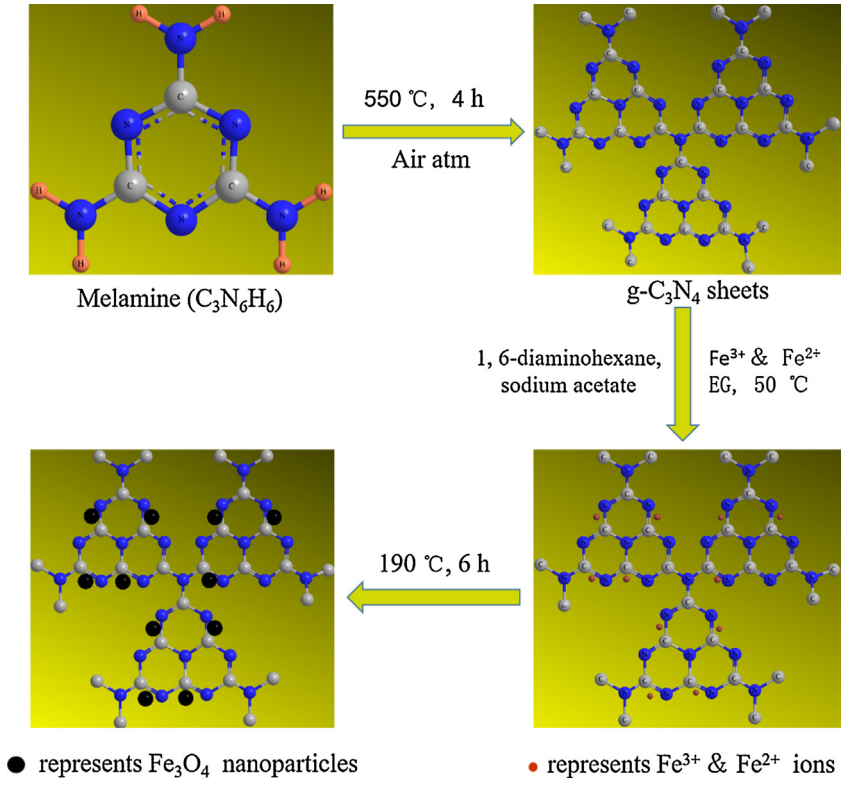

Fig. 1. Schematic illustration of the synthesis of g-C_3N_4-Fe_3O_4 photocatalysts.

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