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# Plasmonic effect-driven strategy for boosting photoelectrocatalytic degradation of rhodamine B using a SnS<sub>2</sub>/Au self-supported photoelectrode

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Photoelectrocatalytic degradation of pollutants is regarded as one of the green disposal schemes for water pollutants under the "dual carbon" strategy. In this paper, a gold-based tin disulfide self-supporting photoelectrode (SnS<sub>2</sub>-Au/Ti foil) was successfully prepared by a two-step method and applied to the photoelectrocatalytic degradation of rhodamine B (RhB) pollutants. The phase structure of the self-supported photoelectrode was characterized by scanning electron microscopy, X-ray diffraction, ultraviolet-visible spectrophotometry, and fluorescence spectrophotometry. The test results for photoelectrocatalytic degradation performance demonstrated that the degradation performance of the pollutant RhB was significantly enhanced via the plasmonic effect of gold in synergy with the intrinsic photocatalytic characteristics of the tin sulfide semiconductor. The photoelectrocatalytic degradation efficiency of the self-supported photoelectrode reached 81.25% within 120 minutes. The composite electrode SnS<sub>2</sub>-Au/Ti foil demonstrated an 38.18% and 8.45% increase in photoelectrocatalytic degradation of RhB efficiency over the single Au and SnS<sub>2</sub> electrodes, respectively. The plasmonic effect-driven strategy for boosting photoelectrocatalytic efficiency opens new perspectives for RhB degradation.

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## 1 Introduction

Driven by rapid industrialization and urbanization, the surging global energy demand has exacerbated environmental degradation, particularly through escalating pollutant emissions. Among these pollutants, Rhodamine B (RhB), a synthetic cationic dye widely used in the textile, paper, and leather industries, has garnered significant attention owing to its resistance to natural degradation and its potent biotoxicity.<sup>1,2</sup> It is classified by the World Health Organization as a Group 3 carcinogen.<sup>3</sup> Currently, the degradation treatment of RhB primarily relies on four main methodologies: photocatalytic oxidation, Fenton and Fenton-like methods,<sup>4,5</sup> combined degradation agents, and biological synergy techniques.<sup>6</sup> However, the presence of secondary pollution, high costs, and low degradation efficiency, among other issues, presents substantial challenges in establishing a green and efficient pathway for rhodamine B degradation. Photoelectrocatalytic degradation technology represents a green and highly efficient degradation approach that leverages the synergistic effects of solar energy and electrical energy. This technology has been extensively applied to degrade methylene blue<sup>7-9</sup> and various other toxic and hazardous substances. The design and fabrication of photoelectrodes are critical factors in determining the

efficiency and effectiveness of photoelectrocatalytic degradation processes.

Tin disulfide (SnS<sub>2</sub>) semiconductor material ( $E_g \approx 2.2$  eV) exhibits effective visible-light absorption and is widely utilized for photocatalysts.<sup>10</sup> Shuai *et al.* successfully synthesized Mn-doped SnS<sub>2</sub> photocatalysts, which significantly enhanced the performance of photocatalytic degradation of RhB.<sup>11</sup> Chen *et al.* prepared SnS<sub>2</sub>/CPAN nanocomposites by compounding cyclized polyacrylonitrile (CPAN), effectively improving the photocatalytic degradation efficiency and stability.<sup>12</sup> The plasmonic effect refers to a range of optical, thermal, and electronic phenomena that arise from the coupling of photons with collective oscillations of free electrons in metals or materials with high free electron density. It has been widely utilized to significantly enhance photocatalytic performance by leveraging three key advantages: a large extinction cross-section, tunable resonant energy, and ultrasensitive sensing capabilities.<sup>13,14</sup> The Au@TiO<sub>2</sub> core-shell nanostructure catalyst developed by Chen *et al.*<sup>15</sup> demonstrates enhanced adsorption capacity and photocatalytic degradation efficiency through synergistic integration of the plasmonic effect from Au cores and interfacial charge transfer engineering in the heterostructure. The strategic integration of the plasmonic effect in Au/SnS<sub>2</sub> heterojunction photoelectrodes is expected to achieve remarkable enhancement in photodegradation efficiency.

In this paper, a gold-based tin sulfide self-supporting photoelectrode (SnS<sub>2</sub>/Au) was fabricated on a titanium foil substrate

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through a combination of ion sputtering and solvothermal methods. The surface morphology and optical behavior of the photoelectrode were comprehensively characterized using scanning electron microscopy (SEM), X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), ultraviolet-visible spectrophotometry (UV-vis), and fluorescence spectrophotometry (FL), respectively. The experimental results reveal that the  $\text{SnS}_2/\text{Au}$  heterostructure not only synergistically extends photon harvesting in the visible light region but also suppresses photoinduced electron-hole recombination, compared with monocomponent  $\text{SnS}_2$  or Au catalysts. The photoelectrochemical degradation rate of RhB on the  $\text{SnS}_2\text{-Au/Ti}$  foil composite self-supporting photoelectrode reached 81.25% within 120 min, which was superior to that of the single  $\text{SnS}_2/\text{Ti}$  foil (72.80%) and the single Au/Ti foil electrode (43.07%). This work presents a novel approach to enhancing the photoelectric degradation of RhB through the utilization of the plasmon effect.

## 2 Experimental

### 2.1 Experimental reagents

Tin(IV) chloride pentahydrate ( $\text{SnCl}_4 \cdot 5\text{H}_2\text{O}$ ), thioacetamide ( $\text{C}_2\text{H}_5\text{NS}$ ), anhydrous sodium sulfite ( $\text{Na}_2\text{SO}_3$ ), rhodamine B ( $\text{C}_{28}\text{H}_{31}\text{ClN}_2\text{O}_3$ ), potassium chloride (KCl), and absolute ethanol ( $\text{C}_2\text{H}_6\text{O}$ ) were all purchased from Shanghai Macklin Biochemical Technology Co., Ltd. The titanium foil and gold target (99.999%) were purchased from Zhongnuo New Materials (Beijing) Technology Co., Ltd. The deionized water used in the experiment was self-prepared by a pure water machine (TTL-6B). The reagents involved in the experiment were all directly purchased and used without further processing.

### 2.2 The preparation of $\text{SnS}_2\text{-Au/Ti}$ foil self-supporting photoelectrode

The self-supported  $\text{SnS}_2\text{-Au/Ti}$  foil photoelectrode was successfully fabricated on the metal titanium substrate using the ion sputtering method and the solvothermal method, as illustrated in Scheme 1.

**(1) The Au/Ti foil was prepared by the ion sputtering method.** The titanium foil was cut into  $2 \times 4$  cm rectangles, and it was cleaned with detergent powder, absolute ethanol, and deionized water for 30 minutes, respectively. Au thin films were

deposited on Ti foils by magnetron sputtering (GVC-2000) under optimized conditions: a target-substrate distance of 2.5 cm, a current of 10 mA, and an Ar pressure of 4.00 Pa. By controlling the sputtering durations (40–200 s),  $\text{Au}_x/\text{Ti}$  electrodes (where  $x$  represents the deposition time in seconds) were fabricated. Unless otherwise specified, all Au/Ti foil electrodes mentioned in this study refer to those prepared with a sputtering time of 120 s.

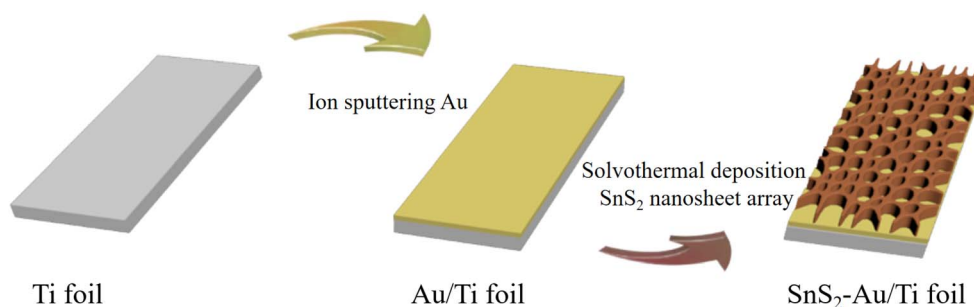
**(2) The  $\text{SnS}_2\text{-Au/Ti}$  foil photoelectrode was prepared by the solvothermal method.** The as-prepared Au/Ti foil or pure Ti foil was immersed in a solution of absolute ethanol containing 50 mM  $\text{C}_2\text{H}_5\text{NS}$  and 25 mM  $\text{SnCl}_4 \cdot 5\text{H}_2\text{O}$  in a 25 mL reaction vessel. The reaction vessel was then transferred to a vacuum drying oven at  $80^\circ\text{C}$ , where it was allowed to react for 4 hours to prepare either the  $\text{SnS}_2\text{-Au/Ti}$  foil electrode or the  $\text{SnS}_2\text{-Ti}$  foil electrode.

### 2.3 Characterization

The crystal structure of the catalyst on the photoelectrode surface was analyzed by X-ray diffraction (XRD, MiniFlex600). The scanning range was set from  $20^\circ$  to  $80^\circ$ , and the scanning speed was set at  $10^\circ \text{ min}^{-1}$ . Microscopic morphology and structure were analyzed by using a scanning electron microscope (SEM, JSM-7800F). The elemental composition and valence state were analyzed by X-ray photoelectron spectroscopy (XPS, Thermo ESCALAB 250). The optical behavior of the self-supporting photoelectrode was analyzed using an ultraviolet-visible spectrophotometer (UV-vis, UV-690) and a fluorescence spectrophotometer (FL, F-7000). The excitation light wavelength of FL spectrophotometer was set to 410 nm.

### 2.4 Photoelectrochemical degradation of RhB measurements

The standard three-electrode system was employed as the photoelectrochemical degradation cell. The CHI 760E instrument served as the electrochemical workstation, and the degradation bias was set at 0.3 V vs. SCE. Specifically, the as-prepared photoelectrode, the saturated calomel electrode, and the platinum sheet were used as the working electrode, the reference electrode, and the counter electrode, respectively. A mixed solution of 20 mM  $\text{Na}_2\text{SO}_3$ , 0.5 M KCl, and  $4 \text{ mg L}^{-1}$  RhB was used as the degradation solution. Visible light irradiation was provided by xenon lamps (CEL-HXF300), and the



**Scheme 1** Schematic illustration of the synthesis process of  $\text{SnS}_2\text{-Au/Ti}$  foil self-supporting photoelectrode.



concentration of RhB during the degradation process was monitored using the ultraviolet-visible spectrophotometer (752G, Shanghai Yidian).

### 3 Results and discussion

#### 3.1 Structure characterization

Fig. 1a–f presents the SEM images of the Ti foil, Au/Ti foil,  $\text{SnS}_2/\text{Ti}$  foil, and  $\text{SnS}_2\text{-Au/Ti}$  foil electrodes, respectively. The SEM image of Ti foil depicts a surface with relatively low roughness (shown in Fig. 1a). Fig. 1b corresponds to the Au/Ti foil electrode, demonstrating that gold nanoparticles are uniformly distributed on the titanium metal substrate. Fig. 1c and d is associated with the  $\text{SnS}_2/\text{Ti}$  foil electrode, where the as-prepared  $\text{SnS}_2$  nanosheet arrays are evenly deposited on the titanium metal substrate, exhibiting a pore size of approximately 200 nanometers. The SEM images of the  $\text{SnS}_2\text{-Au/Ti}$  foil (shown in Fig. 1e and f) are similar to those of the  $\text{SnS}_2/\text{Ti}$  foil, suggesting that the incorporation of an intermediate Au nanoparticle layer does not affect the morphology of  $\text{SnS}_2$ .

Elemental mapping images of  $\text{SnS}_2\text{-Au/Ti}$  foil are illustrated in Fig. 1g<sub>1–3</sub>, showing uniform distributions of S, Au, and Sn

elements on the titanium substrate. Furthermore, the energy-dispersive X-ray spectroscopy (EDX) analysis was employed to analyze the atomic content of corresponding elements (Fig. 1h). The results indicate that the  $\text{SnS}_2\text{-Au/Ti}$  foil electrode is predominantly composed of Sn, S, and Au elements, with an atomic ratio of Sn to S approximately equal to 1 : 2, confirming the formation of  $\text{SnS}_2$ . Notably, Au accounts for only 1.63% of the total elemental composition of the  $\text{SnS}_2\text{-Au/Ti}$  foil electrode. Besides, the EDX spectra of Ti foil,  $\text{Au}_{120}/\text{Ti}$  foil and  $\text{Au}_{200}/\text{Ti}$  foil were displayed in Fig. S1. It is evident that the gold content on the electrode surface increases with prolonged ion sputtering duration.

The crystal structure of the prepared electrode has been characterized, as depicted in Fig. 2. The diffraction peaks at  $44.37^\circ$  and  $64.56^\circ$  are indexed to (200) and (220) planes of Au, respectively (JCPDS card no. 89-3697). Obviously, the XRD diffraction peaks of Au nanoparticles for the  $\text{Au}_x/\text{Ti}$  foil electrode become sharper with an extended sputtering duration, as shown in Fig. 2a. Besides, the diffraction peaks at  $15.05^\circ$ ,  $30.38^\circ$ ,  $50.11^\circ$ , and  $58.55^\circ$  correspond to the (001), (002), (110), and (200) planes of  $\text{SnS}_2$ , respectively (JCPDS card no. 83-1705).

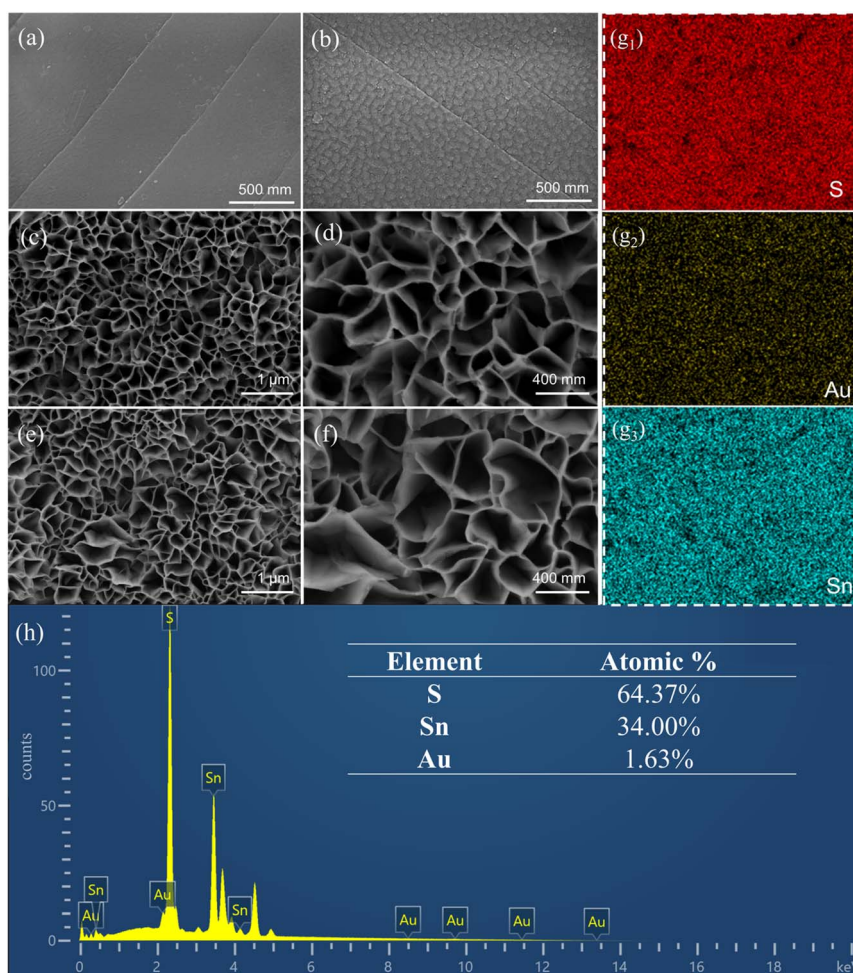


Fig. 1 SEM images of (a) Ti foil electrode, (b) Au/Ti foil electrode, (c and d)  $\text{SnS}_2/\text{Ti}$  foil electrode and (e and f)  $\text{SnS}_2\text{-Au/Ti}$  foil electrode, respectively. (g<sub>1–3</sub>) Elemental mapping images and (h) EDX spectra of  $\text{SnS}_2\text{-Au/Ti}$  foil electrode.



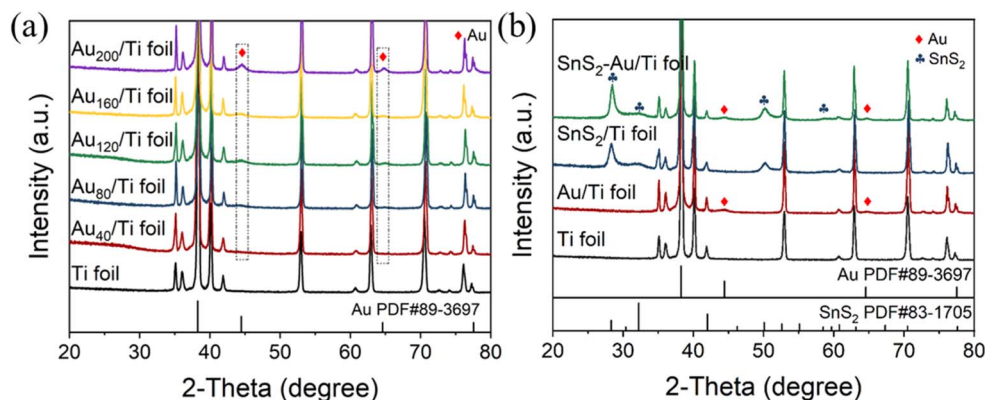


Fig. 2 XRD patterns of (a) the  $Au_x/Ti$  foil electrodes prepared with different sputtering durations, (b)  $SnS_2-Au/Ti$  foil,  $Au/Ti$  foil,  $SnS_2/Ti$  foil and  $Ti$  foil electrodes.

Additionally, the remaining diffraction peaks can be attributed to the titanium metal substrate. A comparison of the XRD diffraction patterns for the  $SnS_2-Au/Ti$  foil,  $Au/Ti$  foil,  $SnS_2/Ti$  foil, and  $Ti$  foil electrodes (Fig. 2b) clearly demonstrates that  $SnS_2$  can be effectively synthesized on both  $Au/Ti$  foil and  $Ti$  foil substrates *via* the solvothermal method.

The XPS analysis was performed on the  $SnS_2-Au/Ti$  foil electrode to characterize the elemental composition and valence state, as shown in Fig. 3. It is obvious that the  $SnS_2-Au/Ti$  foil electrode contains the primary elements of Sn, S, and Au (as shown in Fig. 3a). In Fig. 3b, the peaks at 161.7 and 162.9 eV are attributed to  $S^{2-} 2p_{3/2}$  and  $S^{2-} 2p_{1/2}$ , respectively. The peaks at 486.8 and 495.2 eV correspond to  $Sn^{4+} 3d_{5/2}$  and  $Sn^{4+} 3d_{3/2}$ , respectively (Fig. 3c).<sup>16–18</sup> Furthermore, the weak peaks of  $Au 4f_{7/2}$  and  $Au 4f_{5/2}$  are located at 84.2 and 87.6 eV, respectively (Fig. 3d).<sup>19,20</sup>

### 3.2 Optical property

The optical behavior of self-supported photoelectrodes serves as a critical factor influencing the photoelectrocatalytic degradation performance. The UV-vis absorption spectra and FL emission spectra of  $SnS_2-Au/Ti$  foil,  $Au/Ti$  foil, and  $SnS_2/Ti$  foil electrodes were illustrated in Fig. 4. Obviously, the as-prepared  $Au/Ti$  foil displays a noteworthy absorption peak near 560 nm, which is indicative of the localized surface plasmon resonance (LSPR) effect (Fig. 4a).<sup>21,22</sup> For the  $SnS_2/Ti$  foil electrode, the absorption peak observed at 580 nm can be attributed to the intrinsic bandgap (2.2 eV) of the  $SnS_2$  semiconductor.<sup>23</sup> Furthermore, the light absorption of the as-prepared  $SnS_2-Au/Ti$  composite self-supported photoelectrode exhibits a red shift, indicating that the plasmonic effect of the Au layer can effectively synergize with the inherent light absorption properties of the  $SnS_2$  semiconductor, leading to an enhancement in the overall light absorption capacity of the self-supported  $SnS_2-Au/Ti$  composite photoelectrode within the visible light spectrum.

As shown in Fig. 4b, the FL emission spectra of the as-synthesized electrodes are illustrated, with the peak intensity at 600 nm being proportional to the recombination probability

of photogenerated carriers.<sup>24</sup> The  $Au/Ti$  foil electrode exhibits a substantially higher FL emission peak in comparison to the  $SnS_2-Au/Ti$  foil and  $SnS_2/Ti$  foil electrodes. This phenomenon can be attributed to the plasmonic effect of Au, which generates photogenerated carriers in significantly higher magnitudes and causes much faster recombination than those in semiconductor materials.<sup>25</sup> Most importantly, the  $SnS_2-Au/Ti$  foil electrode exhibits much lower emission intensity, revealing that the synergistic effect of  $SnS_2$  and Au significantly reduces the recombination of photogenerated carriers.

### 3.3 Photoelectrochemical degradation of RhB measurements

Fig. 5a presents the photoelectrocatalytic degradation performance test results of  $SnS_2-Au/Ti$  composite electrodes, which were fabricated by *in situ* growing  $SnS_2$  nanosheet arrays on  $Au_{40}/Ti$  foil,  $Au_{80}/Ti$  foil,  $Au_{120}/Ti$  foil,  $Au_{160}/Ti$  foil, and  $Au_{200}/Ti$  foils, respectively. It can be observed that the photoelectrocatalytic degradation performance of the composite electrode initially increases with the prolongation of the sputtering time of Au nanoparticles and subsequently decreases. The  $SnS_2-Au_{120}/Ti$  foil composite electrode exhibits the highest photoelectric degradation efficiency for RhB within 120 min, achieving a degradation rate of 81.25%. The photo response current density serves as a key indicator for evaluating the photoelectrocatalytic performance of the photoelectrode. As illustrated in Fig. 5b, the  $J-t$  curves depict the behavior under a constant bias of 0.3 V vs. SCE in the same photoelectrochemical degradation cell, measured under intermittent illuminated and dark conditions. The  $Au/Ti$  foil electrode shows a weak photoelectric response due to the plasmonic effect of Au nanoparticles, but exhibits the lowest photoelectric response among the samples because of limited active reaction sites. When the tin disulfide semiconductor was synergistically integrated with the Au nanoparticles, the photo response current density reaches its maximum, indicating optimal photoelectrocatalytic performance of the  $SnS_2-Au/Ti$  foil electrode.

Fig. 5c illustrates the photoelectrocatalytic, photocatalytic, and electrocatalytic degradation performance of the  $SnS_2-Au/Ti$



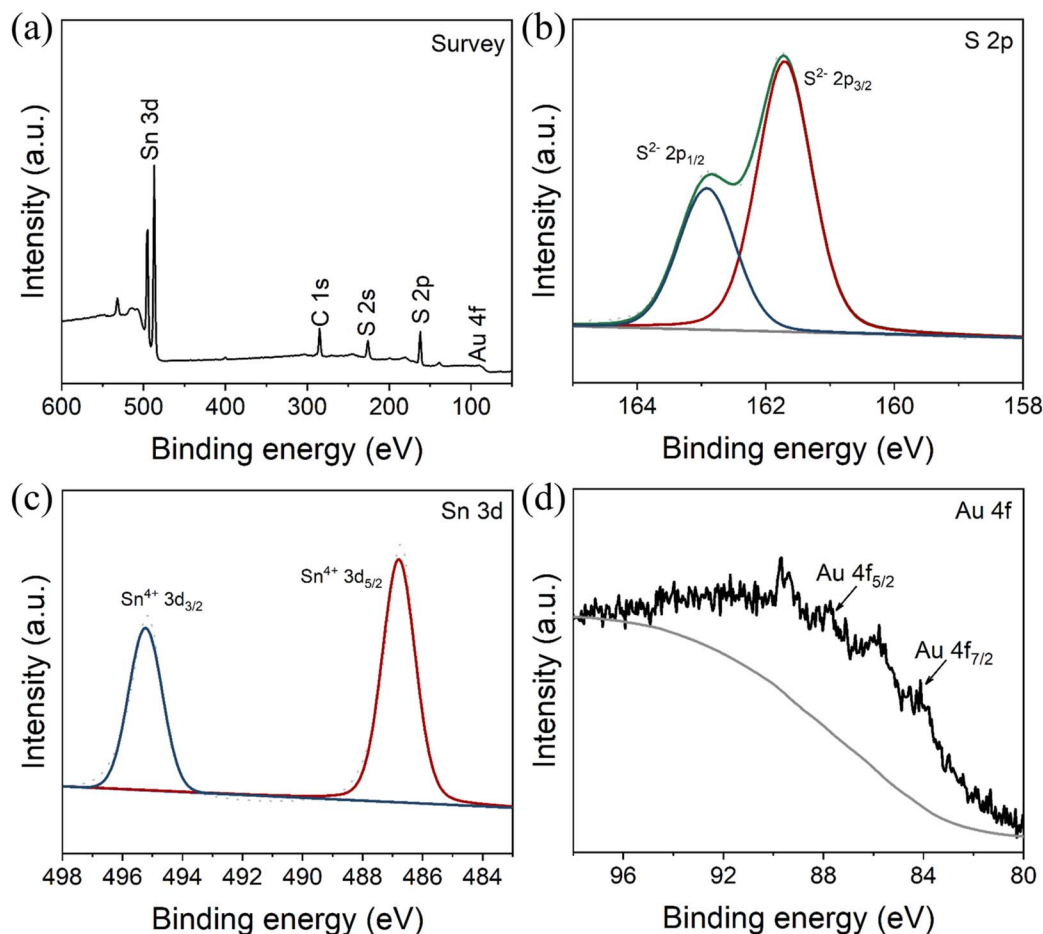


Fig. 3 XPS spectrum of the  $\text{SnS}_2\text{-Au/Ti}$  foil, (a) survey spectra and high-resolution XPS (HRXPS) spectrum of (b) S 2p, (c) Sn 3d, and (d) Au 4f.

foil composite electrode under identical testing conditions. Within 120 min, the pure electrocatalytic degradation and photocatalytic degradation efficiencies of the  $\text{SnS}_2\text{-Au}_{120}/\text{Ti}$  foil composite electrode are 45.59% and 68.99%, respectively, both of which are lower than its photoelectrocatalytic degradation efficiency. Furthermore, the repeated photoelectrocatalytic

degradation performance was also tested to evaluate the stability of the composite electrode, as displayed in Fig. 5d. It can be clearly observed that the degradation rate of the electrode reached approximately 85% within 120 minutes in five repeated experiments, and then stabilized in the third test cycle. This stabilization might be due to the excessive catalyst

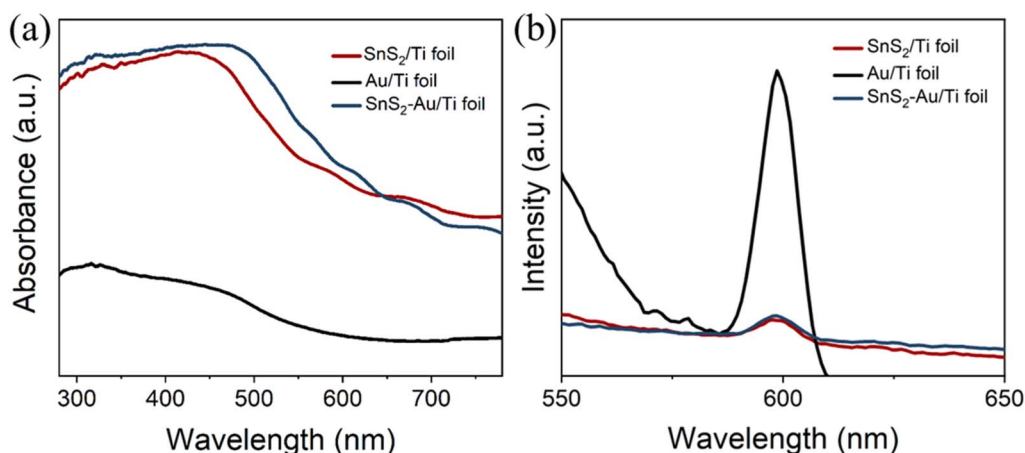


Fig. 4 (a) UV-vis absorption spectra and (b) FL emission spectra of  $\text{SnS}_2\text{-Au/Ti}$  foil,  $\text{Au/Ti}$  foil, and  $\text{SnS}_2/\text{Ti}$  foil electrodes.

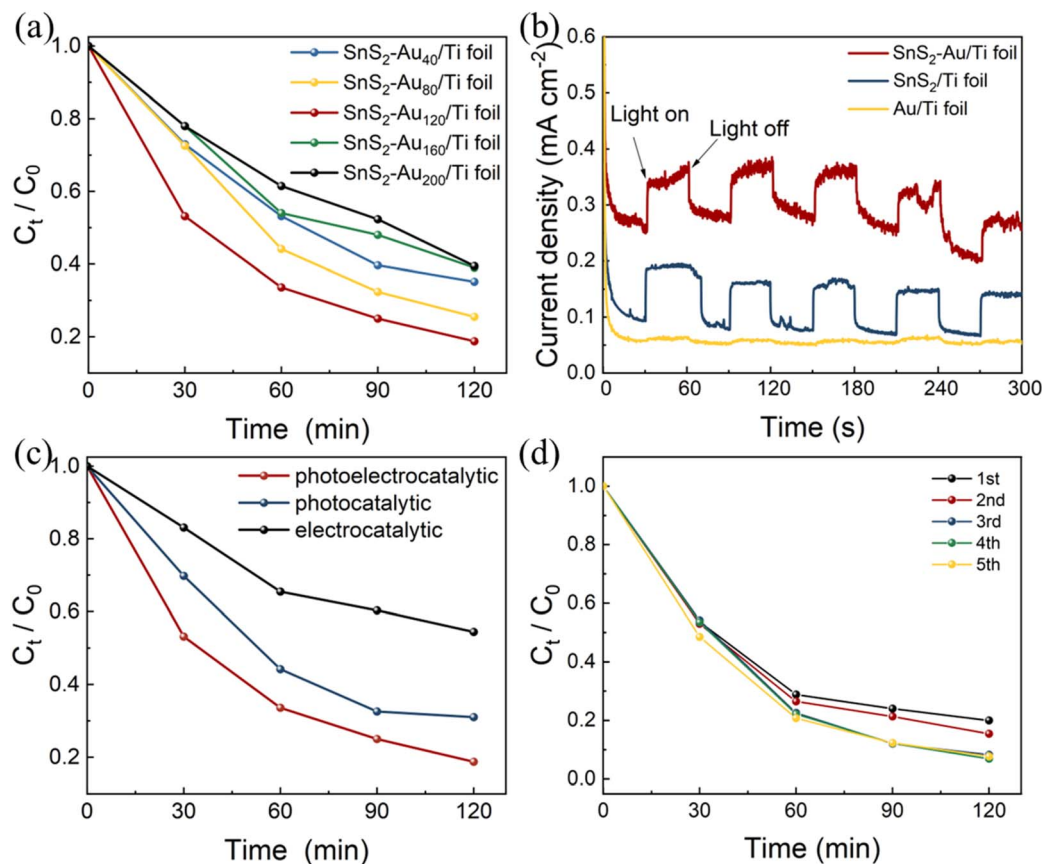


Fig. 5 (a) Photoelectrocatalytic degradation performance of RhB on  $\text{SnS}_2\text{-Au}_x/\text{Ti}$  foil electrodes prepared by varying durations of Au ion-sputtering. (b)  $J$ - $t$  curves of  $\text{SnS}_2\text{-Au/Ti}$  foil,  $\text{Au/Ti}$  foil, and  $\text{Ti}$  foil electrodes under a bias of 0.3 V vs. SCE under intermittent dark and illumination conditions. (c) Comparison of the RhB degradation performance via photoelectrocatalytic, photocatalytic, and electrocatalytic degradation and (d) the repeated photoelectrocatalytic degradation performance of RhB on  $\text{SnS}_2\text{-Au/Ti}$  foil electrode.

shedding from the surface of the composite electrode during the first two degradation cycles.

To further investigate the origin of the photoelectrocatalytic degradation performance of RhB using the  $\text{SnS}_2\text{-Au/Ti}$  foil composite electrode, the  $\text{Ti}$  foil,  $\text{SnS}_2/\text{Ti}$  foil, and  $\text{Au/Ti}$  foil

electrodes were individually evaluated under the same testing conditions, as illustrated in Fig. 6a. The degradation rate of the bare  $\text{Ti}$  foil substrate was about 7.74% within 120 min, which might be attributed to the adsorption effect of the metal substrate on RhB.<sup>26</sup> Besides, the  $\text{SnS}_2\text{-Au/Ti}$  foil composite

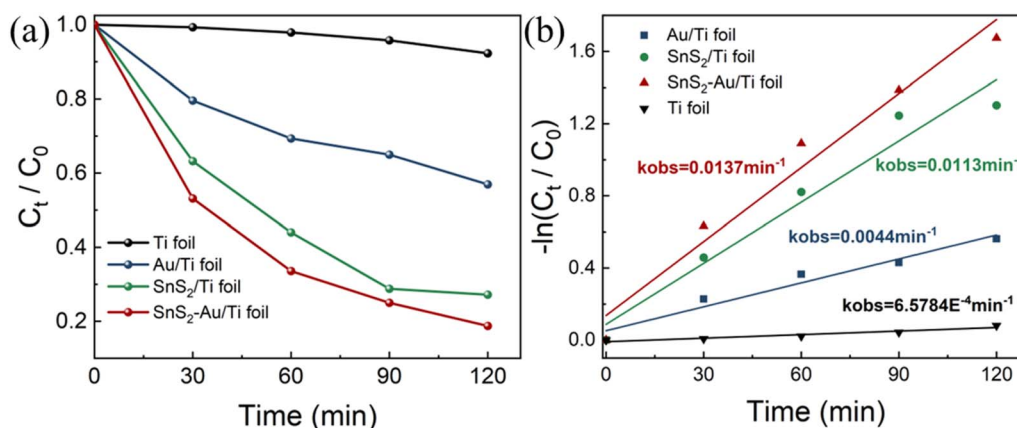


Fig. 6 (a) Photoelectrocatalytic degradation performance and (b) corresponding kinetic curves of  $\text{Ti}$  foil,  $\text{SnS}_2/\text{Ti}$  foil,  $\text{Au/Ti}$  foil, and  $\text{SnS}_2\text{-Au/Ti}$  foil electrodes, respectively.



electrode exhibits a degradation rate of 81.25%, demonstrating an enhancement of 88.87% and 11.59% compared to the pure Au/Ti foil electrode (43.02%) and the SnS<sub>2</sub>/Ti foil electrode (72.81%), respectively. Besides, the pseudo-first-order kinetic model was employed to analyze the degradation kinetics of RhB on various photoelectrodes, as illustrated in Fig. 6b. Among them, the degradation rate of SnS<sub>2</sub>-Au/Ti foil composite electrode was the highest, reaching 0.0137 min<sup>-1</sup>, which was higher than that of Ti foil (0.0006 min<sup>-1</sup>), SnS<sub>2</sub>/Ti foil (0.0113 min<sup>-1</sup>), and Au/Ti foil (0.0044 min<sup>-1</sup>) electrodes. It is evident that the SnS<sub>2</sub>-Au/Ti foil composite electrode has the strongest photoelectrocatalytic degradation activity. Thus, the experimental results demonstrate that the SnS<sub>2</sub>-Au/Ti foil composite electrode, fabricated by loading SnS<sub>2</sub> nanosheet arrays onto a bottom layer of Au nanoparticles, can achieve both the plasmonic effect of Au and the semiconductor photocatalytic effect of SnS<sub>2</sub>. This effectively enhances the photoelectric degradation process, thereby promoting the degradation of RhB.

## 4. Conclusions

In summary, we successfully fabricated the SnS<sub>2</sub>-Au/Ti foil composite self-supporting photoelectrode on a titanium metal substrate through the combination of ion sputtering and solvothermal methods, and demonstrated its application in the photoelectrocatalytic degradation of RhB. The experimental results demonstrate that the synergistic integration of the plasmonic effect in Au with the semiconductor inherent characteristics of SnS<sub>2</sub> enables the composite electrode to exhibit enhanced performance in photoelectrocatalytic degradation of RhB. Under a constant potential of 0.3 V, the system achieves a degradation efficiency of 81.25% within 120 minutes. This strategy of enhancing the semiconductor photodegradation performance *via* the synergy of the plasmonic effect offers a novel perspective for the investigation of pollutant photodegradation.

## Conflicts of interest

There are no conflicts to declare.

## Data availability

All relevant data are within the manuscript and its additional files.

Supplementary information: the EDS spectrum (EDS) of SnS<sub>2</sub>-Au/Ti foil electrode. See DOI: <https://doi.org/10.1039/d5ra04205d>.

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