



# Nanoarchitectonics of Zn<sub>2</sub>SnO<sub>4</sub>/ZnO heterostructure composites for better photocatalytic performance

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## Abstract

In this work, the Zn<sub>2</sub>SnO<sub>4</sub>/ZnO nanocomposites synthesized by one-step hydrothermal method had been characterized by X-ray diffraction (XRD), Fourier transform-infrared spectroscopy (FT-IR), scanning electron microscopy (SEM), high-resolution transmission electron microscopy (HR-TEM), and surface area analysis (BET) techniques, while the photocatalytic activity of the Zn<sub>2</sub>SnO<sub>4</sub>/ZnO had been investigated during the degradation process of organic pollutants under UV irradiation. The results indicate that the Zn<sub>2</sub>SnO<sub>4</sub>/ZnO displays a cube and rod-shape intertwined structure, which allows the material to maintain a large specific surface area while preserving its structural stability. The nanocomposite demonstrates a high photocatalytic activity in the degradation of methylene blue (MB), ofloxacin antibiotics (OFL), and 5% cis–trans cypermethrin emulsion (5% CTC). The heterostructure of the Zn<sub>2</sub>SnO<sub>4</sub>/ZnO effectively inhibited the electron–hole recombination, greatly improved the activity and stability of the catalyst, and effectively promoted the catalytic reaction.

**Keywords** Zn<sub>2</sub>SnO<sub>4</sub>/ZnO · Nanocomposites · Heterostructure · Photocatalytic activity

## Introduction

Pesticide is a substance or mixture of substances used for controlling, preventing, destroying, or reducing the number of pests [1]. It can cause soil, water, and air pollution [2] and harm human [3]. Of particular concern are the widespread use of pesticides and their potential to enter water supplies and cause negative effects on public health [4]. Health care is regarded as a fundamental determining factor in development of general, physical, and mental well-being of people round the globe [5]. At the same time, a number of drugs and pharmaceuticals are emerging as serious environmental contaminants [6]. Fluro-quinolones are widely administered because of their broad-spectrum activity and exceptional tissue permeation [7]. Belonging to this class of drugs, the antibiotic ofloxacin is effective against a number of commonly occurring bacterial

infections. However, its wide usage and administration are also responsible for serious contamination and health problems [8, 9]. Because of persistent and stable nature of such compounds and their metabolites, it is required to degrade such compounds by efficient degradation technologies such as heterogeneous photocatalysis [10, 11]. Most of the organic dyes and their effluents in the textile industries are toxic, carcinogenic, and have become one of the major sources of water pollution [12–14]. There are many types of wastewater treatment technologies, among which the commonly seen ones include adsorption [15], microwave catalysis, and photocatalysis. Adsorption technology is widely applicable due to its high efficiency and simplicity of operation [16]. However, after the adsorption saturation, the adsorbent needs to be regenerated or replaced [17, 18], which increases the treatment cost. Microwave catalysis technology is efficient and rapid, often more energy-efficient than traditional heating methods [19]. But the initial investment in equipment is relatively high, and not all materials are suitable for microwave catalysis, which requires specific catalysts or carriers. The semiconductor-based photocatalysis has opened up for elimination of the contaminant from the environment and has become an interesting area of research [20]. The advantage of photocatalytic technology is utilizing light energy as

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the driving force without generating secondary pollution, so that organic matter can be decomposed into harmless small molecules [21, 22].

Recently, tremendous efforts have been devoted to fabricate high-performance photocatalyst. A large number of inorganic semiconductor materials, especially metal oxides such as TiO<sub>2</sub> [23], ZnO [24], V<sub>2</sub>O<sub>5</sub>, BiOBr [25], CuO, Bi<sub>2</sub>MoO<sub>6</sub>/g-C<sub>3</sub>N<sub>4</sub> [26], and metal sulfides such as Cu<sub>2</sub>S and ZnS have been explored as photocatalyst under either UV light or visible light irradiation for this application [27]. Owing to the advantages of low cost, chemical stability, and environmental compatibility, ZnO has been widely investigated as a photo-catalyst. However, one of the main shortages for pure ZnO is that photon-generated carriers are easily recombined [28]. A large number of approaches have been reported to overcome this problem. Among them, compounded by two substances is an effective way for enhancing the photocatalytic activity [29]. Construction of semiconductor heterostructures has been as an effective strategy to enhance the photocatalytic activity [30, 31].

As important functional materials, ZnO and Zn<sub>2</sub>SnO<sub>4</sub> with band gaps of 3.2 and 3.4 eV, respectively, have been intensively investigated due to their unique properties and great potential applications such as gas sensors [32], solar cells [33], and photocatalytic degradation [34]. Pure Zn<sub>2</sub>SnO<sub>4</sub> typically exhibits a longer degradation time and lower efficiency when it was used for photocatalytic degradation of organic dyes such as water-soluble dyestuffs [35] and Methyl Orange (MO) [36]; the degradation rate of water-soluble dyes reached 90% in 2 h, while it took 1.5 h to degrade MO with the degradation rate of 80.3%. However, the photocatalytic performance of pure Zn<sub>2</sub>SnO<sub>4</sub> can be significantly improved by coupling it with ZnO to form a Zn<sub>2</sub>SnO<sub>4</sub>/ZnO composite photocatalyst. It was found that the degradation rate of Zn<sub>2</sub>SnO<sub>4</sub>/ZnO was three times than that of pure Zn<sub>2</sub>SnO<sub>4</sub> [37]. When Zn<sub>2</sub>SnO<sub>4</sub> is coupled with ZnO, the heterostructure will accelerate the photogenerated electrons and holes separation and thus enhance the photocatalytic efficiency, but the photocatalytic efficiency widely depends on the surface area and the porosity of the nanostructures [38]. Although Zn<sub>2</sub>SnO<sub>4</sub>/ZnO composite photocatalysts have been widely studied and verified in terms of organic dye degradation, it remains relatively limited that the research on their effectiveness in degrading environmental pollutants such as pesticides and antibiotics. The widespread use of pesticides and antibiotics poses a serious threat to the environment and human health. Therefore, it is significant in exploring the potential of Zn<sub>2</sub>SnO<sub>4</sub>/ZnO composite photocatalysts in the degradation of these types of pollutants. In this work, the Zn<sub>2</sub>SnO<sub>4</sub>/ZnO heterostructured composite was prepared by a simple one-step hydrothermal method. It was studied the Zn<sub>2</sub>SnO<sub>4</sub>/ZnO heterostructure effect on the

pollutant's degradation (methylene blue, 5% cis-cypermethrin emulsion and ofloxacin antibiotic).

## Experiment sections

### Materials and method

In this work, ZnSO<sub>4</sub>·7H<sub>2</sub>O (Aladdin, CP, grade 99.0%), SnCl<sub>4</sub>·5H<sub>2</sub>O (Aladdin, AR, grade 99.0%), NaOH (Aladdin, AR grade 99.0%), methylene blue (Aladdin, AR grade 99.0%), 5% cis–trans cypermethrin emulsion (Aladdin, AR grade 95.0%), ofloxacin antibiotics (Aladdin, CP, grade 99.0%), anhydrous ethanol, deionized water, and all other reagents used were analytically pure. Firstly, 1 mmol ZnSO<sub>4</sub>·7H<sub>2</sub>O and 0.5 mmol SnCl<sub>4</sub>·5H<sub>2</sub>O were added to 40 mL deionized water by stirring for 30 min. Then, 15 mmol NaOH was added to the above solution and stirred for 20 min. Finally, the above mixture was transferred to a 100-mL Teflon lined stainless steel autoclave and heated at 200 °C for 12 h. After completion, the white precipitates were collected by centrifuging at 4000 rpm and then washed for three times with deionized water and ethanol and dried at 80 °C.

### Characterization

To examine the crystal quality and crystallization state of the products, the as-prepared sample was investigated by using X-ray diffraction (XRD-6100, CuKα, 40 kV). The morphologies and microstructures were characterized by scanning electron microscopy (SEM, JEOL-2100) and transmission electron microscopy (TEM, JEOL-2010). The specific surface area and pore size distribution were tested by an automatic gas adsorption analyzer (Auto Sorb-IQ). The infrared spectra were recorded at room temperature on a Fourier transform infrared spectrophotometer (IR Prestige-21) as KBr pellets in the 3500 ~ 500 cm<sup>-1</sup> region.

### Photocatalytic measurement

The photocatalytic activities of the products for the degradation of methylene blue (10 mg/L), 5% cis–trans cypermethrin emulsion (1 ml/L) and ofloxacin antibiotic (25 mg/L, pH=7.0) were evaluated by using photochemical reactor (QFN-GHX-I), mercury lamp light source (GY500, wavelength=315~450 nm), and UV/Vis spectrometer (Purkinje, TU-1810).

0.05 g of the as-prepared product was added in 100 mL solutions. The mixtures were continuously stirred for 1 h in the dark to ensure adsorption/desorption equilibrium. And then, the solution was exposed to UV irradiation using a 500-W mercury lamp. The solution was collected at regular

intervals to measure the dye-degradation using UV/Vis spectroscopy.

## Results and discussion

### Microstructure and morphology representation

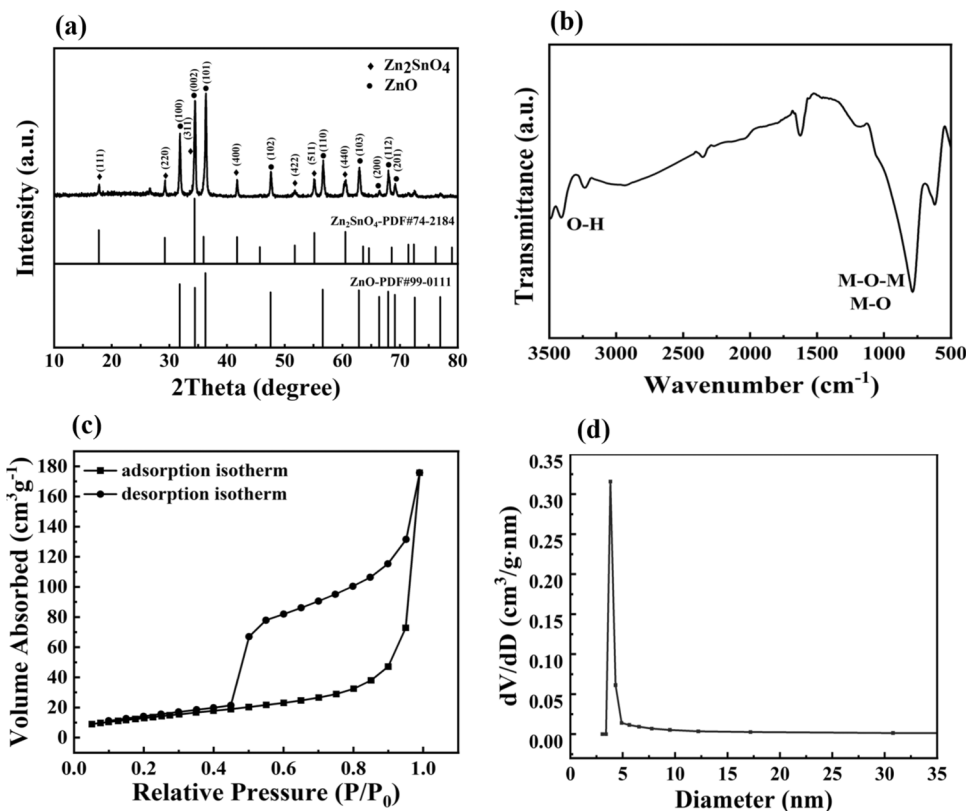
The crystal structure and phase analysis of the synthesized products were investigated by XRD. As shown in Fig. 1a, all the observed diffraction peaks were consistent with the characteristic peaks of Zn<sub>2</sub>SnO<sub>4</sub> and ZnO, and no other impurity peaks were detected. The diffraction peaks at 17.7°, 29.1°, 34.3°, 41.7°, 51.7°, 55.1°, and 60.4° correspond to the (111), (220), (311), (400), (422), (511) and (440) crystal planes of tetragonal Zn<sub>2</sub>SnO<sub>4</sub> (PDF#74–2184). The diffraction peaks at 31.7°, 34.4°, 36.2°, 47.5°, 56.6°, 62.8°, 66.3°, 67.9°, and 69.0° corresponded to the (100), (002), (101), (102), (110), (103), (200), (112), and (201) crystal planes of the inverse spinel structure ZnO (PDF#89–0511), indicating that the product in this experiment was pure Zn<sub>2</sub>SnO<sub>4</sub>/ZnO. All characteristic peaks indicate that the Zn<sub>2</sub>SnO<sub>4</sub>/ZnO is mainly in pure spinel structure without contaminated composition.

Figure 1b is the Fourier transform infrared spectroscopy, and there are obvious absorption bands in the range of 3200~3500 cm<sup>-1</sup> and 1400~1700 cm<sup>-1</sup>. This corresponds to the stretching vibration absorption of the hydroxyl group

(O–H) of the adsorbed water on the surface of the material. This is attributed to the large amount of hydroxyl groups and adsorbed water adsorbed on the surface of the powder. During the photocatalytic process, the photocatalyst is excited by incident photons, generating photo-generated electrons and holes. The adsorbed hydroxyl groups act as electron acceptors, effectively capturing the photo-generated electrons and suppressing their recombination with holes. This electron capture process not only prolongs the lifetime of photo-generated charge carriers but also leads to the formation of reactive species with strong oxidizing properties. These reactive species, in turn, efficiently degrade organic pollutants, thereby enhancing the overall degradation efficiency of the photocatalytic reaction. The absorption band in the range of 500~700 cm<sup>-1</sup> can be attributed to the stretching vibration absorption of typical metal oxides (M–O or M–O–M, M represents Zn or Sn). In addition, the absorption band centered at 1200 cm<sup>-1</sup> is attributed to the symmetric and asymmetric stretching vibrations of Zn–O–Sn. Therefore, the presence of these functional groups proves the existence of the Zn<sub>2</sub>SnO<sub>4</sub>/ZnO.

The results of nitrogen adsorption and desorption experiments of the Zn<sub>2</sub>SnO<sub>4</sub>/ZnO are shown in the Fig. 1c and d. According to the classification of the International Union of Pure and Applied Chemistry (IUPAC), the curve is a type IV isotherm, indicating that porous layer adsorption occurs. The isotherms obtained during the desorption

**Fig. 1** a XRD pattern, b infrared spectrogram, c nitrogen adsorption/desorption isotherm, and d pore diameter distribution of the Zn<sub>2</sub>SnO<sub>4</sub>/ZnO



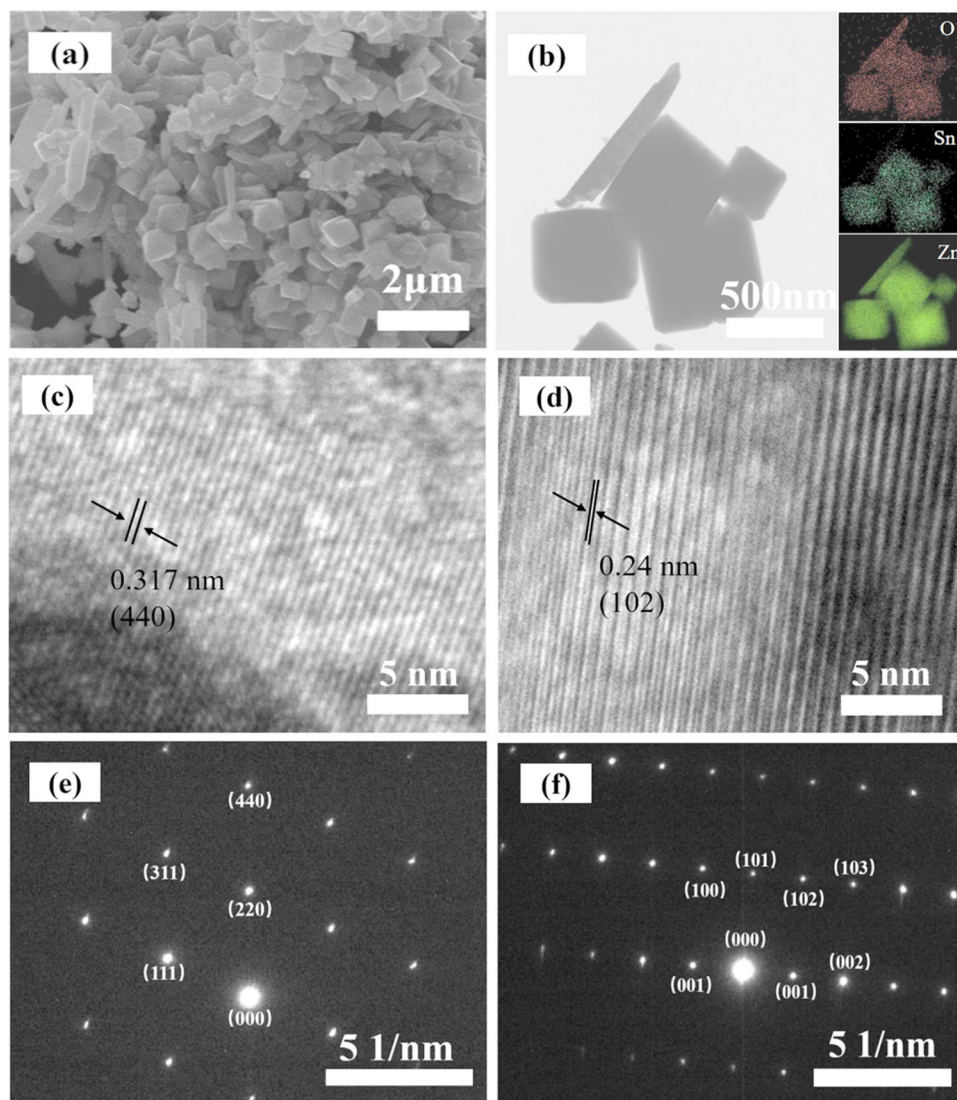
process are inconsistent with the isotherms obtained during the adsorption process. The desorption isotherm is higher than the adsorption isotherm, resulting in desorption hysteresis, showing an H3 hysteresis loop on the BJH isotherm [39]. The results show that the as-prepared  $\text{Zn}_2\text{SnO}_4/\text{ZnO}$  nanomaterials have mesopore structure. According to the BJH pore size distribution curve, there is a peak at 3.82 nm, indicating that the pore size distribution is concentrated at 3.82 nm. The specific surface areas of the  $\text{Zn}_2\text{SnO}_4/\text{ZnO}$  are obtained with  $225.0 \text{ m}^2/\text{g}$ .

The morphological information of the  $\text{Zn}_2\text{SnO}_4/\text{ZnO}$  is shown in Fig. 2a and b. It is evident that the  $\text{Zn}_2\text{SnO}_4/\text{ZnO}$  is composed of many nanocubes and nanorods. The crystal structures of  $\text{Zn}_2\text{SnO}_4$  and ZnO are intertwined to form a unique heterojunction structure. This intertwined material exhibits excellent physical and chemical properties due to its large structural area, which facilitates the transportation and separation of charge carriers. The EDS results show that

the rod-like structure predominantly features the elemental distributions of Zn and O, whereas the cubic structure demonstrates the presence of Sn, Zn, and O, which follows that  $\text{Zn}_2\text{SnO}_4$  takes on a cubic shape, while ZnO takes on a rod shape. Elemental mapping images indicate a uniform distribution of the three elements Sn, Zn, and O that constitute  $\text{Zn}_2\text{SnO}_4$  and ZnO, without any compositional segregation.

In order to study the detailed structure of the  $\text{Zn}_2\text{SnO}_4/\text{ZnO}$ , TEM measurement was further conducted, as shown in Fig. 2c–f. The high-resolution images shown in Fig. 2c and d allow for a clear identification of well-aligned lattice fringes. The interplanar distance of 0.317 nm corresponding to the (440) planes of  $\text{Zn}_2\text{SnO}_4$  are indicated in Fig. 2c, while the interplanar distance of 0.24 nm corresponding to the (102) planes of ZnO is indicated in Fig. 2d. The SAED images shown in Fig. 2e and f reveal clear single-crystal electron diffraction patterns for the sample, indicating a single-crystal structure.

**Fig. 2** a Scanning electron microscopy, b transmission electron microscopy, c, d interstitial void, and e, f selected area electron diffraction of the  $\text{Zn}_2\text{SnO}_4/\text{ZnO}$ .



### Photocatalytic degradation activity

The photocatalytic activity of the as-prepared Zn<sub>2</sub>SnO<sub>4</sub>/ZnO was verified by photocatalytic degradation of MB, OFL, and 5% CTC, as shown in Fig. 3.

The main peak at 664 nm, 287 nm, and 194 nm are the characteristic absorption peak of MB, OFL, and 5% CTC, respectively. The peak height is proportional to the concentration of the solution. With the increase of illumination time, the color of MB solution gradually became lighter. At the same time, the unique pungent taste of 5% CTC gradually disappeared, and the OFL gradually changed from turbidity to clarification. The intensity of the characteristic absorption peak in the spectrum gradually weakened,

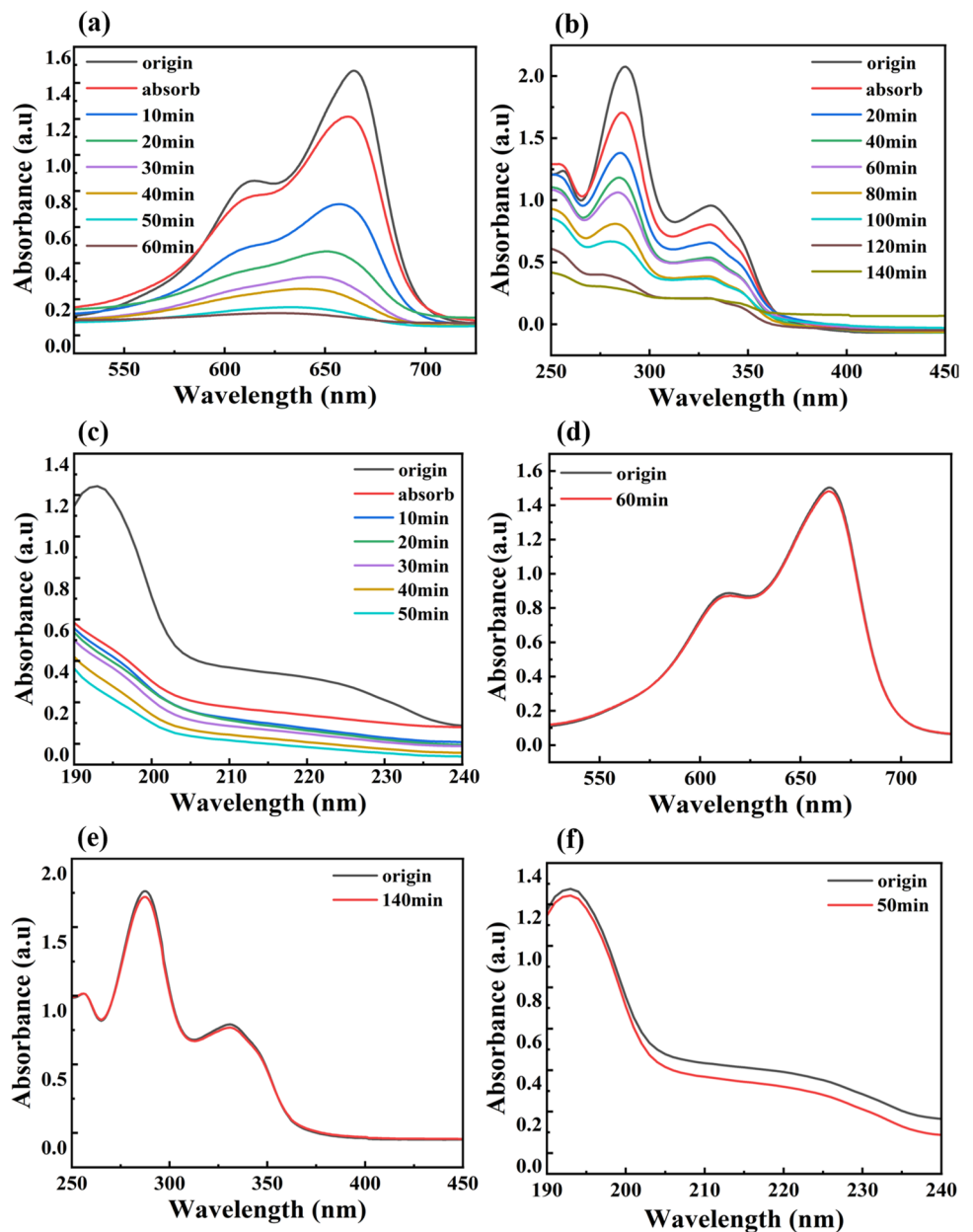
indicating that the solution was degraded under the action of nanomaterial photocatalyst. The degradation rate can be calculated by the following formula:

$$I = \left(1 - \frac{C}{C_0}\right) \times 100\%$$

In the equation, *I*, *C*, and *C*<sub>0</sub> are the degradation rate, the real-time concentration under UV irradiation, and the initial concentration of organic dye, respectively. When calculating the final degradation rate, the *C* value of the solution with the longest illumination time was taken.

Figure 3a–c show the UV-absorbance spectra of Zn<sub>2</sub>SnO<sub>4</sub>/ZnO for degradation of MB, 5% CTC, and OFL. The

**Fig. 3** Absorption spectra of several solutions in the presence of Zn<sub>2</sub>SnO<sub>4</sub>/ZnO photocatalyst under UV-light irradiation for different time intervals. **a** MB. **b** OFL. **c** 5% CTC. **d–f** Results of photocatalytic experiments without Zn<sub>2</sub>SnO<sub>4</sub>/ZnO nano-materials



degradation rates for MB, OFL, and 5% CTC are 93.45%, 83.14%, and 86.54%, and the degradation times are 60, 140, and 50 min, respectively. The photocatalytic performance of the as-prepared  $\text{Zn}_2\text{SnO}_4/\text{ZnO}$  was better than of the reported oxide semiconductor materials [40–47] as shown in Table 1. The photocatalytic activity of the bare ZnO and bare  $\text{Zn}_2\text{SnO}_4$  was compared with that of  $\text{Zn}_2\text{SnO}_4/\text{ZnO}$ . It can be seen that the degradation rate of MB by ZnO is only 48.3%, and the degradation rate of MB by  $\text{Zn}_2\text{SnO}_4$  is 66.1% after 60 min as shown in Fig. 2 s, which are both lower than the 93.45% degradation rate of  $\text{Zn}_2\text{SnO}_4/\text{ZnO}$ . For the reported  $\text{Zn}_2\text{SnO}_4/\text{ZnO}$  photocatalyst [48, 49], the degradation rate for MB reached 91.5% and 60% after 120 min and 140 min, respectively. In contrast, the as-prepared  $\text{Zn}_2\text{SnO}_4/\text{ZnO}$  photocatalyst exhibits significantly superior performance in degrading MB, which the  $\text{Zn}_2\text{SnO}_4/\text{ZnO}$  achieves a higher degradation rate of 93.45% for MB after 60 min.

Generally,  $\text{TiO}_2$  P25 is the most applied as UV light photocatalyst. In order to compare the photocatalytic performance of the as-prepared  $\text{Zn}_2\text{SnO}_4/\text{ZnO}$  with  $\text{TiO}_2$  P25, the supplementary data on the degradation activity of MB, OFL, and 5% CTC by  $\text{TiO}_2$  P25 as shown in Fig. 3s. The degradation rates for MB, OFL, and 5% CTC are 79.65%, 31.86%, and 55.31%, and the degradation times are 60, 140, and 50 min, respectively. It was found that the photocatalytic performance of  $\text{TiO}_2$  P25 was lower than that of the  $\text{Zn}_2\text{SnO}_4/\text{ZnO}$  heterostructure composite.

To demonstrate that the  $\text{Zn}_2\text{SnO}_4/\text{ZnO}$  is the primary agent of degradation, this work conducted an experiment without adding the  $\text{Zn}_2\text{SnO}_4/\text{ZnO}$  as shown in Fig. 3d–f.

**Table 1** Comparison of photocatalytic activities of reported oxide semiconductor materials used for photocatalytic degradation of MB, OFL, and 5% CTC

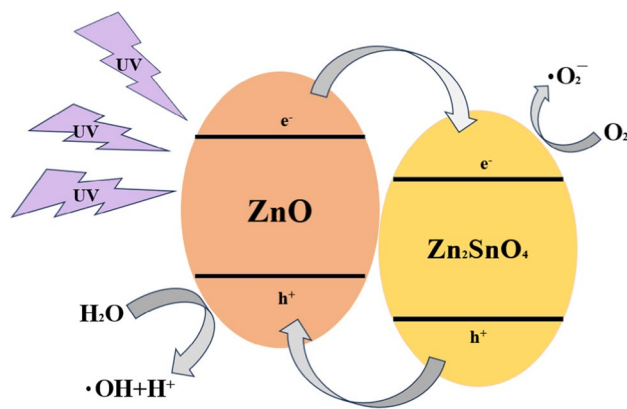
Catalyst	Source	Time/min	Degradation/%	Liquor	References
$\text{Sn}_3\text{O}_4$	Visible	180	33.35	MB	40
Ag-AgO/ $\text{Al}_2\text{O}_3$	UV	90	92	MB	41
FeTi	UV	60	85	5%CTC	42
Bi/Ni- $\text{TiO}_2$	UV	360	70	OFL	43
$\text{Bi}_2\text{S}_3/\text{Bi}_2\text{WO}_6$	Visible	180	87	OFL	44
$\text{Bi}_2\text{MoO}_6$	Visible	90	71	OFL	45
$\text{Zn}_2\text{SnO}_4$	UV	60	87.3	MB	46
ZnO	UV	120	18.6	MB	47
$\text{Zn}_2\text{SnO}_4/\text{ZnO}$	UV	120	91.5	MB	48
$\text{Zn}_2\text{SnO}_4/\text{ZnO}$	UV	140	60	MB	49
$\text{Zn}_2\text{SnO}_4$	UV	60	66.1	MB	This work
ZnO	UV	60	48.3	MB	This work
$\text{Zn}_2\text{SnO}_4/\text{ZnO}$	UV	60	93.4	MB	This work
$\text{Zn}_2\text{SnO}_4/\text{ZnO}$	UV	140	86.5	OFL	This work
$\text{Zn}_2\text{SnO}_4/\text{ZnO}$	UV	50	83.1	5%CTC	This work

The characteristic absorption peak intensities of the MB, OFL, and 5% CTC were only slightly reduced. This finding indicates that UV light itself has minimal degradation properties.

Photocatalytic mechanism of the  $\text{Zn}_2\text{SnO}_4/\text{ZnO}$  is shown in Fig. 4. The conduction band (CB) and valence band (VB) potential values of ZnO and  $\text{Zn}_2\text{SnO}_4$  are  $-0.803$  eV,  $2.417$  eV and  $-0.993$  eV,  $2.217$  eV, respectively [50]. When  $\text{Zn}_2\text{SnO}_4/\text{ZnO}$  is irradiated with UV light,  $\text{Zn}_2\text{SnO}_4$  and ZnO can be both excited.  $\text{Zn}_2\text{SnO}_4/\text{ZnO}$  generates equal numbers of photogenerated electrons and holes. After  $\text{Zn}_2\text{SnO}_4$  absorbs UV light, electrons transfer from valence band to conduction band, causing that photogenerated electron hole pairs are produced. Because the conduction band and valence band potential values of  $\text{Zn}_2\text{SnO}_4$  are both lower than those of ZnO, photogenerated holes transfer from valence band of  $\text{Zn}_2\text{SnO}_4$  to valence band of ZnO. Then, photogenerated electrons transfer from conduction band of ZnO to conduction band of  $\text{Zn}_2\text{SnO}_4$ . This greatly increases the probability of photogenerated holes and electrons migrating to the photocatalyst surface. Subsequently, the holes react with  $\text{H}_2\text{O}$  to form hydroxy radicals ( $\cdot\text{OH}$ ), while the superoxide anion radicals ( $\cdot\text{O}_2^-$ ) are formed by the reaction of excited electrons with absorbed oxygen. The organic dyes can be degraded by  $\cdot\text{OH}$  and  $\cdot\text{O}_2^-$  validly.

The  $\text{Zn}_2\text{SnO}_4/\text{ZnO}$  has good photocatalytic performance and conforms to the heterojunction transfer mode. The reasons for its good photocatalytic performance are as follows: On the one hand, the composite structure enhances the surface adsorption between the photocatalyst and the organic molecule. On the other hand, the  $\text{Zn}_2\text{SnO}_4/\text{ZnO}$  heterojunction promotes the transfer and separation of photogenerated electrons and holes, and greatly improves the photocatalytic activity.

In order to evaluate the stability and reusability of the photocatalyst, MB as a representative organic dye was chosen to degrade using recycling degradation experiment. After



**Fig. 4** Schematic diagram of charge transfer

the end of the first photocatalytic experiment,  $Zn_2SnO_4/ZnO$  was recovered by centrifugation and calcination, then carried out the photocatalytic degradation experiment again, which was repeated for 5 times. As shown in Fig. 4s, the photocatalytic degradation rates of the five experiments were 93.58%, 93.45%, 93.34%, 93.23%, and 93.16%, respectively. This reveals that there is no clear change in the degradation rate after 5 cycles, which indicates that the heterojunction system could enhance the stability of  $ZnO$ .

## Conclusions

In summary, the  $Zn_2SnO_4/ZnO$  composites were successfully synthesized by a one-step hydrothermal method. The detailed characterizations reveal that the as-prepared  $Zn_2SnO_4/ZnO$  possesses pure phase, good crystallinity, and excellent photocatalytic activity for the degradation; the degradation rates for MB, OFL, and 5% CTC are 93.45%, 83.14%, and 86.54%, respectively. The as-prepared photocatalysts might have a potential application in water purification.

**Supplementary Information** The online version contains supplementary material available at <https://doi.org/10.1007/s11581-024-05553-x>.

**Author contributions** Wenlei Wang has done the whole work and written the entire manuscript. Wenquan Hu and Ming Wang have corrected the entire manuscript and supported the research funding. The photocatalytic experiments have been done by Zhikang Chen and Qi Chen.

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## Declarations

**Competing interests** The authors declare no competing interests.

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