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Short communication

Synthesis of phosphonic acid silver-graphene oxide nanomaterials with photocatalytic activity through ultrasonic-assisted method



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ABSTRACT

In this article, phosphonic acid silver-graphene oxide nanomaterials (Nano-PAS-GO) was synthesized from silver nitrate (AgNO₃) solution and phosphoric graphene oxide (PGO) via the convenient ultrasonic-assisted method, and the structure and morphology were characterized, and the photocatalytic activity and recyclability were evaluated through photocatalyzing degradation of Rhodamin B (RhB) aqueous solution, and the possible photocatalytic mechanism was also discussed. Based on those, it was confirmed that Nano-PAS-GO has been synthesized from AgNO₃ solution and PGO colloidal suspension under ultrasonic-assisted condition, and Nano-PAS-GO has consisted of phosphoric acid silver nanoparticles and GO with 2D lattice (2D GO lattice) connected in the form of C-P bonds, and the photodegradation rate of Nano-PAS-GO for RhB aqueous solution has reached 93.99%, and Nano-PAS-GO has possessed the nicer recyclability when the photocatalytic time was 50 min. From those results, the strong and stable interface between PAS nanoparticles and 2D GO lattice connected in the form of the covalent bonds has effectively inhibited the occurrence of the photocorrosion phenomenon.

1. Introduction

Today, the development of visible-light-responsible photocatalysts with high efficiency and strong stability has attracted considerable attention because they can be potentially applied in hydrogen evolution from water splitting, degradation of contaminants and disinfection [1,2]. Among the many photocatalysts that have been reported, silver orthophosphate (Ag₃PO₄) has been a representative photocatalyst with the high photooxidative capability for O₂ production from water under the visible-light irradiation [3] and the high and efficiency decomposition for organic dyes [4]. However, it is worth noticing that Ag₃PO₄ has also been a photocatalyst with the prone photocorrosion in the absence of sacrificial electron acceptors in the photocatalytic process. Such as, the lattice Ag^+ in Ag_3PO_4 is reduced to metallic Ag by the photo-generated electrons $(4Ag_3PO_4 + 6H_2O + 12h^+ + 12e^- \rightarrow$ $12Ag + 4H_3PO_4 + 3O_2$ [3]. So how to improve the robustness of Ag₃PO₄ in the photocatalytic process needs further to explore. In order to solve the problem of photocorrosion of Ag₃PO₄, some Ag₃PO₄-based composites including Ag/Ag₃PO₄ [5], AgX/Ag₃PO₄ (X = Cl, Br, I) [6], TiO₂/Ag₃PO₄ [7], carbon quantum dots (CQDs)/Ag₃PO₄ [8], graphene or graphene oxide (GO)/Ag₃PO₄ [9,10] and so forth have been prepared, which slow down the recombination of photo-generated

electron-hole pairs to enhanced the photocatalysis performance and stability of Ag₃PO₄.

Graphene as one of carbon's allotropes has attracted a great deal of attention resulting from the single-layer graphene possesses the special two-dimensional (2D) structure, extraordinary conductivity, high surface area and strong chemical stability [11-14]. These unique properties make graphene or functionalization graphene including GO to become an ideal precursor for preparation of Ag₃PO₄-based composites. For instance, GO has been used as the precursor in the preparations of GO enwrapped Ag₃PO₄ composite by an ion-exchange method [15], GO/Ag₃PO₄ composite through a liquid phase deposition method [16] and Ag₃PO₄/graphene composite by combination of the electrostatically self-assembly and the hydrothermal methods [17]. Throughout the performance of the photocatalysts and the preparation methods of them, it is found that the photocatalysts have the better stability and the high photocatalysis activity for organic dyes, but the preparation procedures and the reaction time in the preparation methods are relatively complex and long. In order to decrease the complex procedures and to shorten the reaction time, it is very necessary to explore a new method for preparation of Ag₃PO₄ of graphene or GO as matrix materials.

In recent years, sonochemical method has been widely utilized in

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the preparations of graphene and graphene functionalization [18–22], semiconductor photocatalyst, semiconductor photocatalyst composite [23,24], metal oxide nanoparticles [25] and so on [26]. The effect of sonochemistry derives from the ultrasonic cavitation [27,28]. The babbles generated by ultrasonic cavitation can release the enormous energy and generate a strong impact of the micro jet, and they in the dramatic collapse can suddenly and locally generate the high temperature and the high pressure. Based on the effect of sonochemistry, Ag₃PO₄/rectorite composite photocatalyst with the highly efficient and stable properties has been prepared by the ultrasound-assisted precipitation method [24]. Nevertheless, the preparation of Nano-PAS-GO in the form of the covalent bonds linkage between PAS nanoparticles and 2D GO lattice has been rarely reported in the case of ultrasound irradiation.

In this work, the visible-light responsive Nano-PAS-GO was first prepared by $AgNO_3$ solution and PGO colloidal suspension in the case of ultrasound irradiation, and the structure of PGO was also confirmed by SEM, TME, SAED, FTIR, XPS, Raman and TGA, and the structure of Nano-PAS-GO was characterized by XRD, FTIR, and SEM as well. Otherwise, the photocatalytic activity and reusability of Nano-PAS-GO for the degradation of RhB aqueous solution were also evaluated in the case of the visible-light irradiation.

2. Experimental

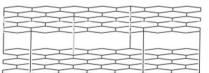
2.1. Materials

Flake graphite (the average flake size = $160 \,\mu$ m mesh and the carbon content = $90.00 \,wt\%$) was purchased from Man Country in China. Nitric acid ($65.00 \,wt\%$), peroxyacetic acid ($35.00 \,wt\%$), acetic anhydride ($85.00 \,wt\%$), phosphoric acid ($85.00 \,wt\%$), silver nitrate were purchased from Beijing Huagong Company, Beijing, China.

2.2. Synthesis of Nano-PAS-GO

2.2.1. Preparation of 2D PGO lattice

In accordance with our previous work [29] and Scheme 1, flake graphite, nitric acid and peroxyacetic acid (flake graphite: nitric acid: peroxyacetic acid = 1.00: 1.50: 1.00) were mixed into a mixture in a flask. And then, the flask was fixed in the ultrasonic cleaning bath (Nominal power: 250 W). After ultrasound irradiation for 35 min, the flask was taken out from the ultrasonic cleaning bath and the liquid was removed from the flask, and the solid was transferred to a porcelain evaporating dish. Later, the solid in the porcelain evaporating dish was placed in a drying box and dried at 100 °C. After 30 min of drying, the porcelain evaporating dish was taken out, and the solid was removed from the porcelain evaporating dish and transferred to the flask. Then, A certain amount of phosphoric acid (flake graphite: phosphoric acid = 10.00: 1.00) and acetic anhydride (flake graphite: acetic anhydride = 1.00: 1.00) were added in the flask that had been fixed in the ultrasonic cleaning bath. After ultrasound irradiation for 25 min, the solid and liquid were separated. The solid was transferred to the



Flake graphite

porcelain evaporating dish and dried at 100 °C. After the solid was completely dry, the porcelain evaporating dish was transferred to the drying box to heat at 170 °C. After 20 min of heating, PGO could be prepared, which was known as 2D PGO lattice in our laboratory.

In order to further increase the content of phosphoric acid in PGO, a certain amount of phosphoric acid (PGO: phosphoric acid = 10.00: 3.00) was again added in the flask. Then, 2D PGO lattice that contained phosphoric acid could be prepared in accordance with the previous procedures.

2.2.2. Synthesis of Nano-PAS-GO

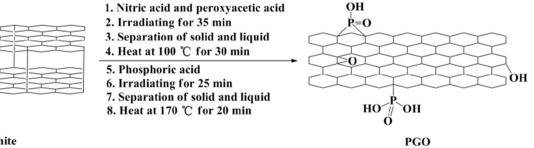
According to Scheme 2., 50.00 mg of PGO and 40.00 mL of distilled water were added in 250 mL of conical flask that had been fixed in the ultrasonic cleaning bath. After ultrasound irradiation for 1 h, PGO colloidal suspension was produced in the conical flask. And then, 0.30 g of AgNO₃ solution that prepared from 10.00 mL of distilled water under the ultrasonic-assisted condition was slowly dripped into the flask by using of a 10 mL of plastic injector in the case of ultrasound irradiation. In about 15 min, AgNO₃ solution was completely dripped into the conical flask. At that moment, the mains switch of ultrasonic cleaning bath was stopped. The solid in the conical flask was transferred to a glass petri dish. After 3 h drying in a vacuum oven at 50 °C, Nano-PAS-GO could be synthesized.

In addition, Ag₃PO₄/GO composite has been also prepared. The detailed procedures are shown in the Supplementary materials. Otherwise, the morphology and structure of Ag₃PO₄/GO composite have been also characterized. XRD patterns of GO and Ag₃PO₄/GO composite are shown in Fig. S1, and SEM micrograph of Ag₃PO₄/GO composite is shown in Fig. S2. To compare XRD pattern and SEM micrograph of Ag₃PO₄/GO composite with the ones of Nano-PAS-GO, it has been confirmed that the crystal structure of them is the same, but the morphology of them is different. Also, the photocatalytic activity of Ag₃PO₄/GO composite has been also investigated. The adsorption rate and the photodegradation rate of Ag₃PO₄/GO composite for RhB aqueous solution are shown in Fig. S3. To compare the adsorption rate and the photodegradation rate of Ag₃PO₄/GO composite with the ones of Nano-PAS-GO, it has been confirmed that they are different. Based on the confirmations, it is believed that the high temperature and high pressure generated by the ultrasonic cavitation and the conventional agitation can be all used to prepare the photocatalyst containing the body-centered cubic structure of Ag₃PO₄, but the morphology and structure and the adsorption rate and photodegradation rate for RhB aqueous solution are different.

2.3. Characterization

2.3.1. Characterization of 2D PGO lattice

Scanning electron microscopy (JEM-2100F, Japan) was used to obtain SEM micrographs of GO and PGO. The corresponding energydispersive X-ray spectroscopy was used to EDS spectrum of PGO. Thereinto, SEM micrographs of GO and PGO were shown in Fig. 1(a) and (c), and EDS spectrum was shown in the Supplementary materials.



Scheme 1. Preparation of 2D PGO lattice under ultrasonic-assisted condition.

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