

Short Communication

Phosphate-assisted one-pot synthesis of silver phosphate–mesoporous silica composite from sodium silicate and silver nitrate

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ABSTRACT

A facile one-pot method for silver phosphate–mesoporous silica composite was reported. In the synthesis procedure, sodium silicate was firstly added dropwise to acidic Pluronic P123 and silver nitrate aqueous solution, followed by addition of phosphate to adjust the pH to near-neutral. Through the salting-out and flocculation effect of phosphate, tiny silver phosphate particles and silicate oligomers were formed and mixed together to build the composite material. It was a new type of mesoporous silica composite which had a structure similar to SBA-15. And it was found to have higher photocatalytic activity than pure silver phosphate in the degradation of methyl orange under ultraviolet light irradiation.

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1. Introduction

Silver phosphate (Ag_3PO_4) has been extensively studied because of its unique optical and electronic properties and its high capabilities in water splitting and organic dyes degradation [1,2]. Recently, numerous methods have been developed to prepare stable silver phosphate with controllable shape and size [3–5]. Despite the fact that Ag_3PO_4 demonstrates highly efficient photocatalytic performance, the high cost of the starting material (AgNO_3) and poor dispersity/solubility for irregular polyhedral microstructures limit large-scale production and application. Therefore, some Ag_3PO_4 -based composite photocatalysts have been reported, including $\text{TiO}_2/\text{Ag}_3\text{PO}_4$ [6,7], $\text{Fe}_3\text{O}_4/\text{Ag}_3\text{PO}_4$ [8], carbon quantum dots (CQDs)/ Ag_3PO_4 [9], $\text{Ag}/\text{Ag}_3\text{PO}_4$ [10–12], $\text{AgX}/\text{Ag}_3\text{PO}_4$ [13], $\text{SnO}_2/\text{Ag}_3\text{PO}_4$ [14], and so forth. But up to now, low-cost fabrication of well-defined Ag_3PO_4 -based photocatalysts with superior photocatalytic activities by a simple process remains a big challenge.

Mesoporous silicas possess high specific surface areas, high pore volumes, and well-ordered pore structures with uniform mesopores. They are frequently employed as catalyst supports,

absorbents, and drug delivery materials [15–17]. The pore system of silica-based mesoporous materials with the narrow pore size distribution is ideal to stabilize different active materials in a high dispersion. Mesoporous silicas, which are transparent for UV–vis light, have proven to be a promising candidate for fabrication of responsive photocatalytic composite [18]. SBA-15 is mesoporous silica with two-dimensional hexagonal arrays of channels, usually synthesized under strongly acidic conditions by relatively expensive silicon alkoxides such as tetraethoxy silane (TEOS) as Si source [19,20], and Ag_3PO_4 can be easily dissolved in acid. These limit the direct fabrication of Ag_3PO_4 -mesoporous silica composite. Sodium silicate [21,22] is a cheaper Si source than TEOS, herein, a one-pot method for the preparation of Ag_3PO_4 -mesoporous silica composite was reported using sodium silicate and silver nitrate under acidic P123 aqueous solution followed by an aid of phosphate to quasi-neutral pH. The obtained composite was a highly ordered material, and it had a similar structure with SBA-15 but with larger mean pore diameter. It was found to have higher photocatalytic activity than pure silver phosphate for degradation of methyl orange (MO). This method not only provided efficient and reproducible control for the improvement of the photocatalytic performance of silver phosphate, but also decreased the high cost of silver phosphate for practical environmental applications. To the best of our knowledge, reports on synthesis and photocatalytic performance of such a photocatalyst are rare.

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2. Experimental

2.1. Preparation of Ag_3PO_4 -mesoporous silica composite

All of the chemicals were purchased from Tianjin Kermel chemical reagent Co. Ltd., China except Pluronic P123 from Sigma. In a typical synthesis, 1.54 g of P123 was dissolved in 3.0 ml HNO_3 (65%) and 31 ml of water, followed by 0.25 g of AgNO_3 . While stirring, a mixture of 3.8 g of sodium silicate and 30 ml of water at ambient temperature was slowly added; then, a solution of 6.64 g $\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$ and 20 ml of water was added dropwise. The yellowish precipitation was formed by flocculation with the dropwise adding of Na_2HPO_4 solution. After transfer to a Teflon-flask for hydrothermal treatment at 100 °C for 24 h, the solid were filtered, washed with water, and dried in air. The surfactant was removed with alcohol by Soxhlet extractor. The obtained product was designated as SPMSC1.

2.2. Preparation of silver phosphate

Na_2HPO_4 aqueous solution (10 ml, 0.2 M) was added dropwise to the aqueous solution of AgNO_3 (10 ml, 0.6 M), and golden yellow solids were collected by filtration, washed with distilled water, and dried at 60 °C for 24 h.

2.3. Characterization

Transmission electron microscopy (TEM) images were obtained on a Hitachi H-7650 electron microscope operating at an accelerating voltage of 100 kV. Small angle X-ray diffraction (SAXRD) data were recorded on Rigaku D/MAX 2400 diffractometer equipped with a $\text{CuK}\alpha$ X-ray source operating at 40 kV and 50 mA. The X-ray powder diffraction (XRD) was carried out on a MiniFlex 600 instrument (Japan) with $\text{Cu K}\alpha$ radiation ($\lambda = 1.5406 \text{ \AA}$) at 40 kV and 15 mA. X-ray photoelectron spectroscopy (XPS) analysis was measured on an EDAX GENESIS 60S X-ray photoelectron spectrometer using Mg as the exciting source. X-ray fluorescence (XRF, Horiba XGT-1000WR) was used to determine the chemical compositions of SPMSC1. Nitrogen sorption isotherms were obtained with a Quantachrome Nova volumetric adsorption analyzer at 77.3 K with samples outgassed at 150 °C for 12 h. Pore size distribution was calculated from the desorption isotherm using the BJH model and the BET surface area from the relative pressure of 0.057–0.346.

2.4. Photocatalytic evaluation

Photocatalytic activities of SPMSC1 and Ag_3PO_4 were evaluated by degradation of methyl orange (MO) under a 175 W Hg lamp with 365 nm wavelengths. MO solutions (110 ml, 30 mg/l) containing 50 mg of SPMSC1, 8.5 mg or 17.2 mg of Ag_3PO_4 and a blank test were put in a glass beaker, respectively. Before irradiation, the solution was stored in the dark for 20 min under stirring. 3 ml of the sample solution was taken at given time intervals and separated through centrifugation (12,000 rpm, 2 min). The supernatants were analyzed by recording variations of MO at the absorption band maximum (464 nm) in the UV-vis spectra using a U-3010 spectrophotometer (Hitachi).

3. Results and discussion

SPMSC1 was not calcined, surfactant was removed with alcohol. Transmission electron micrograph (TEM) image of SPMSC1 in Fig. 1 revealed that the morphology of SPMSC1 was sheet particles with thickness of ca. 100–200 nm (Fig. 1a, low magnification) and showed that it also had 2D-hexagonal ordering (Fig. 1b, high

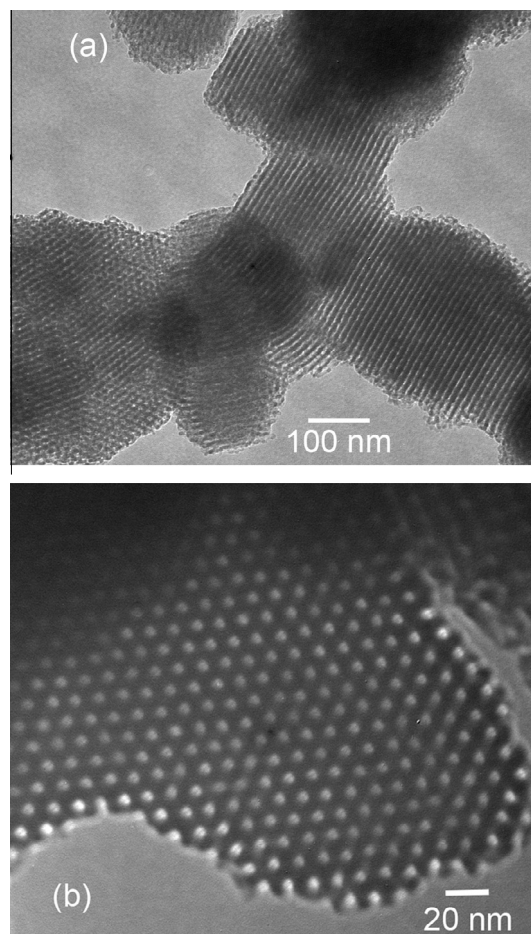


Fig. 1. TEM images of SPMSC1 after the surfactant was removed with alcohol: (a) low and (b) high magnification.

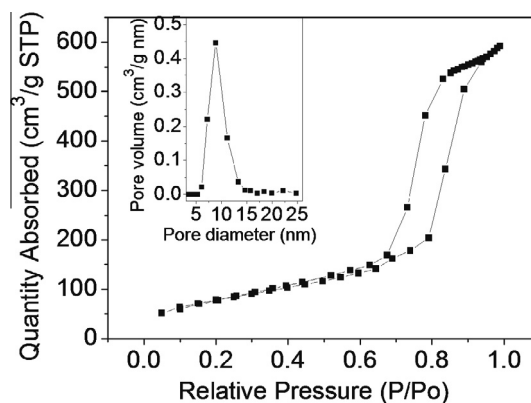


Fig. 2. Nitrogen adsorption isotherm of SPMSC1. Inset: BJH pore size distribution derived from the desorption branch.

magnification). No evident particles were observed in the nanopores of SPMSC1.

Fig. 2 displayed a typical type IV nitrogen sorption isotherm with an H1 hysteresis loop, which was the characteristic of mesostructure ordered from SPMSC1. The BET surface area was $287.5 \text{ m}^2/\text{g}$ and the total pore volume at $P/P_0 = 0.99$ was $0.917 \text{ cm}^3/\text{g}$.

The small-angle X-ray scattering (SAXS) of SPMSC1 (Fig. 3a) showed five resolved peaks that were indexable as (100), (110), (200), (210) and (300) reflections associated with $p6mm$

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