β-FeSe nanorods composited g-C3N4 with enhanced photocatalytic efficiency
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A series of β-FeSe nanorods composited g-C3N4 were prepared. The structure, morphology, chemical state, photocatalytic activity, electrochemical impedance and photoluminescence of β-FeSe/g-C3N4 composites were well characterized. It is found that the decolourization rate of 3 wt% β-FeSe/g-C3N4 composites reaches 4.4 times than that of g-C3N4. The improved photocatalytic properties could be ascribed to the reduced recombination of photogenerated electrons and holes, which is derived from the excellent ability of β-FeSe to capture and transfer electrons. This work provides an alternative co-catalyst for decolourizing organic matter.

1. Introduction

The energy crisis and the increasingly serious environmental pollution problem are the severe challenges facing human survival and development [1–4]. Today, with the depletion of fossil energy, the use of clean solar energy has become an alternative solution. In this regard, photocatalyst plays an important role in harvesting solar energy, which can convert solar energy into chemical energy and use sunlight to degrade organic pollutants. Since the 1970s, TiO2 has been used as a photocatalyst to split water [5–7]. Nowadays, more semiconductor materials, such as ZnO [8], SrTiO3 [9] and CdS [10] were developed as photocatalysts. Among them, g-C3N4 stands out for its wider absorption spectrum and higher efficiency in activating molecular oxygen into superoxide radicals [11–13]. Nevertheless, the performance of g-C3N4 is still insufficient for its bulk structure and the high carrier recombination probability. Based on it, there are usually two approaches to enhance the photocatalytic activity, one of which is nanostructure design with more active sites. As far as we know, various nanostructured g-C3N4, like nanoparticles, nanospheres, nanorods, nanowires and particularly nanosheets have been developed for photocatalysts with higher...
activity [13]. Moreover, g-C3N4 coupled with semiconductors and noble metal nanoparticles can also improve photocatalytic activity [14,15]. In the photocatalytic process, photogenerated electrons and holes undergo two types of reactions, one for driving photocatalytic reactions and the other for recombination. In fact, the latter tends to dominate even though it is harmful to photocatalysis. Therefore, the effective separation of photogenerated carriers to avoid recombination is particularly important. The aforementioned composite materials offer enhanced migration efficiency of photogenerated electrons and holes, and hence suppressed recombination. As for noble metal composites, Au, [16] Ag [17] and Pt [18] have been used to couple with g-C3N4.

FeSe has two crystalline states, a hexagonal phase (α-FeSe) and a tetragonal phase (β-FeSe) [19]. Among them, β-FeSe exhibits metallic behaviour above \( T_c = 8 \) K and becomes a superconductor below that temperature [19]. The room-temperature resistivity of β-FeSe is about 1 mΩ cm [20]. β-FeSe has a layered structure, which consists of a quasi-two-dimensional layer composed of edge-sharing FeSe\(_4\) tetrahedra stacking along the \( c\)-axis. Intercalating metal ions or even neutral molecules into [Fe\(_2\)Se\(_2\)] layers will transfer electrons to [Fe\(_2\)Se\(_2\)] layer, [21–24] which indicates that β-FeSe has excellent ability to capture electrons. Moreover, β-FeSe nanoparticles, [25] nanoflakes [26] and nanorods [27,28] have been synthesized through various preparation methods.

Considering that the recombination of photogenerated carriers is the main reason for hindering the photocatalytic performance of g-C3N4, the composition of co-catalyst such as semiconductors and noble metal nanoparticles can promote the separation of photogenerated carriers and improve the photocatalytic efficiency. β-FeSe does not contain precious metal elements and is a potential electron capturer as mentioned above. It is a fascinating question whether g-C3N4 composites with β-FeSe nanorods has excellent photocatalytic activity. Here, we prepared a series of β-FeSe nanorods composited g-C3N4 and studied their photocatalytic properties for decolourizing Rhodamine B (RhB). The photocatalytic activity of g-C3N4 is greatly improved after the composition. This work provides a promising co-catalyst for photocatalysis.

2. Experimental set-up

2.1. Materials

RhB (analytical grade), H\(_2\)O\(_2\) (30 wt%, analytical grade) and Na\(_2\)SO\(_4\) (99%) were obtained from Innochem. Urea (99.999%) and Nafion solution (5 wt%) were provided by Aladdin. Iron pieces (99.99%) and selenium shots (99.999%) were purchased from Alfa Aesar.

2.2. Preparation of β-FeSe/g-C3N4 composites

Firstly, g-C3N4 was prepared through high-temperature pyrolysis of urea [29]. About 20 g of urea is contained to the three-quarter height position of the crucible, which was covered by a lid and was sintered at 550°C for 2.5 h. The synthesis of β-FeSe nanorods is described as follows. β-FeSe crystals were synthesized following the method described in [30]. The iron pieces and selenium shots weighed in a nominal ratio were sealed in a quartz tube, which was placed in a muffle furnace at 750°C, kept for 5 days and then heated to 1075°C for 3 days. It was then quickly transferred to a muffle furnace with 420°C, kept for 2 days and then quenched in liquid nitrogen. The obtained β-FeSe crystals were ground into powders using a mortar and then dispersed in absolute ethanol, which was sonicated for 2 h in a high-power ultrasonic instrument. Finally, β-FeSe nanorods were obtained by centrifugal separation. β-FeSe nanorods and g-C3N4 at a ratio of 1, 3, 5 and 10 wt% were dispersed in absolute ethanol. The mixture was sonicated for 2 h to achieve uniform mixing. Then they were dried at 80°C to evaporate the solvent. The obtained sample was further sintered at 150°C for 5 h to get well-joined β-FeSe /g-C3N4 composites.

2.3. Characterization

X-ray diffraction (XRD) data were measured by a PANalytical X’pert Pro diffractometer using Cu target radiation. The morphologies were identified by scanning electron microscopy (SEM, Hitachi S-4800). The X-ray photoelectron spectroscopy (XPS) data were collected on a Thermo ESCALAB 250 Xi system. The photoluminescence spectra (PL) were recorded by a Hitachi F-4600 fluorescence spectrometer. The electrochemical impedance spectroscopy (EIS) was measured as follows. Firstly, 5 mg of the 3 wt%
β-FeSe/g-C3N4 composites and 10 μl of 5 wt% Nafion solution were mixed homogeneously in ethanol with 1 ml. The obtained paste was spread on indium tin oxide conductive glass, which was kept at 200°C for 1 h and then used as the working electrode. Moreover, the counter electrode was made of a platinum foil, the reference electrode was made of a saturated Ag/AgCl electrode, and the electrolyte was 0.5 M Na2SO4 solution. The EIS measurements using the above three-electrode cells were conducted on a CHI 660C electrochemical workstation.

### 2.4. Photocatalytic properties

The photocatalytic properties were characterized by decolourizing RhB on a Shimadzu UV-2450 spectrophotometer using a Xe lamp of 300 W with a filter having a cut-off wavelength of 420 nm under visible light irradiation. In this experiment, 50 mg of β-FeSe/g-C3N4 composites were added to 50 ml RhB solution, which was stirred continuously for 90 min in dark.

### 3. Results and discussion

The crystal structure of β-FeSe is shown in figure 1a. It consists of a quasi-two-dimensional layer composed of edge-sharing FeSe4 tetrahedra stacking along the c-axis [19]. The XRD patterns of as-prepared materials are displayed in figure 1b. A strong diffraction peak at 27.6°, which corresponds to the (002) reflection, the typical characteristic of g-C3N4, can be observed [31]. From it, the distance between the stacking layers of the graphitic structure can be derived to be 0.33 nm, which is consistent with the reported g-C3N4 [32]. The diffraction peaks of β-FeSe can be recognized for 10 wt% β-FeSe/g-C3N4 composites, as shown in figure 1b.

SEM characterization was conducted to obtain the morphology features of the samples. Figure 2a displays the SEM image of β-FeSe, from which β-FeSe nanorods could be clearly recognized. The nanorods have diameters of about 30 nm and lengths between 0.3 and 1.2 μm. Figure 2b displays the morphology of g-C3N4. It consists of small pieces of uneven particle size with the order of micrometres (figure 2b). As exhibited in figure 2c,d, β-FeSe nanorods are dispersed on the outer surface and embedded inside of g-C3N4.

Figure 3 displays the XPS spectra of 3 wt% β-FeSe/g-C3N4 composites. The survey spectrum in figure 3a displays that there are C, Se, N, O and Fe in the composites. The signals of Fe and Se are weak because of their extremely low content. The peaks of 284.64 and 287.98 eV belong to C 1s. Among them, the former is ascribable to graphitic carbon [33–35] and the peak located at 287.98 eV is derived from sp2-hybridized carbon (N–C=N) [29]. The spectrum of N 1s in figure 3c can be fitted with three peaks. Among them, the peak of 398.22 eV is due to C=N–C, [36] the peak of 398.80 eV is ascribed to N-(C)3 bond, [36] and the peak located at 400.24 eV corresponds to N-H bond [37]. The Se 3d spectrum (figure 3d) consists of two peaks at 55.22 and 59.61 eV for Se 3d5/2 and 3d3/2, respectively [27]. The Fe 2p spectrum (figure 3e) can be fitted with the peaks at 710.11 and 723.77 eV, which is consistent with the results of Fe 2p in the literature [28,38].

![Figure 1](https://example.com/fig1.png)  
*Figure 1. (a) Schematic diagram of the crystal structure of β-FeSe. (b) XRD patterns of as-prepared materials.*
The photocatalytic properties of as-prepared materials were evaluated by degrading RhB dyes. Figure 4a shows the dark adsorption of RhB on g-C3N4 and 3 wt% β-FeSe/g-C3N4 composites. It can be seen that RhB is greatly adsorbed by the samples. As shown in figure 4b, after exposure to visible light for 180 min, the concentration of RhB was still 90% of the original for g-C3N4, while the decolourization efficiency is greatly enhanced for β-FeSe/g-C3N4 composites. The optimized ratio is 3 wt% β-FeSe/g-C3N4 composites. Further increase in the β-FeSe content leads to a lower decolourization efficiency, which is due to excess β-FeSe shielding the light that reaches the g-C3N4 surface and thus affecting the absorption of light. Moreover, the reaction kinetics of RhB
decolourization can be fitted by the first-order reaction kinetics \( \ln \left( \frac{C_0}{C} \right) = kt \) when \( C_0 \) is of the order of millimolar, where \( C_0 \) is the concentration at which RhB reaches the equilibrium of absorption and desorption in the dark, \( C \) is the concentration under visible light and \( k \) is the first-order reaction rate constant. The results of photocatalytic kinetics are shown in figure 4c. The \( k \)-value is calculated to be 0.00077, 0.0022, 0.0034 and 0.0029 \( \text{min}^{-1} \) for g-C\(_3\)N\(_4\), 1, 3 and 5 wt% \( \beta \)-FeSe/g-C\(_3\)N\(_4\) composites. So the decolourization rate of 3 wt% \( \beta \)-FeSe/g-C\(_3\)N\(_4\) composites reaches 4.4 times than that of g-C\(_3\)N\(_4\).

To further improve the photocatalytic efficiency, a low concentration of H\(_2\)O\(_2\) (103 \( \mu \)l /100 ml) was used as an efficient scavenger adding in the solution. It can be seen from figure 5a that the photocatalytic efficiency of 3 wt% \( \beta \)-FeSe/g-C\(_3\)N\(_4\) composites with H\(_2\)O\(_2\) is greatly improved. The RhB in solution is completely decomposed in 60 min. The improved photocatalytic performance can be

![Figure 4](image1.png)

**Figure 4.** (a) The dark adsorption of RhB on g-C\(_3\)N\(_4\) and 3 wt% \( \beta \)-FeSe/g-C\(_3\)N\(_4\) composites. (b,c) Visible light irradiation photocatalytic activities of as-prepared materials for degrading RhB.

![Figure 5](image2.png)

**Figure 5.** (a) Photocatalytic activities of decolourization of RhB under visible light for as-prepared materials. (b) Cyclic performance of 3 wt% \( \beta \)-FeSe/g-C\(_3\)N\(_4\) composites with H\(_2\)O\(_2\).
explained as follows. On the one hand, H₂O₂ may undergo photolysis by visible light and generate \( \cdot \text{OH} \) radicals, on the other hand, H₂O₂ can capture the photogenerated electrons to form \( \cdot \text{OH} \) radicals, which are the main contributors to the photocatalytic process [39–41], thus further enhancing photocatalytic activity. The cyclic performance of 3 wt% \( \beta \)-FeSe/g-C₃N₄ composites is shown in figure 5b. The photodecolourization rate is almost unchanged after four cycles, manifesting the stability of this photocatalyst.

EIS measurement was performed to obtain the reason for the improvement of photocatalytic performance for 3 wt% \( \beta \)-FeSe/g-C₃N₄ composites. As shown in figure 6a, the 3 wt% \( \beta \)-FeSe/g-C₃N₄ composites show smaller arc radius than that of g-C₃N₄, indicating the former has a smaller charge-transfer resistance than g-C₃N₄ and has faster interfacial charge-transfer process [42]. Moreover, photoluminescence spectrum, which is often conducted to understand the charge separation efficiency, [43] was performed. As shown in figure 6b, the intensity of photoluminescence decreases for 3 wt% \( \beta \)-FeSe/g-C₃N₄ composites. It manifests that the recombination of photogenerated carriers decreases for the former.

As discussed above, a possible mechanism of improved photocatalytic efficiency for \( \beta \)-FeSe/g-C₃N₄ composites is given as shown in figure 7. Under visible light, electrons of g-C₃N₄ are excited, which are rapidly transferred to \( \beta \)-FeSe for its excellent ability to capture electrons. The photogenerated electrons then react with \( \text{O}_2 \) to produce superoxide radical anion \( \text{O}_2^- \). The \( \text{O}_2^- \) react with water and generate \( \cdot \text{OH} \), which degrade RhB to be \( \text{CO}_2 \) and \( \text{H}_2\text{O} \) [39–41]. Moreover, photogenerated holes directly capture the electrons of RhB and discolor it. Since the photogenerated electrons are entrapped and transferred by \( \beta \)-FeSe nanorods, the recombination of the electrons and holes are improved, which was confirmed by the EIS and PL spectra. In other words, the photogenerated electrons and holes involved in the discoloration reaction of RhB are increased. Thus, the photocatalytic efficiency is enhanced for \( \beta \)-FeSe/g-C₃N₄ composites.

Figure 6. (a) EIS at 0.6 V (versus Ag/AgCl) in 0.5 M Na₂SO₄ solution under visible light irradiation. (b) PL under 330 nm excitation at 298 K.

Figure 7. Proposed photocatalysis mechanism diagrams of decolourization of RhB for \( \beta \)-FeSe/g-C₃N₄ composites.
4. Conclusion

In summary, β-FeSe nanorods were used as co-catalyst composed with g-C$_3$N$_4$. The photocatalytic efficiency is remarkably enhanced for β-FeSe/g-C$_3$N$_4$ composites. The decolourization rate of 3 wt% β-FeSe/g-C$_3$N$_4$ composites reaches 4.4 times that of g-C$_3$N$_4$. The RhB in solution is completely decomposed within 60 min for 3 wt% β-FeSe/g-C$_3$N$_4$ composites with H$_2$O$_2$. The photogenerated electrons can be entrapped and transferred by β-FeSe nanorods, which reduces the recombination of the electrons and holes and improves the photocatalytic efficiency. This work provides a promising co-catalyst for photocatalytic discolorization of organic matter.

Data accessibility. Our data are deposited at Dryad Digital Repository: http://dx.doi.org/10.5061/dryad.3310h18 [44]. Authors’ contributions. W.Z. and S.F. designed this work; S.S. and W.Z. performed the experiments; Z.W. and Z.L. analysed the data; S.S. wrote this paper. All authors gave final approval for publication.

Competing interests. The authors declare no competing interests.

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