

CTAB-assisted hydrothermal synthesis of silver vanadates and their photocatalytic characterization

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ABSTRACT

Silver vanadates, visible-light-driven photocatalysts, have been synthesized using hydrothermal treatment with the assistance of cetyltrimethylammonium bromide (CTAB). It is found that CTAB during the hydrothermal synthesis significantly affects the morphology, crystal structure, optical absorbance, and photocatalytic properties of silver vanadates. The pure silver vanadate oxides (SVO) consisted of mixed structures of $\text{Ag}_4\text{V}_2\text{O}_7$ and $\alpha\text{-Ag}_3\text{VO}_4$, with $\alpha\text{-Ag}_3\text{VO}_4$ as the major phase. As the amount of CTAB increased, the crystal phase of CTAB-SVO sample approached $\text{Ag}_4\text{V}_2\text{O}_7$. The band gaps for all CTAB-SVO samples were found to be in the range of 2.29–2.43 eV. The reactivity of 0.5%CTAB-SVO (surface area, $6.51 \text{ m}^2 \text{ g}^{-1}$) on isopropanol was 10 times higher than that of P25 (surface area, $49.04 \text{ m}^2 \text{ g}^{-1}$) under visible light irradiation. The photocatalytic activity of CTAB-SVO sample decreased with an increase of $\text{Ag}_4\text{V}_2\text{O}_7$. The highest photocatalytic activity of 0.5%CTAB-SVO had the highest intensities of surface hydroxyl groups, which were detected using the in-situ DRIFT technique. The density of surface hydroxyl groups, not the crystalline structure, was confirmed as the key factor influencing the photocatalytic activity.

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1. Introduction

Recently, a great number of monoclinic and perovskite visible-light-active photocatalysts have been developed using a solid-state reaction, such as InMO_4 ($M = \text{V}, \text{Nb}, \text{Ta}$) (Ye et al., 2002), Ag_3VO_4 (Konta et al., 2003), BiVO_4 (Zhou et al., 2006), and $\text{In}_2\text{O}_3/\text{CaIn}_2\text{O}_4$ (Chang et al., 2007). The valence bands of these oxide photocatalysts consist of V 3d, Nb 4d, Ta 5d, Bi 6s, and Ag 4d orbitals hybridized with the O 2p orbitals, respectively, resulting in a higher valence band, and thus leading to a narrower band gap. The disadvantage of solid-state reactions is the requirement of high purity metal oxides powders as precursors, which need to be mechanically mixed and reacted at 850°C (or higher) for at least 12 h. To reduce energy consumption, exploiting low cost metal oxide catalysts with high visible-light photocatalytic activity is desirable. The surfactant-assisted hydrothermal synthesis method has been considered as a relatively simple and high yield process to grow various metal oxides, such as TiO_2 (An et al., 2008), ZnO (Wei and Chang, 2008), and $\text{TiO}_2\text{-SiO}_2$ (Zhang et al., 2005), at much lower temperature (less than 200°C), leading to the reduction of energy consumption.

In the present study, a series of silver vanadate photocatalysts was synthesized using a surfactant-assisted hydrothermal method. The effects of the amount of surfactant on the crystal structure, morphology, and photocatalytic properties of silver vanadate products are discussed in detail.

2. Experimental

2.1. Preparation of photocatalysts

Silver vanadate materials were synthesized as follows: AgNO_3 and various molar ratios of CTAB (cationic surfactant cetyltrimethylammonium bromide, $\text{C}_{19}\text{H}_{49}\text{BrN}$) were dissolved in 180 ml of de-ionized water and stirred for 1 h, with the AgNO_3 concentration kept at 0.03 M. The transparent solution was added dropwise to 60 ml of 0.03 M NH_4VO_3 under continuous stirring. The pH value of the mixture was then adjusted to 7 with ammonia solution. After being aged at room temperature for 24 h, the mixture was poured into a 320 ml Teflon-lined stainless reactor, heated to 140°C for 4 h, and then cooled to room temperature naturally. The resulting products were washed, first with water and then absolute ethanol by centrifugation/dispersion cycles 3 times to remove the remaining CTAB, NO_3^- , and NH_4^+ residues, and dried in an oven at 100°C for 6 h. Samples were labeled as x%CTAB-SVO, where x is the molar ratio

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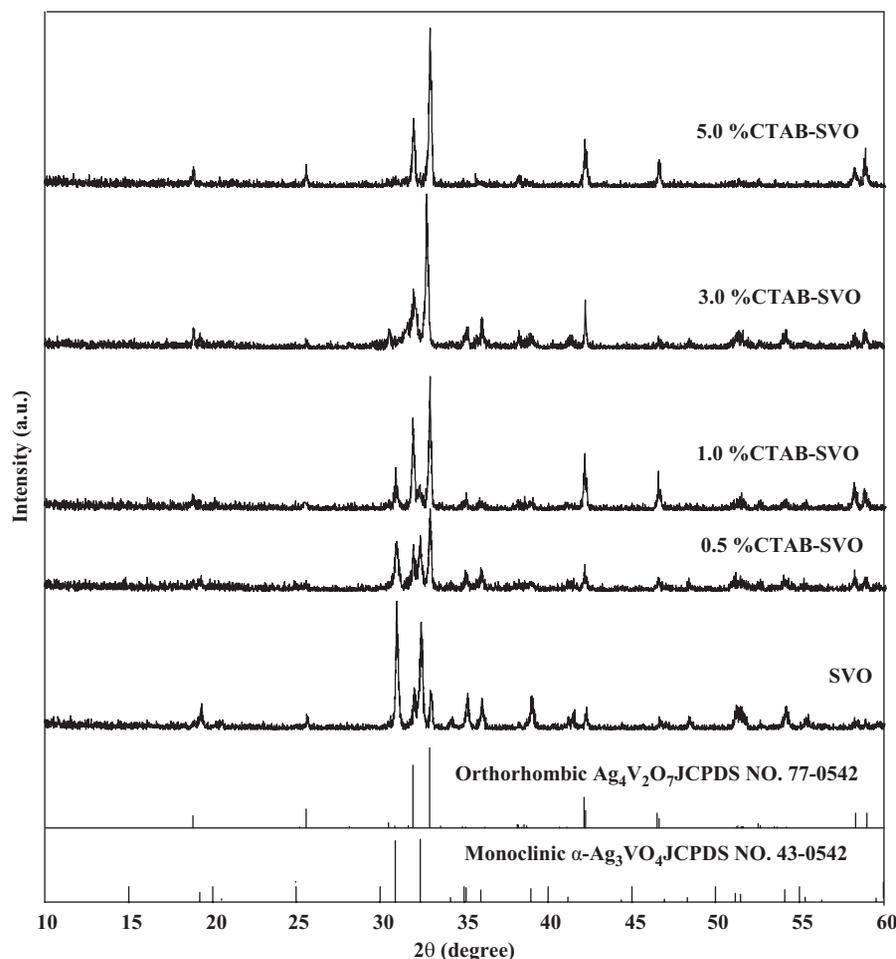


Fig. 1. XRD patterns of silver vanadates and CTAB-SVO photocatalysts.

of CTAB/Ag. In this study, five samples were prepared: SVO (without CTAB), 0.5%, 1%, 3%, and 5% CTAB-SVO.

2.2. Characterization of photocatalysts

The X-ray diffraction (XRD) patterns of the powders were measured using an X-ray diffractometer (PANalytical X'Pert PRO) with Cu radiation ($\lambda = 0.15418$ nm) in the region of 10–80°. The surface morphology was examined using a Field Emission Scanning Electron Microscope (JEOL JSM-6700F). UV–vis spectra were collected using a spectrophotometer (JASCO V-500), equipped with an integrated sphere assembly over the range of 300–750 nm.

2.3. Photocatalytic activity

Photoactivity for the gaseous isopropanol (IPA) was tested in a continuous-flow annular photoreactor (Huang et al., 2007) containing ca. 0.05 g of photocatalyst evenly spread on a Pyrex tube. The photocatalyst was treated initially under dry nitrogen gas (100 ml/min) for 30 min before the reactant mixture was introduced. The reactant mixture (20 ml/min) was prepared by introducing oxygen gas that was continuously bubbling through the IPA solvent-containing bottle at room temperature, yielding an IPA concentration of ca. 160 ppmv. After the reactant mixture was purged in the dark for 60 min to achieve adsorption equilibrium of IPA on the catalyst, the catalyst was irradiated by a white fluorescent daylight lamp (TFC, FL10W-EX).

A constant amount of gaseous IPA was periodically withdrawn from the sampling port and injected into the quadrupole mass spectrometer (SRS QMS300) to determine the concentration.

3. Results and discussion

3.1. XRD patterns analysis

Powder XRD patterns of the as-prepared SVO and CTAB-SVO photocatalysts under various CTAB concentrations are shown in Fig. 1. Characteristic peaks are indexed to the standard cards of $\text{Ag}_4\text{V}_2\text{O}_7$ (JCPDS 77-0097) and $\alpha\text{-Ag}_3\text{VO}_4$ (JCPDS 43-0542). The CTAB-free solution was composed of the mixed structures of $\text{Ag}_4\text{V}_2\text{O}_7$ and $\alpha\text{-Ag}_3\text{VO}_4$. In the solution containing 0.5%CTAB, the samples had nearly equal amounts of $\text{Ag}_4\text{V}_2\text{O}_7$ and $\alpha\text{-Ag}_3\text{VO}_4$. With an increase of the molar ratio of CTAB in the reaction solution, the peaks of the $\text{Ag}_4\text{V}_2\text{O}_7$ crystal sharply increased, while the other peaks of $\alpha\text{-Ag}_3\text{VO}_4$ significantly decreased. An overall enhancement of the intensity of the diffraction peaks of $\text{Ag}_4\text{V}_2\text{O}_7$ is clearly visible for 5%CTAB-SVO, indicating that an increase of the concentration of CTAB might be favorable for the formation of $\text{Ag}_4\text{V}_2\text{O}_7$ crystal.

3.2. Morphology of silver vanadates

Fig. 2 shows the corresponding SEM images of as-prepared samples. Irregular ragged particles with sharp edges and clear grain boundaries were observed on pure SVO samples, as shown in

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