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Rapid microwave-assisted synthesis of silver decorated-reduced graphene oxide nanoparticles with enhanced photocatalytic activity under visible light



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ABSTRACT

A facile, fast, and scalable microwave irradiation (MWI) method for the synthesis of Ag nanoparticles (Ag NPs) dispersed on graphene sheets has been developed. The reduction of graphene oxide takes place in ethanol solution within 2 min of MWI without any additional reducing agent or complicated treatment. The morphology and microstructure of the as-prepared hybrid were characterized by Scanning electron microscopy (SEM) and Transmission electron microscopy (TEM) images. The result showed the Ag NPs with an average size of 5–10 nm decorated on the rGO sheets. X-ray powder diffraction (XRD) determined that the crystallographic structure of Ag is face-centered cubic and there was a strong interaction between Ag NPs and rGO sheets. Fourier transform infrared spectroscopy (FTIR) and X-ray photoelectron spectroscopy (XPS) showed that GO had been reduced to rGO in our hybrid. Moreover, visible photocatalytic activity of the rGO–Ag nanocomposites was tested using Rhodamine B (RhB) as the model contaminant. This result indicates that rGO–Ag nanocomposites display distinctly enhanced photocatalytic activities. The investigation gave a promise to the development of original yet highly efficient graphene-based photocatalysts that utilize visible light as an energy source.

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1. Introduction

A great challenge for this century lies in cleaning up the waste generated during industrial, agricultural, and domestic activities. Artificial photocatalytic systems are highly desirable because of abundant sunlight resource and less of carbon emission. Various ultraviolet light catalysts are reported to remove dye pollutants [1,2]. However, the ratio of ultraviolet light (~3%) is much lower than visible-light (~50%) in natural light. Therefore, visible-light catalyst is much more suitable for practical application [3–8]. Plasmon-induced Ag@AgX (Cl, Br, I) photocatalyst has been synthesized by direct precipitation or ion-exchange reaction

followed by reduction [9,10]. Nevertheless, the use of these photocatalysts is limited due to their low electron transport efficiency.

Graphene, a two-dimensional nanometer-thick highly conducting and thermally stable material, has sparked great excitement in the fields of chemistry, physics, and materials sciences and continues to attract extensive interest for fundamental scientific inquiry and for the prospects of potential applications and new advanced technologies [11–16]. The combination of graphene with the size-tunable properties of metal and semiconductor nanocrystals offers many interesting applications in a wide range of fields including heterogeneous catalysis, nanoelectronics, and devices [17–20]. Recently, Zhu et al. reported the synthesis of plasmon-induced graphene oxide (GO)/Ag@AgX (Cl, Br, I) photocatalyst via a water/oil system [21]. However, the use of highly toxic and dangerously unstable

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chloroform is needed to synthesize GO/Ag@AgX (Cl, Br, I) requires great care. In addition, Wen et al. reported the solvothermal synthesis of graphene/Ag/TiO₂/nanocomposites [22]. These photocatalytic results indicated that the incorporation of graphene oxide efficiently enhanced the photocatalytic activity of Ag@AgX and Ag/TiO₂ nanoparticles. Their nice response to visible-light could be attributed to the existence of metallic Ag nanoparticles, which gives rise to distinct plasmon resonance in the visible region. However, in these cases, tedious synthesis processes and toxic reactants are necessary. Consequently, a more facile and green method is strongly desired.

Nowadays, microwave irradiation (MWI) as highly effective technology has been widely used in the synthesis of nanoparticles because it has a more homogeneous heating process and can speed up the reaction rate by orders of magnitude compared with conventional heating [23,24]. In addition, MWI has been utilized to obtain graphene from GO, indicating its well reducing action; it also allows a variety of metal salts, thus resulting in the synthesis of metallic and bimetallic nanoparticles supported on the graphene sheets [25,26].

In this study, we propose a facile and efficient microwave-assisted strategy to synthesize reduced graphene oxide–silver (rGO–Ag) hybrid materials in ethanol solution without any additional reducing agent or complicated treatment. Photodegradation of Rhodamine B (RhB) shows that the as prepared composites in this method have potential ability for environmental purification.

2. Experimental procedure

All of the chemical reagents were of analytic-grade and used without further purification (purchased from Shanghai Chemical Reagent Co., Ltd., Shanghai, China).

2.1. Synthesis of GO and microwave synthesis of rGO–Ag hybrid materials

Graphene oxide was prepared from natural flake graphite powder by a modified Hummers' method [27]. As a typical procedure to synthesize the rGO–Ag nanocomposite, AgNO₃ (2 mmol) and GO (20 mg) were dispersed into absolute ethanol (100 mL), respectively, with ultrasonication for 1 h. Then the two mixtures were mixed and magnetically stirred for 6 h. Subsequently, the slurry was placed in a microwave oven under cyclic microwave radiation (30 s on for every 30 s interval) for several cycles at 800 W. Noted that cyclic microwave radiation was employed instead of continuous to avoid bumping. The resulted suspension was centrifuged and washed copiously with ethanol three times to remove any impurities. The final product was dried at 60 °C in vacuum for 24 h to dry out and labeled as rGO–Ag.

2.2. Photocatalytic experiment

The visible light catalytic activity of rGO–Ag nanocomposites was estimated by degradation of RhB. 25 mg catalyst was suspended in 50 mL RhB solution with a concentration of 4 mg/L. The suspension was firstly stirred

in dark for 30 min to ensure that the surface of catalyst was saturated with RhB. Then the suspension was exposed to a 250 W high-voltage mercury lamp for different time spans, namely 0–60 min with an interval of 10 min. After that the catalyst was centrifuged and monitored by measuring the absorbance of the solution samples with a UV–vis spectrophotometer (Optizen POP) at $\lambda_{\max}=552$ nm.

2.3. Characterization and analysis

The products were characterized by X-ray powder diffraction (XRD) measurements, which were performed on a Bruker D8 with Cu K α radiation at 40 kV and 40 mA. The scan rate was 0.02°/s. Microstructures of the products were obtained by Field emission scanning electron microscopy (FE-SEM, S4800, Hitachi Ltd., Japan) and Transmission electron microscopy (TEM, JEM-2100, JEOL Ltd., Japan). Fourier transform infrared spectroscopy (FTIR) patterns were measured on a Nicolet IR200 FTIR spectrophotometer (Thermo Electron Ltd., USA). X-ray photoelectron spectroscopy (XPS) measurements were performed using a PHI-5702 spectrometer (Physical Electronics Ltd., USA) with an Al K X-ray source (1486.6 eV). UV–vis spectroscopy measurements were performed on a UV-3600 UV–vis spectrophotometer (Shimadzu Ltd., Japan) in water dispersion.

3. Results and discussion

3.1. Structure and properties of rGO–Ag hybrid materials

The morphologies of pristine GO and the as-prepared rGO–Ag nanocomposites have been characterized by TEM as shown in Fig. 1. Fig. 1(a) clearly demonstrates the gauze-like morphology characterization of pure GO. As seen in Fig. 1(b) for rGO–Ag nanocomposite, Ag nanoparticles (Ag NPs) with sizes of several to tens of nanometers were found uniformly deposited on the surface of GO sheets. As described in the experimental section, Ag ions were introduced via the strong cooperation with the terminal oxygen-containing functional groups on the GO nanosheets [28]. During the microwave treatment, the hydrolysis of Ag ions occurs through deoxidization as well as the reduction of GO [29,30]. As a result, Ag NPs were attached onto the surface of GO. The existence of Ag NPs was further evidenced by XRD.

XRD is an effective method to investigate the phase and structure of the synthesized materials. Fig. 2 shows the XRD pattern of GO and rGO–Ag nanocomposite. For GO, as shown in Fig. 2(a), the most striking peak (observed at $2\theta=12.29^\circ$), corresponding to a d_{002} basal spacing of approximately 0.702 nm. This indicates that the oxidation of the pristine natural graphite and most oxygen is bonded to the planar surface of graphite after the oxidization [31]. In the case of rGO–Ag composite, as shown in Fig. 2(b), the peaks at 2θ values of 38.18° , 44.34° , and 64.60° correspond to the (111), (200), and (220) crystal planes of the face-centered cubic Ag (JCPDS-040783). No obvious diffraction peaks of GO were observed in the rGO–Ag nanocomposite, suggesting that GO was reduced to graphene and rGO–Ag

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