



Research article

An investigation of the biochar-based visible-light photocatalyst via a self-assembly strategy

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ABSTRACT

In order to reduce the cost of commercial visible-light photocatalyst, a self-assembly strategy was deployed in producing a Ti-coupled N-embedded chicken feather biochar based catalyst (TINCs). The TINCs were manufactured by blending Ti-contained cross-link agent with hydrolyzed N-embedded chicken feather. These synthesis materials were well characterized with X-ray diffraction, Fourier transform infrared, Scanning electron microscopy, Transmission electron microscope, Energy dispersive X-ray spectroscopy, X-ray photoelectron spectroscopy, Raman spectroscopy, Surface area analysis and UV–vis absorption spectra. There were multilayered graphene oxide-like structures observed on TINCs, which were similar to the TiO₂-graphene oxide material. Correspondingly, the TINCs had presented a 90.91% degradation rate of Rhodamine B under visible-light after 240 min. The corresponding TOC of the solution had dropped by 56.26%. Every slice of TINCs was constituted by multilayered graphene oxide-like framework, interspersing with TiO₂ nanoparticles uniformly. Some mechanisms were also analyzed. The cost analysis investigated that TINCs was promising in industrialization.

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1. Introduction

Recently, the photocatalytic degradation became more and more popular in environmental engineering because of its tolerance, efficiency and convenience (Sharma and Lee, 2016a,b; Vaiano et al., 2015). However, the energy consumption with ultraviolet (UV) light source had restricted its application. So far, various visible-light photocatalysts had been developed by scientists, such as nitrogen doped TiO₂ particles (N-TiO₂) and TiO₂-doped graphene oxide (TiO₂-GO) (Cong et al., 2013; Wang et al., 2013a; Su et al., 2016). The nitrogen atoms were demonstrated helpful in band gap narrowing in composites. Thus, the optical response regions of nitrogen doped materials were expanded to visible-light (Sharma and Lee, 2016a,b; Vaiano et al., 2015). Besides, the development of TiO₂-GO was also popular nowadays (Min et al., 2012). Compared with other traditional photocatalysts, TiO₂-GO was more recyclable and efficient because of its peculiar electronic property, agile structure, and large specific surface area (Sharma and Lee, 2016a,b). With an excellent hole conductivity, the p-type

graphene oxide (GO) had improved the separation of photo electrons generated by TiO₂-GO during photocatalytic reaction. The degradation efficiencies were also higher than previous studies (Su et al., 2016; Min et al., 2012; Liu et al., 2017). Though the remarkable visible-light responses had been presented by these synthetic photocatalysts, there were still some restrictions before a commercial popularization. Firstly, the nanoparticles were easily reunited with each other, which could lead to a catalytic deactivation (Sharma and Lee, 2016a,b). Secondly, some of the precursors were too expensive (especially the GO), hazardous (nitric acid), or even toxic (ammonia) to be ignored in the actual practices (Wu et al., 2015). Besides, the high energy consumption (ultrahigh-temperature sintering or pyrolysis treatment over 1000 °C) and contamination conditions (acid fog) during the synthetic procedure were also unexpected (Liu et al., 2017; Wu et al., 2015).

As the essential factors affecting actual applications, the preparation method and precursors for catalyst should be considered. A simple and environmental friendly synthesis attempt is necessary in scale-up industrial application. This method should establish on basis of minimizing the steps of preparation, disusing either harmful reagents or expensive precursors and particularly avoiding toxic byproducts (Shiraishi et al., 2014; Chen et al., 2018). In this way, the

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traditional biochar based functional materials were noticed (Reddy, 2015; Reguyal et al., 2017). Some nitrogen contained environmental friendly raw materials should be considered by replacing the expensive graphene oxide and dangerous nitric acid.

Consisted with keratin microfibers, the chicken feathers (CFs) were so stable and hardly decomposed that regarded as solid waste, they were usually treated by burning open the air (Reddy, 2015; Wang et al., 2013b). However, this bio-resource material was potential in formulating the nitrogen contained, graphene oxide like biochar (Souza et al., 2017). The framework of feather was revealed by previous studies. A miracle “multilayer beta sheets” structures of micro folded polypeptides were observed in the feather keratin. Overlapped with each other, the “beta sheets” were linked by disulfide bonds and van der Waals forces, which could be easily destroyed in high temperatures (Senoz et al., 2012; Mena et al., 2017; Srinivasan and Sarmah, 2015). There were carboxyl, hydroxyl and amidogen functional groups detected on the surfaces of “beta sheets”. All of the groups were much more easily crossed with each other at high temperatures (200 °C) (Tuna et al., 2015). In addition, the cross-link process could easily influenced by cross-link agent. The tetrabutyl titanate, a kind of Ti precursor, was also a kind of wildly used cross-link agent in practice. Thus, it is potential to introduce the tetrabutyl titanate into the recombination system of keratin rebuilding (Senoz et al., 2012). This self-assembly method, with pure raw material without any hazardous agents or additives, was environmental friendly and safety.

In this way, we introduced the cross-link technology in and created a simple green cross-link hydrothermal carbonizing method to achieve a Ti-coupled N-embedded CFs biochar based visible-light photocatalyst (TINCs), which with a resemble interface of TiO₂-GO. The visible-light photocatalysts were characterized by X-ray diffraction (XRD), Fourier transform infrared (FT-IR), Scanning electron microscopy (SEM), Transmission electron microscope (TEM), Energy dispersive X-ray Spectroscopy (EDX), Brunauer-Emmett-Teller (BET) analysis and UV–vis absorption spectra. In addition, The X-ray photoelectron spectra (XPS) of chicken feather carbon (CFC) and TINCs were swept. Besides, the visible-light-catalytic activities were well investigated by the simulated Rhodamine B (RhB) wastewater.

2. Materials and methods

2.1. Chemicals and materials

Rhodamine B, P25, KOH, HCl, absolute ethyl alcohol, and tetrabutyl titanate (Ti precursor) used in this study are all in analytical grade. Waste CFs were gathered from the Double Tree Poultry Market in Hohhot, China. Grease was washed out with surfactant and deionized water.

2.2. Catalyst preparation

TINCs were prepared according to the hydrothermal cross-link

and chicken feather carbonized method (Tuna et al., 2015). The cleaned CFs were dried at 60 °C in oven for 24 h at atmosphere. Initially, about 8 g CFs were smashed into about 1 inch and loaded in six Teflon lined hydrothermal autoclave reactor (75 mL). Meanwhile, buffer solutions were made up by dissolving KOH in absolute ethyl alcohol (0.2 g/L). Thereafter, each reactor was filled with certain amount of Ti precursor. Afterwards, 20 mL of absolute ethyl alcohol or buffer solution was added into these reactors. These reactors were settled and heated in the air dry ovens at 200 °C and/or 220 °C for 8 h, respectively, followed by a carbonization process in argon atmosphere at 450 °C for 1 h. Thus, the composites were triturated to powder with agate mortar and washed with deionized water and alcohol alternately twice. Qualitative filter paper was used to separate the TINCs from the solutions. After a 12 h drying at 60 °C in oven, the final products were achieved. The synthesis conditions and appearances of TINCs were listed in Table 1.

2.3. Photocatalytic experiments

Rhodamine B degradation experiments were deployed to examine the photocatalytic activities of TINCs particles under visible-light irradiation. Simulated solar reactor was taken with a 500 W xenon lamp. In addition, a 400 nm cut-off long-pass filter was used to delete ultraviolet light. The powders and liquor were loaded with quartz tubes in size of $\Phi 12 \text{ mm} \times 140 \text{ mm}$ (over 50 mL). Each tube could be placed uniformly revolving around the lamp. The photocatalytic experiments were carried out with 0.05 g of TINCs sample powder in 50 mL of RhB solution (initial concentration of 20 mg/L) (Li et al., 2013). The temperature was kept at about 20 °C by recirculating cooling system when light was on. A magnetic stirrer was used in each tube at the stirring speed of 700 rpm. The xenon lamp would not turn on until an adsorption-desorption equilibrium was reached after 1 h. During the experiment, each 5 mL suspensions was withdrawn and filtered in time intervals of 1 h. Changes in total organic carbon (TOC), total inorganic carbon (TIC) and total carbon (TC) were measured and recorded by each hour, too. As the same process as the photocatalytic experiments were deployed, three parallel experiments were taken.

2.4. Characteristic and analytical methods

The surface morphologies and elemental mapping of TINCs were tested by field emission scanning electron microscopy (SEM, HITACHIS-4800). TEM and EDX were recorded by FEI Tecnai G² F20 S-TWIN. XRD patterns were detected on a Panalytical Empyrean with Cu K α radiation ($\lambda = 1.78897 \text{ \AA}$) at 40 kV and 40 mA. FT-IR analyses were conducted on a Nicolet Avatar 670 FI-IR spectrometer (Nicolet Corp., USA). The absorption peaks were distributed from 4000 to 400 cm⁻¹ with multiple scans and 4.0 cm⁻¹ resolution. The Brunauer-Emmett-Teller (BET) specific surface areas were obtained from N₂ adsorption at 77 K using Micromeritics ASAP 2020 surface area analyzer (Autosorb-1C-TCD physical adsorption

Table 1
The list of samples names and their raw material proportions with different conditions.

| Samples Names | Raw materials | | | | Temperature of crosslink and carbonization | Physic Characteristic | | Visible-light Photo-catalytic ability |
|-------------------|-----------------|---------------------------------|---------|---------|--|-----------------------|------------|---------------------------------------|
| | Chicken feather | Ti precursor (cross-link agent) | KOH | Alcohol | | Color | Appearance | |
| TINC-S | ~8 g | 8 mL | – | 20 mL | 200 °C/450 °C | bright black | sand-like | negative |
| TINC-N | ~8 g | 8 mL | – | 20 mL | 220 °C/450 °C | dark black | powder | negative |
| TINC ₁ | ~8 g | 4 mL | 0.004 g | 20 mL | 220 °C/450 °C | grey black | powder | positive |
| TINC ₂ | ~8 g | 8 mL | 0.004 g | 20 mL | 220 °C/450 °C | grey black | powder | positive |
| TINC ₃ | ~8 g | 12 mL | 0.004 g | 20 mL | 220 °C/450 °C | grey black | powder | negative |
| CFC | ~8 g | – | 0.004 g | 20 mL | 220 °C/450 °C | dark black | particle | negative |

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