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A facile route to strontium titanate nanocubes/reduced graphene oxide nanocomposites and their enhanced adsorption and photocatalytic activity

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ABSTRACT

Novel strontium titanate nanocubes/reduced graphene oxide (STO/rGO) nanocomposites were successfully synthesized via a two-step route, including a rapid sol-precipitation process and a subsequent hydrothermal process. TEM images indicate that the nanocomposites are composed of rGO-supported monodispersed 5–10 nm STO nanocubes. Compared with pure STO, STO/rGO nanocomposites exhibit better adsorption and photocatalytic performance. Particularly, the sample with 6 wt% rGO exhibits the best enhanced photocatalytic performance. The photocatalysis measurements indicate that the rGO sheets not only exhibit superior adsorptive property but also behave as the photocatalysis supporter due to the synergistic effect of the accelerated separation of photogenerated electron-hole pairs. The novel synthesis route provides possibility to design perovskite/rGO nano-photocatalysts.

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1. Introduction

Because of their fundamental interest, as well as potential applications in nanostructure-based photonics and electronics, considerable attention has been focused on the syntheses and properties of semiconductor nanostructures [1–12]. As a new generation of sewage treatment technology, photocatalysis shows excellent performance, which mainly benefited from the active photogenerated electron-hole pairs generated in valence band (VB) and conduction band (CB). As a kind of special bimetallic oxides, perovskite oxides with a typical structure ABO₃ have been widely used in solar cells, solid oxide fuel cells, photovoltaic materials, superconductivity and thermoelectric materials [13–16]. Therein, strontium titanate (STO) has attracted great attention due to its outstanding electrochemistry performance, good conductivity and remarkable stability [17–21]. However, there are few researches on STO used as photocatalyst for two major reasons: (i) Quite small surface area results in limited photocatalytic performance; (ii) poor adsorption which does not make good use of photocatalytic reaction sites. On the other hand, various carbonaceous materials such as graphene and carbon nanotubes have been extensively studied for wastewater treatment due to the π - π stacking interactions between carbon aromatic domains and organic dye molecules. It is well-known that graphene as a supporter

* Corresponding authors. E-mail addresses: slin@bupt.edu.cn (S. Lin), mlei@bupt.edu.cn (M. Lei). can drastically enhance the reaction rate of photocatalysis, owing to the synergistic effect between semiconductor photocatalyst and graphene sheets [22–27]. Therefore, developing novel STO-graphene nanocomposites for efficient photocatalysis is promising and meaningful.

In this work, novel SrTiO₃/reduced graphene oxide (STO/rGO) nanocomposites were successfully synthesized in two steps: (i) a rapid sol-precipitation process; (ii) a subsequent hydrothermal process. In order to measure the photocatalytic performance of the as-prepared samples, Rh B was used to mimic the waste water conditions. As a result, STO/rGO nanocomposites show both enhanced adsorption and photocatalytic activity, which mainly benefit from the synergistic effect between STO and rGO sheets. A controllable shape, a facile synthesis method as well as the advanced graphene supporter contributes to the realization of STO/ rGO nanocomposites implemented to solve water pollution.

2. Experimental

2.1. Synthesis of STO/rGO nanocomposites

STO nanocubes were synthesized from tetrabutyl titanate (Ti $(OBu)_4$) in 99% triethylene glycol (TEG) solvent. Firstly, 16 mmol Sr $(OH)_2 \cdot 8H_2O$, 16 mmol Ti $(OBu)_4$, 8 mL 25% NH₄OH and 0.74 g PVP (K30) were dispersed in 40 mL TEG solvents. Then, the mixture was maintained at 160 °C for 2 h with vigorous stirring. After natural cooling, a yellow transparent hyperstable sol was formed







and further precipitated by deionized water. After separation, the as-prepared STO nanocubes were rinsed with deionized water and alcohol, and fully dried in vacuum at 60 °C for 12 h. Graphene oxide (GO) was synthesized from natural graphite powder via a modified Hummer's method [28]. As a typical process for synthesis of nanocomposite, STO nanocubes were dispersed in deionized water and GO sheets were dissolved in alcohol, 30 min vigorous stirring was carried out before mixing with each other. Then, the resultant suspension was transferred into a 35 mL of Teflon sealed autoclave and maintained at 120 °C for 4 h. In such a supercritical condition, GO sheets were reduced to rGO. Black precipitates were obtained and rinsed with deionized water and alcohol several times, then dried in vacuum at 50 °C. As a comparison, the contents of rGO in STO/rGO nanocomposites were controlled as 0, 2, 4, 6, 8 and 10 wt%.

2.2. Materials characterizations and photocatalytic performance

The crystalline properties of the samples were obtained from an X-ray diffractometer (X'pert PRO). The microstructures were characterized by transmission electron microscope (TEM, HT-7700). Raman spectra were obtained from the spectrometer (LabRAN Aeamis, 523 nm exciton wavelength). The photocatalysis of STO nanocubes and STO/rGO nanocomposites were evaluated by measuring the photo-decomposition of Rhodamine B dye (Rh B). UV-irradiation was provided by a 400 W mercury lamp with intensity on the mixture surface of 40 mW/cm². The photocatalysis measurement of the sample is performed according to the literature [27].

3. Results and discussion

As shown in Fig. 1a, nine marked diffraction peaks are well agree with the XRD patterns of STO, suggesting the sample is of STO phase and is well crystalline. The XRD patterns did not depict distinct difference between pure-STO and STO/rGO nanocomposites because the content of rGO is too little. The only difference is that a weak amorphous peak in red curve around 20° due to the effect between STO and rGO sheets, which preliminarily proving the existence of rGO. In order to further define the existence of rGO sheets, Raman spectroscope was used due to its special response of sp² and sp³ hybridized carbon atoms. As shown in Fig. 1b, the distribution of D band and G band of the nanocomposites appear around 1348 and 1590 cm⁻¹ respectively, which are matching well with previous studies [29,30].

morphologies of the pure STO and the nanocomposites can be observed visually by TEM. Fig. 2a and b display 5–10 nm monodispersed STO nanocubes. High resolution TEM (HRTEM) image (Fig. 2c) shows crystal lattice stripe clearly with a spacing, *d*, of 0.278 nm, corresponding to the (110) lattice plane of STO. TEM images of STO/rGO nanocomposites are shown in Fig. 2d–f. Particularly, the edge and the curly stripe of graphene sheets can be clearly seen in Fig. 2d and e, respectively. HRTEM image of STO/ rGO nanocomposites (Fig. 2f) exhibits a single STO nanocube located near the edge of graphene sheets, and the interaction between STO and rGO is observed visually.

The photocatalytic process is generally considered as a twostep process including adsorption and degradation. Fig. 3 shows both adsorption and degradation of Rh B dye over six samples. In dark part of Fig. 3a, it is obvious that the adsorptive properties of samples are increased with the content of rGO. It can be attributed to the large surface area of rGO which provides a large number of adsorptive sites. It is well known that the adsorption process can increases the concentration of the organic molecules (Rh B in this work) near the catalytic surface and further influence the catalytic process. The UV photocatalysis of STO/rGO (Fig. 3a) does not exhibit a monotonous relation with the content of rGO but an optimal value of 6 wt% rGO. According to the theory of photocatalysis, an excited electron in the VB will break free and enter the CB, generating holes in the VB at the same time. Photoexcited electrons and holes can react with H₂O to create radical oxygen species, which can undergo chain reactions to decompose the pollutants. The low catalytic activity of pure STO may due to the fast electron-hole pair recombination (10^{-9} s) which seriously decreases the concentration of photon-generated carriers around catalysts surface [31-33]. The band gap between STO and rGO is \sim 0.1 eV, therefore, the enhanced photocatalysis activity of STO/ rGO can be explained by the transfer of photon-generated electrons from STO CB to rGO and the inhibition of electron-hole recombination. During above process, the π - π bond inside single rGO sheet also provides a shortcut that improves the electron transfer.

In addition, the degradation follows a pseudo-first-order reaction, which can be represented by following equation:

$$-\ln\left(\frac{C}{C_0}\right) = kt \tag{1}$$

where C is the concentration of the Rh B dye and C_0 is the initial concentration of the dye (Rh B). k is the velocity constant of the reaction value and t is the irradiation time (UV-irradiation). The fitting results are shown in Fig. 3b, all five different rGO ratio STO/



Fig. 1. (a) XRD Patterns and (b) Raman spectra of STO and STO/rGO.

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