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Flower-like ZnO-Ag₂O composites: precipitation synthesis and photocatalytic activity

Lingling Xu^{1*}, Bo Wei², Weilong Liu², Hailin Zhang¹, Chunyan Su³ and Jixin Che⁴**Abstract**

Ag₂O-decorated flower-like ZnO composites were fabricated through a chemical precipitation process. X-ray diffraction analysis confirms the co-existence of cubic Ag₂O and wurtzite ZnO phases. Scanning electron microscopy images reveal Ag₂O nanoparticles located on the rough surface of ZnO flowers. The photocatalytic activities of the composites with various mole ratios were evaluated by the degradation of methyl orange (MO) under ultraviolet irradiation, which confirms that the composite shows superior activity to that of pure ZnO and Ag₂O. The improvement can be ascribed to the deposited Ag₂O forming the p-n junction at the interface of ZnO and Ag₂O, resulting in the transfer of photocarriers and suppressing the electron-hole recombination rate.

Keywords: ZnO; Ag₂O; Photocatalytic activity; Composite

Background

Semiconductor photocatalysts for clean hydrogen energy production and environment decontamination have attracted much interest [1,2]. When the excitation energy is higher than or equal to the band gap energy of the semiconductor, photoinduced electron-hole pairs would be generated and utilized in initiating oxidation and reduction of organic compounds. ZnO can be used as a photocatalyst and has drawn increasing attention because its photocatalytic activity is comparable to that of TiO₂ [3,4]. It has been reported that the photocatalytic activity is closely correlated with the morphology of photocatalysts [5]. Hierarchical ZnO with flower-like morphology shows promising application in decomposition of organic pollutant due to the increased optical absorption efficiency and large specific surface area [6,7]. However, due to the wide band gap of ZnO (3.2 eV), only a few part of natural solar radiation can be utilized and the photogenerated electron and hole pairs are liable to recombination, leading to low quantum yields. To improve its photocatalytic activity, one of strategy is to complex ZnO with a narrow-band semiconductor forming composites with a wider range

light absorption and a reduced rate of the recombination of photogenerated electrons and holes.

Many reports focused on the enhanced photocatalytic performance of ZnO composites by coupling with suitable semiconductors, such as TiO₂, ZnS, Bi₂O₃, and CuO [8-12]. The efficiency improvement on the degradation of organic dye can be ascribed to the effective separation of photoinduced carriers. Furthermore, the separation of photoinduced electrons and holes would be greatly enhanced and more efficient especially in the inner electric field, which was formed by a p-n-type semiconductor composite, such as CuO/ZnO and NiO/ZnO [12,13]. Ag₂O is a p-type semiconductor with a band gap of about 1.3 eV. Recently, the modification of TiO₂ and Bi₂O₃ was carried out using Ag₂O nanoparticles decorated on the surface of photocatalysts [14-17]. Based on the heterojunction of Ag₂O and TiO₂, the recombination of photogenerated electrons and holes was greatly inhibited by transferring for the energy band matching and the build-up inner electric field, resulting in the photocatalytic activity enhancement [15,16]. However, to the best of our knowledge, there is no report in the literature on the photocatalytic properties of the p-n junctions of hierarchical mesoporous ZnO-Ag₂O composites.

In this paper, flower-like ZnO-Ag₂O composites were fabricated through a chemical co-precipitation process. The as-prepared composite including Ag₂O particles

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deposited on the petal surfaces of ZnO flowers shows high crystallization. Compared with ZnO flowers and Ag₂O particles, the photocatalyst ZnO-Ag₂O composites with wide mole ratios exhibited enhanced photocatalytic properties that was confirmed by the degradation of methyl orange (MO) under ultraviolet irradiation.

Methods

Preparation of flower-like ZnO

All the chemicals used for the synthesis of flower-like ZnO are analytical grade reagents. Zinc nitrate solution (0.001 M) was prepared by dissolving a proper amount of Zn(NO₃)₂ in deionized water. The materials - 20 mL of Zn(NO₃)₂ solution, 20 mL of deionized water, 0.25 g of sucrose, and 1.2 g of urea - were added into a 50-mL Teflon-lined stainless steel autoclave. The autoclave was sealed, heated at 90°C for 2 h, and finally cooled to room temperature naturally. The white precipitation (precursor) was filtered and washed several times with deionized water, followed by drying in air at 90°C for 2 h. The precipitations were heat-treated at 600°C in air for 2 h (heating rate of 5°C min⁻¹) in a muffle furnace to obtain the final hierarchical ZnO flowers.

Preparation of Ag₂O nanoparticles

Ag₂O nanoparticles were synthesized from AgNO₃, NaOH, and polyethylene glycol 8000 (PEG-8000) aqueous solution by the precipitation method. Firstly, 1.75 g of AgNO₃ and 0.2 g of PEG-8000 were dissolved in 100 mL of deionized water. After a continuous stirring for 15 min, 0.05 M NaOH aqueous solutions were dropped into the above aqueous solution with the final pH = 14. Finally, Ag₂O nanoparticles were washed thoroughly with deionized water followed by drying in air at 90°C for 2 h.

Chemical synthesis of flower-like ZnO-Ag₂O composites

Flower-like ZnO-Ag₂O composites with different mole ratios were prepared by the chemical precipitation method. A typical experimental process for the composite with a mole ratio of 1:1 is given as follows: 0.4 g of flower-like ZnO was dispersed in 100 mL of deionized water, and 2 g of PEG-8000 was added into the mixture in order to immerse the ZnO thoroughly. Subsequently, 1.8 g of AgNO₃ was added to the suspension, and the mixture was stirred magnetically for 30 min. Then 0.2 M of NaOH was dropped into the above mixture with the final pH value of 14. Finally, flower-like ZnO decorated by Ag₂O nanoparticles was washed repeatedly with deionized water followed by a filtration and drying in air at 90°C for 2 h. In order to assess the relationship between the component and the photocatalytic activity of the composites, variable mole ratios of ZnO to Ag₂O composites were prepared through a similar process.

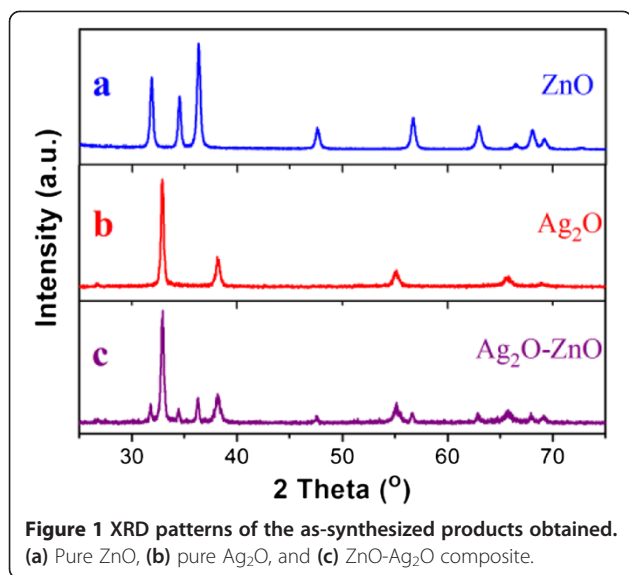
Characterizations and photocatalytic testing

X-ray diffraction (XRD) measurement was carried out using a Rigaku-D/max 2500 diffractometer (Rigaku, Shibuya-ku, Japan) with Cu-K α radiation ($\lambda = 0.15418$ nm) for crystallization identification. The morphology, particle size, and chemical composition of the product were examined by scanning electron microscopy (SEM; Hitachi S-4800, Chiyoda-ku, Japan). X-ray photoelectron spectroscopy (XPS) experiments were performed with a Thermo Fisher K-Alpha X-ray photoelectron spectrometer (Thermo Fisher Scientific, Waltham, MA, USA) using Al K α radiation (12 kV, 6 mA). The binding energies of elements were calibrated using C 1s (284.6 eV) as reference. Room-temperature ultraviolet-visible (UV-vis) absorption spectrum was recorded on a spectrophotometer (PerkinElmer Lambda-35, Waltham, MA, USA) in the wavelength range of 300 to 800 nm. The UV-vis diffuse reflectance spectra (DRS) were measured using a Shimadzu UV-2550 spectrophotometer (Shimadzu, Kyoto, Japan). Room-temperature photoluminescence (PL) spectra were collected with a laser micro-Raman (JY HR800, HORIBA, Kyoto, Japan).

MO was employed as a representative dye pollutant to evaluate the photocatalytic activity of ZnO-Ag₂O composites. Next, 0.02 g of ZnO-Ag₂O composites was suspended into 60-mL 2×10^{-5} M of MO aqueous solution and stirred for 30 min in a 200-mL beaker in the dark to reach an adsorption/desorption equilibrium for MO on the surface of ZnO-Ag₂O composites. Then the mixture was irradiated by 16-W ultraviolet irradiation (Philips, Amsterdam, The Netherlands) at room temperature. After the reaction mixture was irradiated for a given time, the samples of 4 mL were withdrawn at each time and centrifuged for 20 min. The quantitative determination of MO was performed by measuring its absorption with a UV-vis spectrophotometer (PerkinElmer Lambda-35).

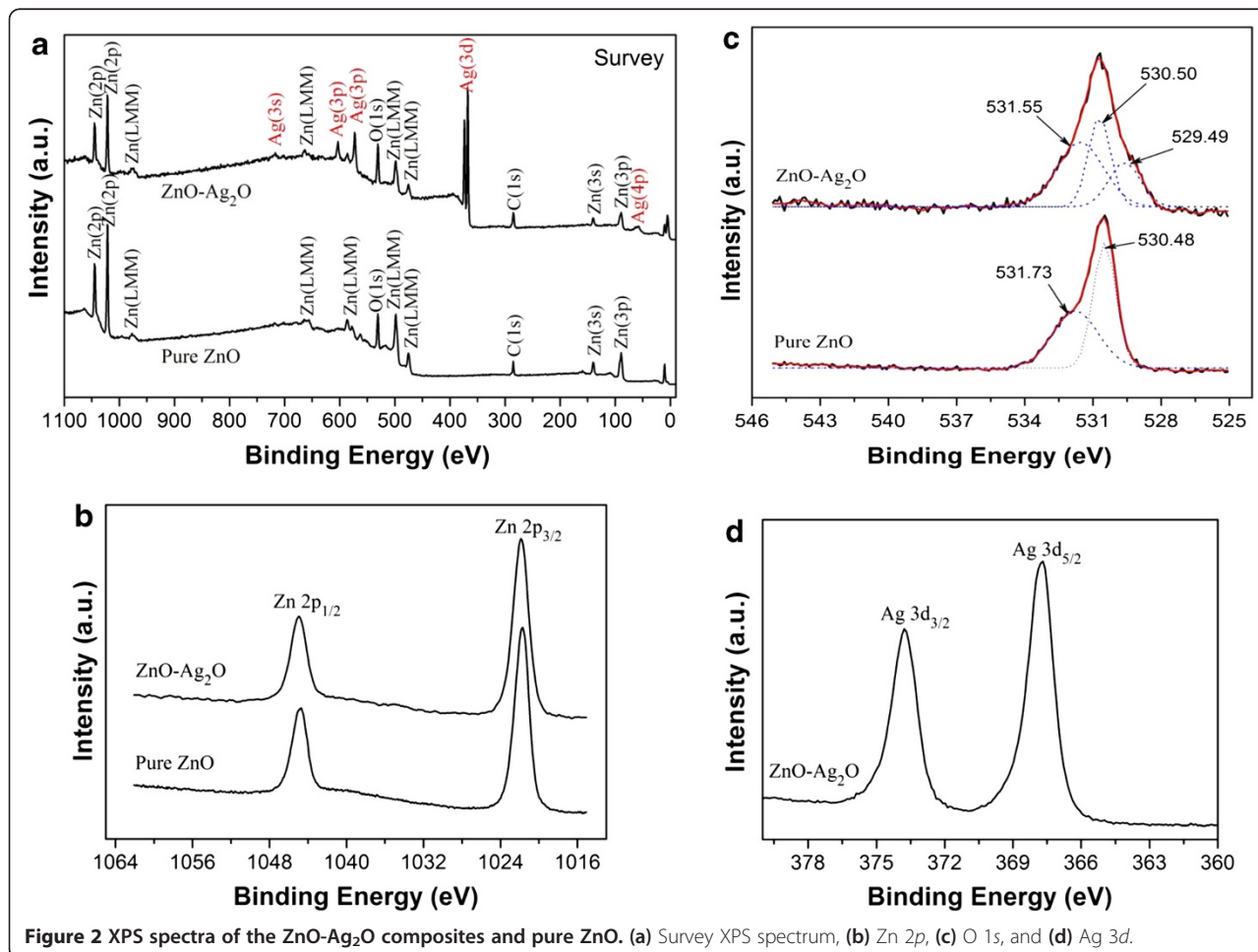
Results and discussion

The structure and phase purity of Ag₂O nanoparticles, flower-like ZnO, and ZnO-Ag₂O (1:1) composites were examined by XRD, and the patterns are shown in Figure 1. The diffraction peaks of the ZnO consist of three strong diffraction peaks, which can be mainly indexed as the wurtzite phase of ZnO (JCPDS card no. 36-1451) in Figure 1a. Meanwhile, the diffraction peaks in Figure 1b can be indexed to the cubic structure of pure Ag₂O (JCPDS card no. 76-1393), with no additional peak detected, indicating the pure phase of Ag₂O products. For the composite sample, the diffraction peaks in Figure 1c can be ascribed to two sets of strong diffraction peaks (JCPDS card nos. 36-1451 and 76-1393), revealing that ZnO and Ag₂O coexist in the composite. The relative intensity of diffraction peaks in Figure 1c shows that the



content of Ag₂O is much more than that of ZnO for its intense and sharp diffraction peaks.

To investigate the surface compositions and chemical states of the as-prepared ZnO-Ag₂O (1:1) composite, XPS was carried out, and the results are shown in Figure 2. The full-scan spectrum in Figure 2a shows the presence of C, O, Zn, Ag, and O peaks, which confirmed the presence of these elements in the products. The carbon peak comes from the adventitious carbon on the surface of the sample. The Zn 2*p* consists of two peaks positioned at 1,020.9 and 1,044.2 eV for Zn 2*p*_{3/2} and Zn 2*p*_{1/2} (Figure 2b), which were observed in both ZnO-Ag₂O composites and pure ZnO [18]. As Figure 2c shows, O 1*s* can be deconvoluted by three nearly Gaussian curves in the ZnO-Ag₂O composite, indicating that there are three different O species in the sample. The lowest binding energy component of 529.5 eV is attributed to O²⁻ ions surrounded by Ag atoms with their full complement of nearest-neighbor O²⁻ ions [19]. The middle binding energy component is usually attributed to chemically adsorbed oxygen on the surface of the catalysts [20]. The highest component is attributed to O²⁻ ions in ZnO [21]. However, O



1s only can be deconvoluted by two nearly Gaussian curves in pure ZnO. The binding energy components of 530.5 and 531.7 eV are attributed to chemically adsorbed oxygen and O²⁻ ions in ZnO, respectively. The peaks with binding energies of 367.8 and 373.8 eV correspond to Ag 3d_{5/2} and Ag 3d_{3/2}, respectively, which is a characteristic of Ag⁺ in the Ag₂O product in Figure 2d [21]. Consequently, the as-synthesized products could be determined as ZnO-Ag₂O composites based on the results of XRD and XPS measurements.

In order to obtain the detailed information about the morphology of the synthesized Ag₂O nanoparticles, SEM observation of flower-like ZnO and ZnO-Ag₂O (1:1) composites was carried out, and the results are given in Figure 3. Figure 3a shows the SEM image of ZnO flower by the hydrothermal process. It reveals that the ZnO has a diameter of 5 to 10 μm and possesses a flower-like rough surface with petals emitted from the center. A typical ZnO flower image is shown in Figure 3b. Obviously, it is about 1 μm at the widest point of the flower petals which are ended with a tip. Moreover, there are a large amount of holes on the petals, which can greatly enlarge the contact area between the organic dyes and ZnO. The ending part of saw-like petals is shown as inserted in Figure 3b. It can be seen that holes on the petals present an irregular shape with an average diameter below 100 nm. Considering the annealing process, it can be deduced that the holes are coming from amounts of gases evaporating with the decomposition of the precursor at the relatively high temperature. The rough surfaces of

ZnO provide a very good platform to locate Ag₂O nanoparticles in high density during the co-precipitation process. Figure 3c shows the morphology of the Ag₂O nanoparticles obtained by the precipitation method. Obviously, the diameter of Ag₂O particles is in the range of 100 to 500 nm. The enlarged view as inserted in Figure 3c shows that the Ag₂O presents a rough surface with small spherical particles. For the composited sample, the morphology maintained the flower of ZnO, while Ag₂O clusters were observed on the petals. From the insert in Figure 3d, it shows that the Ag₂O cluster was composed of dozens of Ag₂O nanoparticles.

It is known that MO dyes are usually chosen as model pollutants to simulate the actual photocatalytic degradation of organic pollutants. The degradation efficiency was calculated using Equation 1:

$$\text{Degradation (\%)} = \frac{C = C_0 - \Delta C}{C_0 \times 100} = \frac{A = A_0 - \Delta A}{A_0 \times 100} \quad (1)$$

where C₀ represents the initial concentration, ΔC represents the changed concentration, C represents the reaction concentration, A₀ represents the initial absorbance, ΔA represents the changed absorbance, and A represents the reaction absorbance of the MO at the characteristic absorption wavelength of 464 nm.

In the experiments, the photocatalytic activities of the as-prepared samples with different ZnO-Ag₂O composites, pure ZnO flowers, and Ag₂O particles are shown in Figure 4a. Surprisingly, the ZnO-Ag₂O (1:1) composite

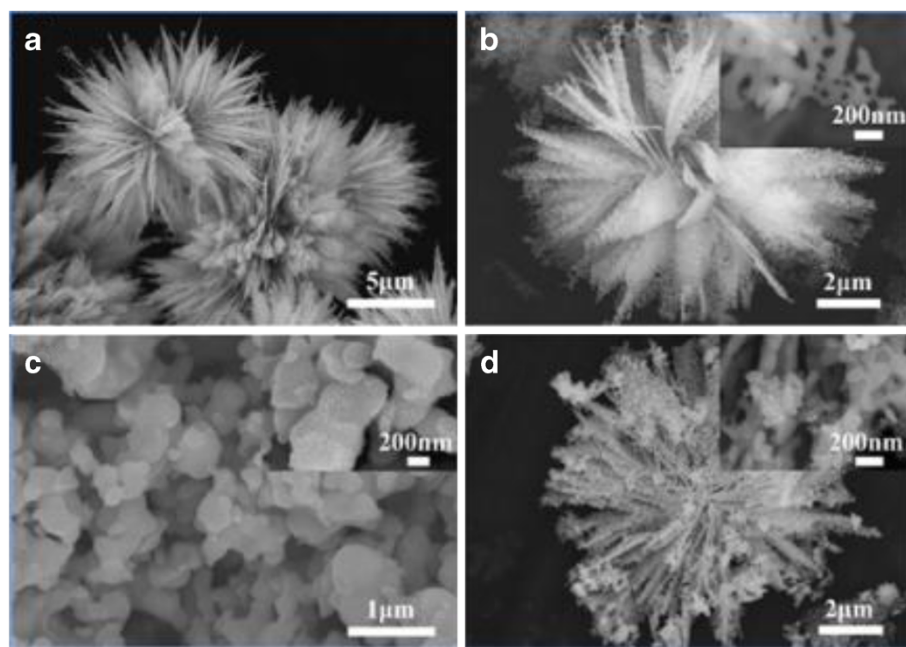


Figure 3 SEM images of pure ZnO, pure Ag₂O, and ZnO-Ag₂O composite. (a) Low-magnification SEM image of pure ZnO, (b) high-magnification SEM image of pure ZnO, and (c, d) typical images of pure Ag₂O and ZnO-Ag₂O composite.

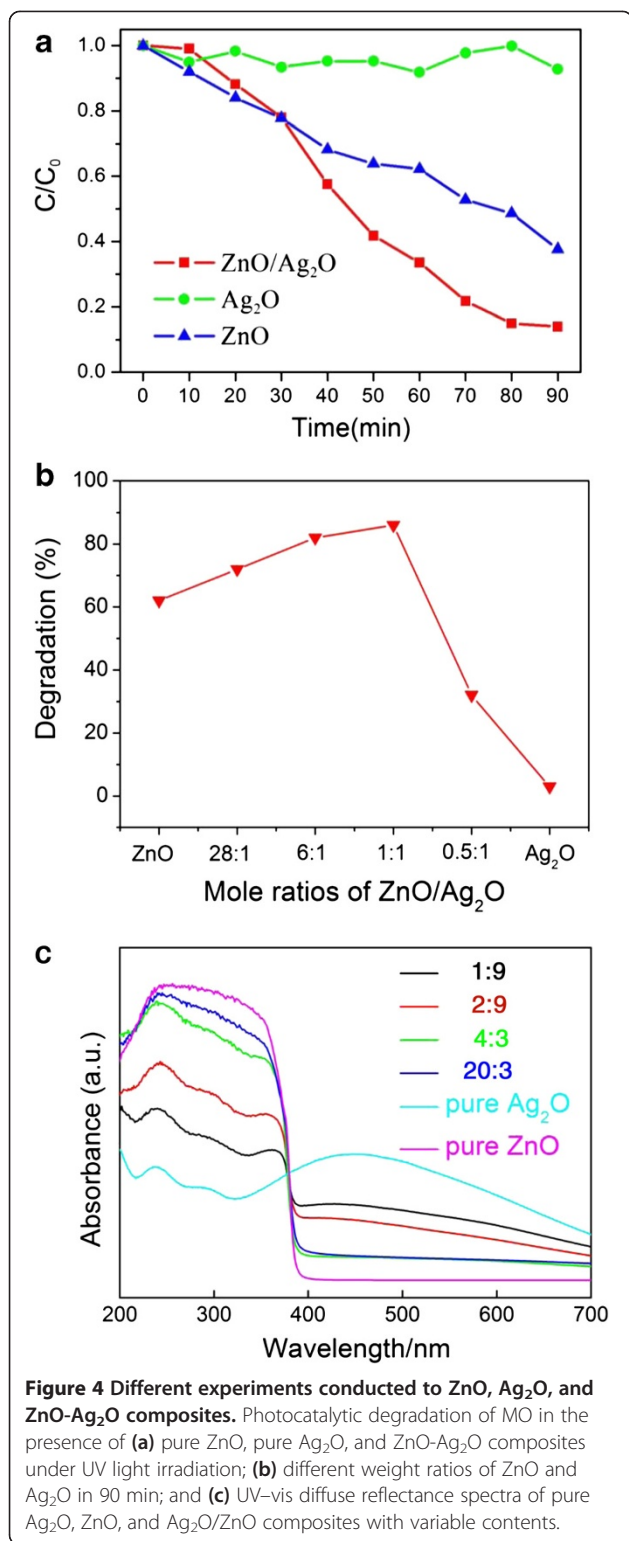


Figure 4 Different experiments conducted to ZnO, Ag₂O, and ZnO-Ag₂O composites. Photocatalytic degradation of MO in the presence of (a) pure ZnO, pure Ag₂O, and ZnO-Ag₂O composites under UV light irradiation; (b) different weight ratios of ZnO and Ag₂O in 90 min; and (c) UV-vis diffuse reflectance spectra of pure Ag₂O, ZnO, and Ag₂O/ZnO composites with variable contents.

exhibits superior photocatalytic activity, which is higher than that of pure ZnO flowers and Ag₂O nanoparticles; for example, the required time for an entire decolorization of MO over ZnO-Ag₂O catalysts is less than or equal to 90 min, much shorter than the corresponding value over

pure ZnO flower and Ag₂O particles. Moreover, the correlation between the photocatalytic activity and the component mole ratios is shown in Figure 4b. Obviously, the photocatalytic activity increases gradually with an increase of the Ag₂O mole ratios (1:1 > 6:1 > 28:1 > 0.5:1) except ZnO-Ag₂O (0.5:1). The composite ZnO-Ag₂O (1:1) performed the highest activity among the samples we examined during a given time for 90 min. In addition, the ZnO-Ag₂O composite shows higher photocatalytic activity than the pure components, ZnO and Ag₂O. UV-vis diffuse reflectance spectra of pure Ag₂O, ZnO, and Ag₂O/ZnO composites with variable contents are shown in Figure 4c. Obviously, the absorption in the UV range is gradually quenched, while there is an obvious increase in the visible light range with the elevated loading of Ag₂O. As for the UV light-excited photocatalytic process, the ability of UV light absorption is crucial for the effective excitation of photoinduced electron and holes. Thus, the photocatalytic activity would be determined by both the quantity of excited photoinduced carriers and the effective separation process in the inner electric field.

Room-temperature photoluminescence measurements are widely used to characterize semiconductor nanoparticles, which possess a broad range of absorption, narrow emissions with high quantum yields, and size-tunable emission wavelength. The emission spectra of pure ZnO and ZnO-Ag₂O composites excited at the emission peak of 325 nm are given in Figure 5. The photoluminescence spectrum of ZnO is composed of two emission bands: a near band edge emission positioned in the UV range and a visible emission band resulting from the defects [22,23]. Both the composite sample and pure ZnO present a band edge emission peak centered at 380 nm,

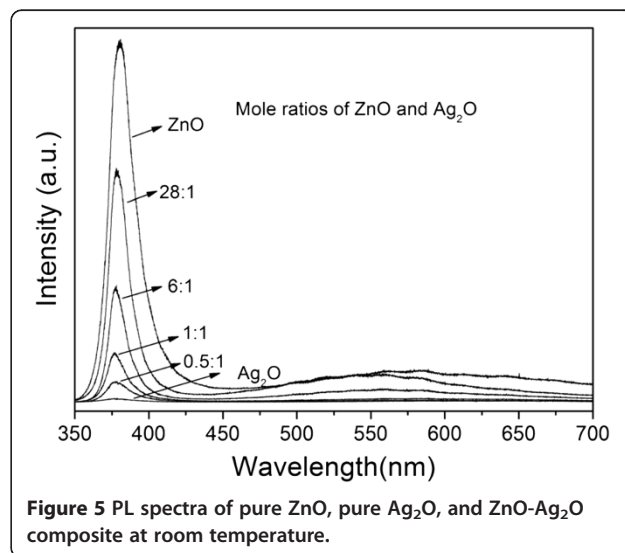
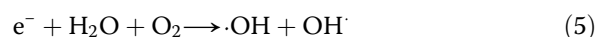


Figure 5 PL spectra of pure ZnO, pure Ag₂O, and ZnO-Ag₂O composite at room temperature.

while the band edge emission intensity of pure ZnO is drastically quenched by the increased loading of Ag₂O particles, indicating the existence of a direct interaction between Ag₂O and ZnO enhancing the nonirradiative relaxation of excitons formed in ZnO. The results demonstrate that the Ag₂O particles block both direct and trap-related charge carrier recombination pathways since Ag₂O particles on the ZnO surface can extract electrons from the conduction band of ZnO and act as a sink which can store and shuttle photogenerated electrons [14,15].

As shown in Figure 6, the schematic band structure of the synthesized ZnO-Ag₂O composite was proposed to discuss the possible process of the photocatalytic degradation of MO. When the catalysts are excited by ultraviolet light irradiation, electrons (e⁻) in the valence band (VB) can be excited to the conduction band (CB) with simultaneous generation of the same amount of holes (h⁺) in the VB, as demonstrated in Equations 2 and 3. The photoactivated electrons and holes in the ZnO-Ag₂O composite could inject into a reaction medium and participate in the photocatalytic reaction process. The photoinduced holes (trapped by H₂O) produce hydroxyl radical species (·OH) and the photoinduced electrons (trapped by O₂ and H₂O) produce hydroxyl radical species (·OH), which are extremely strong oxidants for the degradation of organic chemicals (Equations 4 and 5) [24]. It is known that ZnO is an n-type semiconductor while Ag₂O is a p-type semiconductor. Thus, the Fermi levels of both

n-type and p-type tend to obtain equilibrium, resulting in the energy bands of ZnO downward with the upward shifts of the Ag₂O band. Moreover, there will be an inner electric field in the interface between ZnO and Ag₂O in the composite, leading to a positive charge in the ZnO region and a negative charge in the Ag₂O part. After the illumination of UV light, the photoinduced electrons and holes are created in the composite and subsequently transferred by the drive of inner field. Photoinduced electrons in the CB of Ag₂O would move to the positively charged ZnO, while the holes of ZnO will be transferred to the negatively charged Ag₂O part by the potential energy. Hence, the photoinduced electrons and holes could be effectively separated through charge transfer process at the interface of the two semiconductors, and the photocatalytic process can be described as follows:



The results in this paper show that ZnO-Ag₂O composites have higher photocatalytic activities than pure ZnO and pure Ag₂O, which is mostly attributed to the inner electric field introduced by the n-type ZnO and p-type Ag₂O effectively separating the photoinduced electrons and holes.

Conclusions

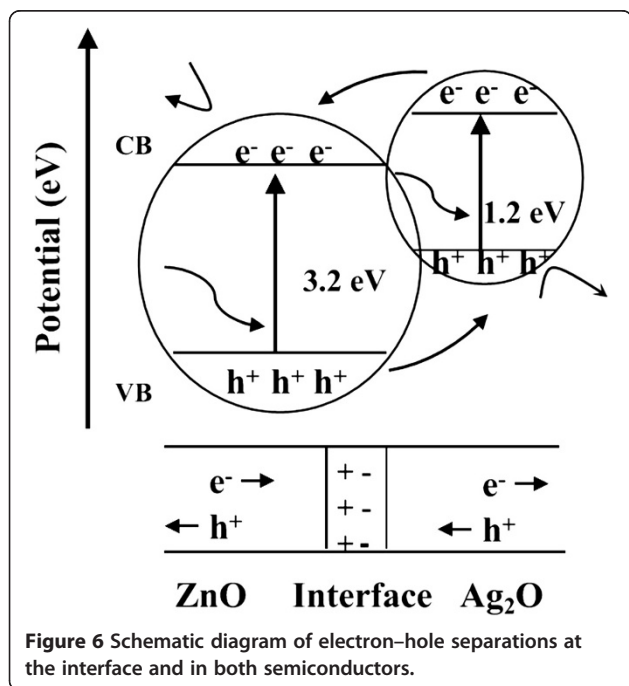
Flower-like ZnO-Ag₂O composites were prepared by a chemical co-precipitating method. The XRD profiles confirm that the composite is composed of cubic-phase Ag₂O and wurtzite-phase ZnO. Ag₂O particles decorated on ZnO composite flowers show higher photocatalytic activity than pure components under UV irradiation for the degradation of MO. The activity dependence on the component reveals that the increased Ag₂O deposited on the composite greatly enhanced the photocatalytic activity, which can be attributed to the p-n junction in the composite effectively inhibiting the recombination of electron-hole pairs.

Competing interests

The authors declare that they have no competing interests.

Authors' contributions

LLX planned the experiments, analyzed the data, and drafted the paper. BW and WLL supervised the project, analyzed the results, and wrote the paper. HLZ performed the experiments and collected the data. CYS and JXC helped with the analysis of the data. All the authors discussed the results and commented on the manuscript. All authors read and approved the final manuscript.



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