

Novel Pt,Ru-Codoped $\text{In}_2\text{S}_3/\text{Ta}_2\text{O}_5$ heterostructured films for the effective enhancement of visible-light photocatalytic degradation of rhodamine B

Natkritta Boonprakob

`natkritta.boon@uru.ac.th`

Uttaradit Rajabhat University

Natthakit Huatong

Uttaradit Rajabhat University

Narong Chanlek

Synchrotron Light Research Institute

Kemawadee Udonpan

Thepsatri Rajabhat University

Chaiyuth Sae-Kung

Thailand National Metal and Materials Technology Center

Chen Zhao

Guangdong University of Technology

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Novel Pt,Ru-Codoped In₂S₃/Ta₂O₅ heterostructured films for the effective enhancement of visible-light photocatalytic degradation of rhodamine B

Natkritta Boonprakob^{1*}, Natthakit Huatong², Narong Chanlek³, Kemawadee Udonpan⁴,
Chaityuth Sae-Kung⁵ and Chen Zhao⁶

¹ *Program of Chemistry, Faculty of Science and Technology, Uttaradit Rajabhat University, Uttaradit 53000, Thailand.*

² *Program of Chemistry, Faculty of Education, Uttaradit Rajabhat University 53000, Uttaradit*

³ *Synchrotron Light Research Institute, Nakhon Ratchasima 30000, Thailand.*

⁴ *Department of Chemistry, Faculty of Science and Technology, Thepsatri Rajabhat University, Lop Buri, 15000, Thailand.*

⁵ *National Metal and Materials Technology Centre, National Science and Technology Development Agency, Pathum Thani, 12120, Thailand.*

⁶ *School of Materials and Energy, Guangdong University of Technology, Guangzhou 510006, China*

Corresponding Author

*Email address: natkritta.boo@uru.ac.th

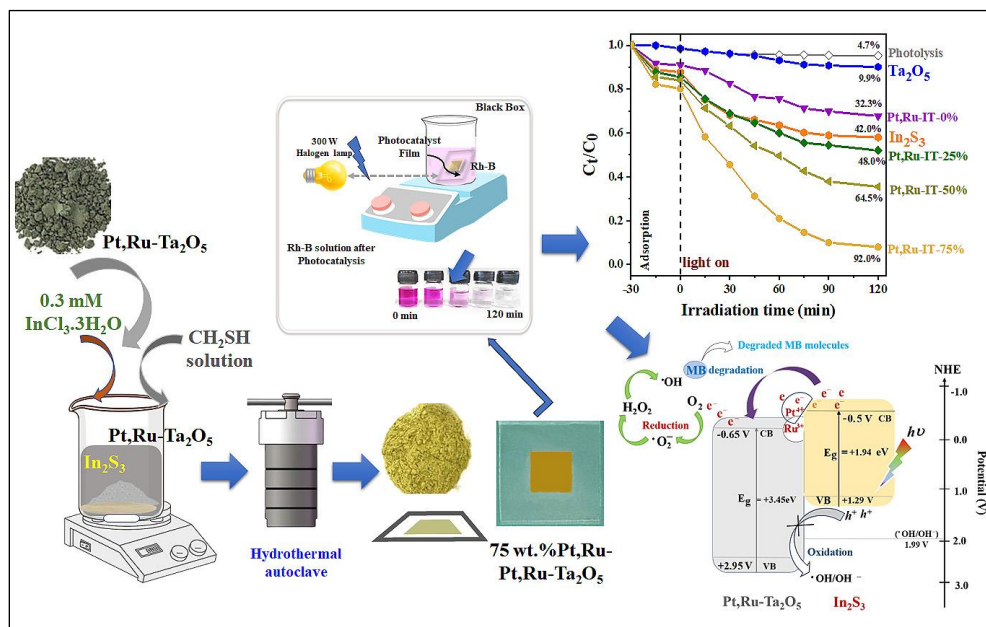
Phone: +66 88 9826149

Highlights,

- We can successfully synthesize a novel visible-light-driven In₂S₃/Pt,Ru-Ta₂O₅ heterostructured nanocatalyst film.
- Strong surface and microstructural examination data show that the composite In₂S₃/Ta₂O₅ doped with Pt and Ru ions has enhanced crystallinity, demonstrating that it is an effective photocatalyst.
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- The optimum photocatalysts exhibited 92.0% was reached in 120 min illumination. RhB dye degradation efficiency and high recyclability.

Graphitic Abstract



Abstract

A visible light-responsive Pt,Ru Noble metals were introduced by wet impregnation after the $\text{In}_2\text{S}_3/\text{Pt,Ru}$ -doped Ta_2O_5 heterostructured catalyst film was produced using a combination hydrothermal and doctor blading method. XRD, UV-Vis diffuse reflectance spectroscopy, SEM, TEM-EDS, and XPS were used to evaluate the physicochemical properties after the composition was enhanced by varying the In_2S_3 content (25–75 wt.%). In addition to exhibiting a narrow bandgap (~ 2.14 eV) and increased visible light absorption, the optimized $\text{In}_2\text{S}_3/\text{Pt,Ru-Ta}_2\text{O}_5$ composite with 75 wt.% In_2S_3 additionally had a larger surface area and uniformly distributed plate-like In_2S_3 on the Ta_2O_5 . XPS investigations indicated strong electronic interactions between the Pt/Ru species and Ta_2O_5 , as evidenced by binding energy shifts and the formation of partially oxidized metal species and oxygen vacancies, which serve as effective charge-trapping sites. Rhodamine B degradation under visible light irradiation was carried out to measure photocatalytic performant, and after 120 min., up to 92.0% removal was achieved. Superoxide radicals ($\cdot\text{O}_2^-$) were exhibited to be the predominant reactive species in scavenger analysis, suggesting effective interfacial electron

transportation. The interaction of defect-mediated electronic interactions, metal-induced charge separation, and heterojunction formation are responsible for the enhanced performance. These results indicate that a successful approach for altering the electronic structure and improving the charge carrier dynamics in heterostructured oxide-based photocatalysts is the logical integration of In_2S_3 with Pt,Ru-modified Ta_2O_5 .

Keywords: In_2S_3 , Ta_2O_5 , Heterostructure, Nanocatalyst, Rhodamine B, Interfacial charge transfer

Introduction

Rhodamine B (RhB), a cationic xanthene dye used extensively in the textile industry, is frequently detected in industrial effluents, given its high aqueous solubility and chemical stability [1]. The development of effective remediation solutions is necessary due to the persistence and potential carcinogenicity of RhB, that present major health and environmental hazards. Despite the wide employment of conventional approaches, such as adsorption using low-cost materials, these methods are often limited by secondary waste generation and the incomplete mineralization of organic contaminants [2,3]. Photocatalytic advanced oxidation processes (AOPs) have attracted a lot of interest in recent years because of their ability to generate highly reactive species such as hydroxyl ($\cdot\text{OH}$) and superoxide ($\cdot\text{O}_2^-$) radicals, which enable the efficient decomposition of organic contaminants. Semiconductor-based photocatalysts, particularly metal oxides such as Ta_2O_5 , ZnO , WO_3 , and BiVO_4 , have been extensively investigated because of their structural stability and tunable electronic properties [4,5]. Among these materials, Ta_2O_5 is of particular interest due to its chemical robustness and favorable electronic structure. Nevertheless, its wide bandgap (~ 3.9 eV) limits visible light absorption, and the rapid recombination of photogenerated electron–hole pairs significantly restrict its photocatalytic efficiency [6,7]. Various modification strategies have been proposed to overcome these limitations, including heterojunction construction, defect engineering, and metal doping, all of which aim to customize the electronic structure and improve the charge carrier dynamics. Incorporating transition metal dopants into the Ta_2O_5 lattice can induce distortion, modify the local electronic environment, and promote the formation of oxygen vacancies, which act as effective charge-trapping sites and facilitate charge separation [8]. Notably, Ru incorporation has been reported to enhance n-type conductivity and shift the Fermi level toward the conduction band via the Burstein–Moss effect, thereby improving the charge transport properties [9]. Moreover, the higher electronegativity of Ru compared to Ta promotes charge redistribution and enhances catalytic activity. Noble metal species such as Pt can further function as electron sinks via strong metal–support interactions, effectively suppressing electron–hole recombination

and enhancing the efficiency of charge separation [10,11]. Another strategy involves coupling wide-bandgap oxides with narrow-bandgap semiconductors to extend the visible light absorption. Indium sulfide (In_2S_3), an n-type semiconductor with a bandgap of 2.0–2.4 eV [12] and a relatively negative conduction band potential, has been widely investigated regarding visible-light-driven photocatalysis. The formation of In_2S_3 -based heterostructures significantly suppresses charge recombination and promotes interfacial charge transfer. In addition, its strong interaction with cationic dye molecules enhances the adsorption behavior and photocatalytic efficiency [13]. Despite such improvements, studies on heterostructured systems integrating In_2S_3 with noble metal (Pt,Ru)-modified Ta_2O_5 remain limited, notably in terms of catalyst films designed to perform visible-light-driven photocatalysis under simulated solar irradiation [14,15]. The synergistic combination of dual noble metal doping and sulfide/oxide heterojunction engineering is expected to modify the electronic structure, establish defect states, and facilitate interfacial charge transfer processes. Therefore, in this work, a visible-light-responsive $\text{In}_2\text{S}_3/\text{Pt,Ru-Ta}_2\text{O}_5$ heterostructured catalyst film was fabricated, and the effects of composition, electronic structure, and surface properties on the photocatalytic performance were elucidated. Furthermore, the roles of reactive species and catalyst stability were evaluated to gain insights into the underlying photocatalytic mechanism [16,17].

To the best of our knowledge, this work presents the first demonstration of a visible-light-driven $\text{In}_2\text{S}_3/\text{Pt,Ru-Ta}_2\text{O}_5$ heterostructured catalyst film, where the synergistic integration of dual noble metal doping and sulfide/oxide heterojunction engineering enables defect-mediated interfacial charge transfer and dominant $\cdot\text{O}_2^-$ radical pathways, resulting in a markedly enhanced photocatalytic degradation efficiency toward RhB.

Materials and Methods

Fabrication of $\text{In}_2\text{S}_3/\text{Pt,Ru-doped Ta}_2\text{O}_5$ films

Pt,Ru-doped Ta_2O_5 nanocomposite powders were first prepared via wet impregnation. Platinum species were introduced into Ta_2O_5 at atomic concentrations of 0.25, 0.5, 1.0, 2.5, and 5.0 at.% using analytical-grade precursors (Sigma-Aldrich, 99.0%). The mixtures were thoroughly ground, sieved through a 200-mesh stainless steel sieve to remove agglomerates, dried, and subsequently calcined at 400 °C for 3 h in air. Further, Ru-doped Ta_2O_5 powders with concentrations of 0.5, 1.0, 2.5, 5.0, and 10.0 at.% were synthesized under identical conditions. Prior to the heterostructure fabrication, the optimal Pt and Ru loadings were determined based on the photocatalytic degradation of RhB under simulated visible light irradiation ($\lambda > 420$ nm) using a UV cutoff filter. The results indicated that 1.0

at.% Pt and 5.0 at.% Ru yielded the highest photocatalytic activity; these were, therefore, selected for subsequent synthesis. The $\text{In}_2\text{S}_3/\text{Pt,Ru-Ta}_2\text{O}_5$ heterostructured nanocomposites were subsequently synthesized via a hydrothermal method by varying the weight ratios of In_2S_3 to $\text{Pt,Ru-Ta}_2\text{O}_5$ (25, 50, and 75 wt.%). Pure In_2S_3 was prepared using 3.0 mM $\text{InCl}_3 \cdot 4\text{H}_2\text{O}$ as the indium precursor and thioacetamide as the sulfur source. The indium solution was added dropwise to the thioacetamide solution under continuous stirring, followed by adjusting the pH to 3.0 using 0.1 M HClO_4 . The resulting mixture was subjected to hydrothermal treatment, after which the precipitate was collected by centrifugation and dried overnight. The obtained samples were denoted as pure In_2S_3 , $\text{Pt,Ru-IT-0\%}$ ($\text{Pt,Ru-Ta}_2\text{O}_5$), $\text{Pt,Ru-IT-25\%}$, $\text{Pt,Ru-IT-50\%}$, and $\text{Pt,Ru-IT-75\%}$, with the last three corresponding to 25, 50, and 75 wt.% In_2S_3 in the composite, respectively, with pure Ta_2O_5 used as a reference. Regarding the fabrication of the films, the as-prepared powders were deposited onto conductive substrates using a doctor blading technique. Briefly, 0.5 g of the catalyst powders was mixed with glacial acetic acid and Triton X, followed by the addition of deionized water to form a homogeneous slurry. The slurry was then coated onto precleaned glass substrates ($2 \times 2 \text{ cm}^2$) via doctor blading and subsequently annealed at 350°C for 1 h. A schematic illustration of the fabrication process is provided in Figure 1.

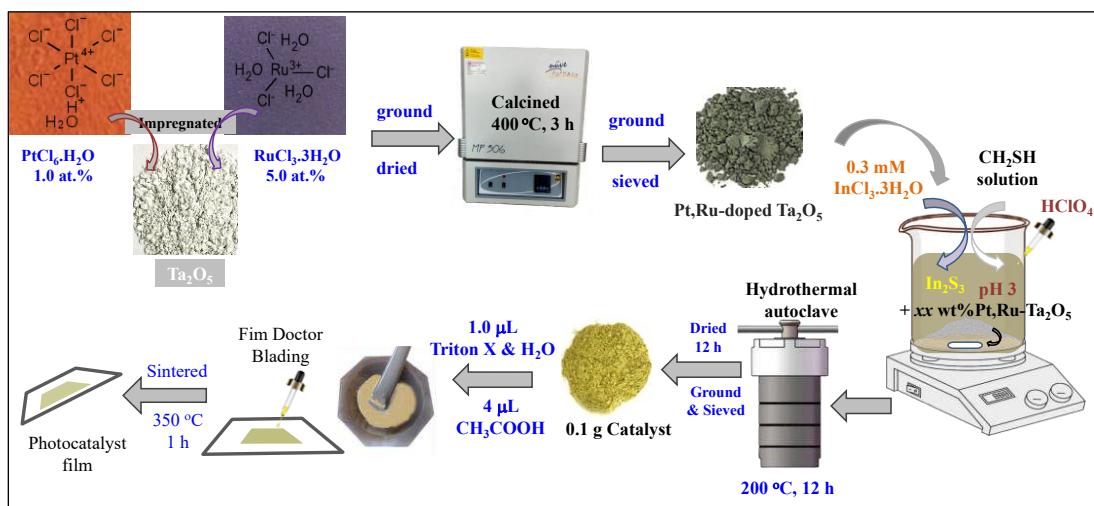


Fig. 1 Schematic illustration of nanocomposite film fabrication.

Sample characterization

The crystallographic structure and phase composition of the as-synthesized samples were analyzed using an X-ray diffractometer (Rigaku, Japan) with $\text{Cu K}\beta$ radiation ($\lambda = 1.387 \text{ \AA}$), employing a scanning range of $2\theta = 10^\circ\text{--}70^\circ$ and a step size of $0.02^\circ \text{ min}^{-1}$. UV-Vis diffuse reflectance spectroscopy (DRS) spectra were recorded in the

wavelength range of 200–800 nm using an Agilent Cary 300 spectrophotometer (USA), with BaSO₄ as the reference. The optical absorption properties and bandgap energies were subsequently determined from the DRS data. The specific surface area and pore volume were measured using an Autosorb 30 analyzer (USA) based on N₂ physisorption at 77 K after vacuum degassing. In addition, the Brunauer–Emmett–Teller (BET) surface area and pore size distribution were evaluated using a Micromeritics 3Flex surface analyzer based on the relative pressure (P/P_0) model. The surface morphology, particle size, and microstructural features were examined using field emission scanning electron microscopy (FE-SEM; JEOL JSM-7001F, Japan). High-resolution transmission electron microscopy (HR-TEM; JEOL JEM-2010 Plus, 200 kV, Japan) coupled with energy-dispersive X-ray spectroscopy (EDS) was employed to investigate the internal structure and elemental composition and spatial distribution. The lattice spacing (d-spacing) and crystallinity were further analyzed using selected area electron diffraction (SAED). Finally, the surface chemical composition and oxidation states were analyzed via X-ray photoelectron spectroscopy (XPS; PHOIBOS 100, Al $K\alpha$ radiation, 1.4 keV analyzer, Germany), and the XPS spectra were calibrated using the C 1s peak at 284.6 eV.

Photocatalytic degradation of RhB

The photocatalytic degradation efficiency of a 5.0 mg L⁻¹ RhB aqueous solution, used as a model pollutant, was evaluated using visible-light-driven photocatalyst films under a solar simulator (300 W tungsten lamp). The photocatalyst films were vertically positioned facing the incident light source at a fixed distance of 10 cm, as illustrated in Figure 2.

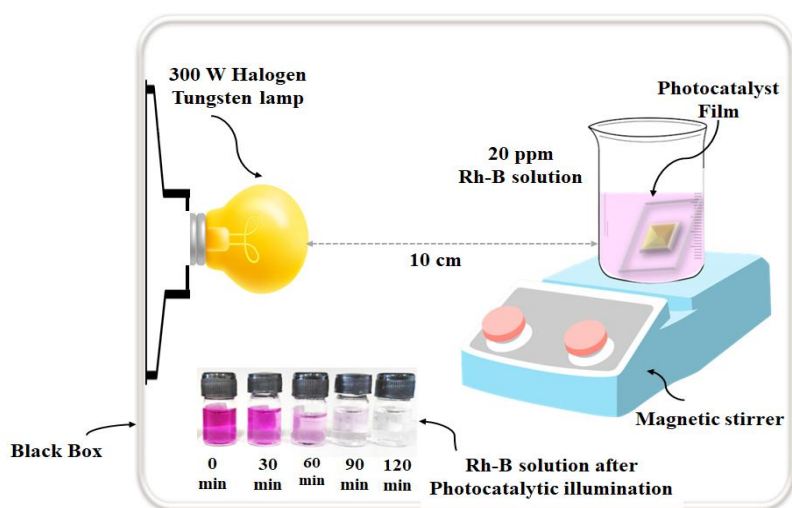


Fig. 2 Schematic illustration of the photocatalytic degradation of RhB under visible light irradiation for 120 min.

Prior to irradiation, the system was placed in the dark for 30 min in order to attain an adsorption–desorption equilibrium between the catalyst surface and RhB molecules. The photocatalytic experiments were conducted in a 100-mL reactor for a total irradiation period of 120 min. A disposable syringe was utilized to remove 4.0 mL aliquots at prearranged intervals, and a UV-Vis spectrophotometer (Shimadzu M1, Japan) with a maximum absorption wavelength of 554 nm was utilized for measurement. The photocatalytic degradation efficiency (%) of RhB was calculated according to Equation 1,

:

$$\% \text{ PC} = \frac{C_0 - C_t}{C_0} \times 100 \quad (1)$$

where C_0 is the initial concentration (mg L^{-1}), and C_t is the concentration at time t (mg L^{-1}). The apparent photocatalytic reaction rate constants were determined using a pseudo-first-order kinetic model, as expressed in Equation 2:

$$\ln \frac{C_t}{C_0} = -k_t t \quad (2)$$

where k is the apparent rate constant (min^{-1}) [18]. The stability and reusability of the photocatalyst films were evaluated through repeated photocatalytic degradation cycles under identical conditions. After each cycle, the catalyst film was washed several times with deionized water, dried, and subsequently calcined at 400 °C before reusing. To elucidate the photocatalytic mechanism, scavenger experiments were conducted using *p*-benzoquinone (*p*-BZQ), *tert*-butyl alcohol (*t*-BuOH), and ethylenediaminetetraacetic acid (EDTA) disodium salt as quenching agents for superoxide radicals ($\cdot\text{O}_2^-$), hydroxyl radicals ($\cdot\text{OH}$), and photogenerated holes (h^+), respectively.

Results and Discussion

X-ray diffraction analysis

The X-ray diffraction (XRD) patterns of Ta_2O_5 , Pt,Ru-IT-0%, Pt,Ru-IT-25%, Pt,Ru-IT-50%, Pt,Ru-IT-75%, and In_2S_3 are presented in Figure 3. All samples exhibit well-defined diffraction peaks, indicating the achievement of good crystallinity via the controlled synthesis process. The diffraction peaks of pristine Ta_2O_5 can be indexed to JCPDS No.

25-0922 [19, 20], corresponding to the characteristic planes of crystalline Ta₂O₅. In contrast, the XRD pattern of In₂S₃ corresponds well with JCPDS No. 65-0459 [21], confirming the formation of the cubic phase, with reflections at (220), (311), (222), (331), (440), (531), and (533). Regarding the doped samples, Ru-doped Ta₂O₅ exhibits an enhanced peak intensity relative to pristine Ta₂O₅, suggesting improved crystallinity. Conversely, the Pt-doped and Pt,Ru co-doped samples display a reduced peak intensity and slight peak broadening, indicating decreased crystallinity. A minor negative shift (-0.02° to -0.06°) in the diffraction peaks can be observed for the Pt-containing samples, which can be attributed to the partial substitution of Pt⁴⁺ ions into the Ta⁵⁺ lattice sites, given their comparable ionic radii (Pt⁴⁺ $\approx$ 68 pm; Ta⁵⁺ $\approx$ 70 pm) [22]. This substitution induces lattice distortion and structural disorder within the Ta₂O₅ framework. No distinct diffraction peaks corresponding to Pt, PtO_x, or RuO₂ can be detected; this is attributed to the low loading content and high dispersion of the noble metal species [23]. The Pt,Ru-IT-0% sample exhibits the lowest crystallinity, indicating that the simultaneous incorporation of Pt and Ru significantly disrupts crystal growth and lattice ordering [24]. In terms of the composite samples (Pt,Ru-IT-25%, Pt,Ru-IT-50%, and Pt,Ru-IT-75%), diffraction peaks corresponding to both the Ta₂O₅ and In₂S₃ phases can be observed, confirming the successful formation of the heterostructure. A gradual decrease in the peak intensity with an increasing In₂S₃ content suggests that the incorporation of In₂S₃ influences the crystallinity of Ta₂O₅. In addition, the absence of characteristic RuO₂ peaks ($2\theta \approx 35.3^\circ$ and 54.5°) indicates that the Ru species are either highly dispersed or incorporated into the Ta₂O₅ lattice rather than forming separate crystalline phases [25]. Similarly, the slight shift of the main diffraction peak in Pt-doped Ta₂O₅ further supports lattice distortion induced by Pt incorporation. In principle, these results indicate that noble metal doping and heterostructure formation led to decreased crystallinity and lattice distortion; these are beneficial for enhancing photocatalytic performance by promoting defect formation and facilitating charge transfer within the composite system [26].

The results demonstrate that noble metal incorporation and heterostructure formation led to lattice distortion and reduced crystallinity; these are strongly correlated with defect formation and enhanced charge transfer within the composite system, which contributes to an improved photocatalytic performance [27].

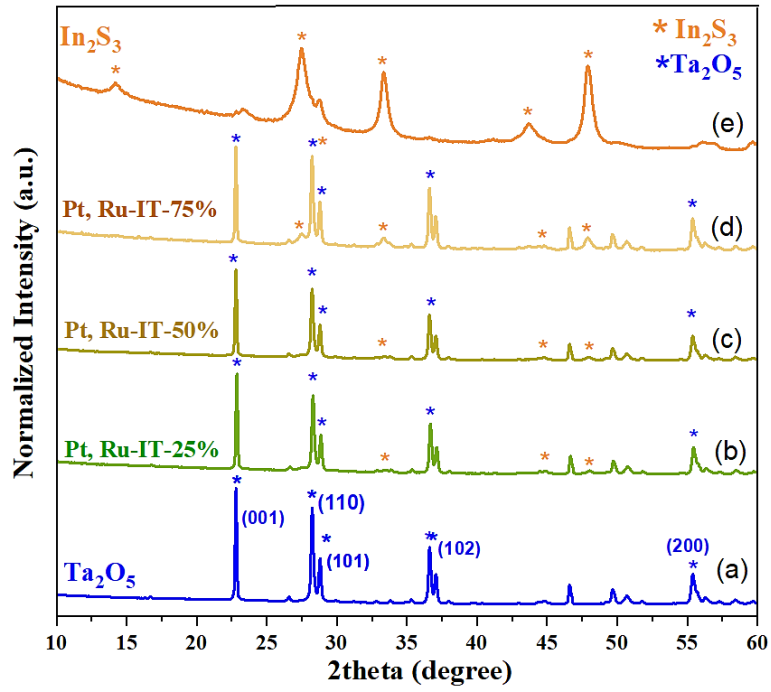


Fig. 3 X-ray diffraction patterns of the samples: (a) Ta_2O_5 , (b) Pt,Ru-IT-25%, (c) Pt,Ru-IT-50%, (d) Pt,Ru-IT-75%, and (e) In_2S_3 .

UV-Vis diffuse reflectance spectroscopy

The DRS spectra of Ta_2O_5 , Pt,Ru- Ta_2O_5 , and In_2S_3 are presented in Figure 4a, and those of the composite samples (Pt,Ru-IT-25%, Pt,Ru-IT-50%, and Pt,Ru-IT-75%) are shown in Figure 4b. A slight redshift in the absorption edge, accompanied by an extended absorption tail toward the visible light region, can be observed for the modified and composite samples. The optical absorption properties were analyzed using the Kubelka–Munk (KM) function (Equation 3), where R_∞ represents the absolute diffuse reflectance of an infinitely thick sample. The bandgap energies (E_g) were determined using the Tauc relation (Equation 4), where α is the absorption coefficient, h is Planck's constant, ν is the photon frequency, and A is a proportionality constant [28].

$$F(R_{\infty}) = \frac{(1 - R_{\infty})^2}{2R} \quad (3)$$

$$(\alpha h\nu)^{1/2} = A(h\nu - E_g) \quad (4)$$

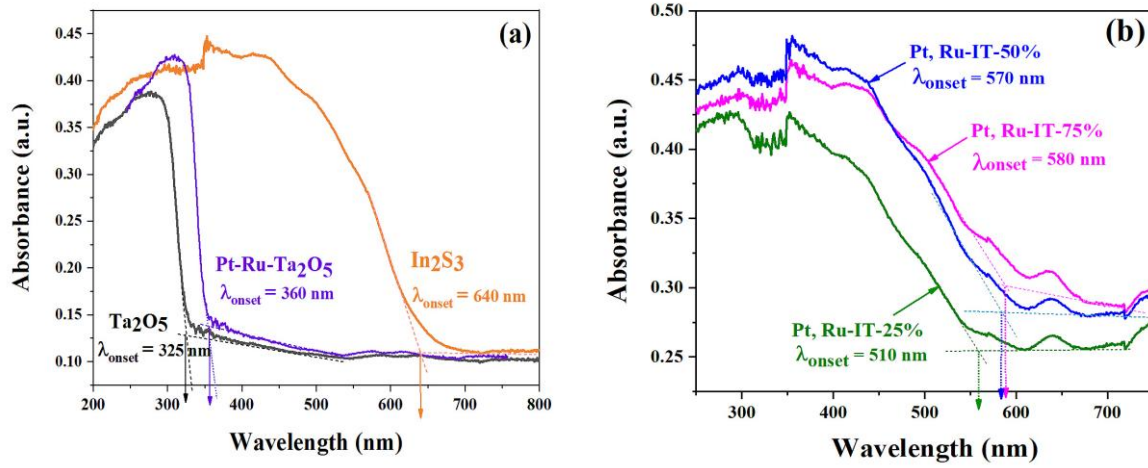


Fig. 4 UV-Vis diffuse reflectance spectra of the photocatalysts: (a) Ta₂O₅, Pt,Ru-doped Ta₂O₅, and pure In₂S₃; and (b) Pt,Ru-IT-25%, Pt,Ru-IT-50%, and Pt,Ru-IT-75%.

The exponent was taken as $\frac{1}{2}$, indicating a direct bandgap transition. The bandgap values were estimated by extrapolating the linear region of the Tauc plots to the energy axis. The calculated bandgap energies and textural properties of the prepared samples are summarized in Table 1. A pronounced redshift and bandgap narrowing can be observed upon the incorporation of In₂S₃ together with Pt and Ru species, indicating an enhanced visible light absorption ability. The reduced bandgap facilitates the utilization of lower-energy photons and promotes the production of photogenerated electron–hole pairs, thereby improving the photocatalytic activity [29]. Furthermore, pure In₂S₃ has the largest pore volume and specific surface area (S_{BET}), whereas the composite samples are arranged as follows: Pt,Ru-IT-25% < Pt,Ru-IT-50% < Pt,Ru-IT-75%. A key component for improving the photocatalytic performance is the increase in surface area that results from coupling In₂S₃ with Pt,Ru-Ta₂O₅. This increases the quantity of active sites and optimizes the adsorption–desorption processes before irradiation. Additionally, Table 1 displays the bandgap energies, S_{BET} values, pore volumes, and visual appearance of the catalyst films (Figure 5). This confirms the relationship between optical and textural characteristics and the observed photocatalytic activity, with a

strong correlation between the heterostructured samples enhanced photocatalytic activity, surface characteristics, and bandgap modulation.

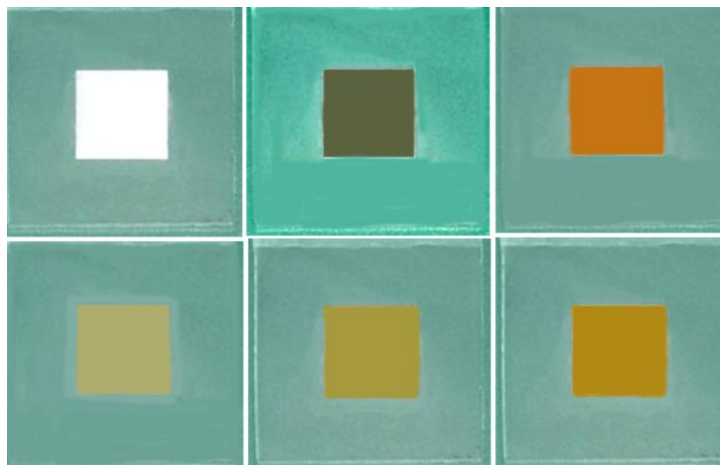


Fig. 5 Photographs of the catalyst films: (a) Ta₂O₅, (b) Pt,Ru-IT-0%, (c) Pt,Ru-IT-25%, (d) Pt,Ru-IT-50%, (e) Pt,Ru-IT-75%, and (f) In₂S₃ (1 cm²).

Table 1 Bandgap energy, S_{BET}, pore volume, and color of the samples.

Sample	E_g (eV)	SSA _{BET} (m ² ·g ⁻¹)	Pore volume (cm ³ ·g ⁻¹)	Color of films
Pure Ta ₂ O ₅	3.92	3.49	0.012	Pale white
Pt,Ru-IT-0%	3.45	4.85	0.022	Dark gray
Pure In ₂ S ₃	1.94	30.56	0.665	Fire orange
Pt,Ru-IT-25%	2.43	10.92	0.074	Pale yellow
Pt,Ru-IT-50%	2.18	11.82	0.081	Yellow
Pt,Ru-IT-75%	2.14	16.82	0.093	Mustard yellow

Microstructural examination

The morphologies of the In₂S₃, Ta₂O₅, Pt,Ru-IT-0%, and Pt,Ru-IT-75% samples were examined via scanning electron microscopy (SEM; Figure 6(a–d)). Pristine Ta₂O₅ exhibits hexagonal morphology with significant particle agglomeration and particle sizes ranging from 200 to 500 nm. In contrast, pure In₂S₃ displays a plate-like structure composed of smaller, irregularly shaped particles, with sizes in the range of 40–100 nm.

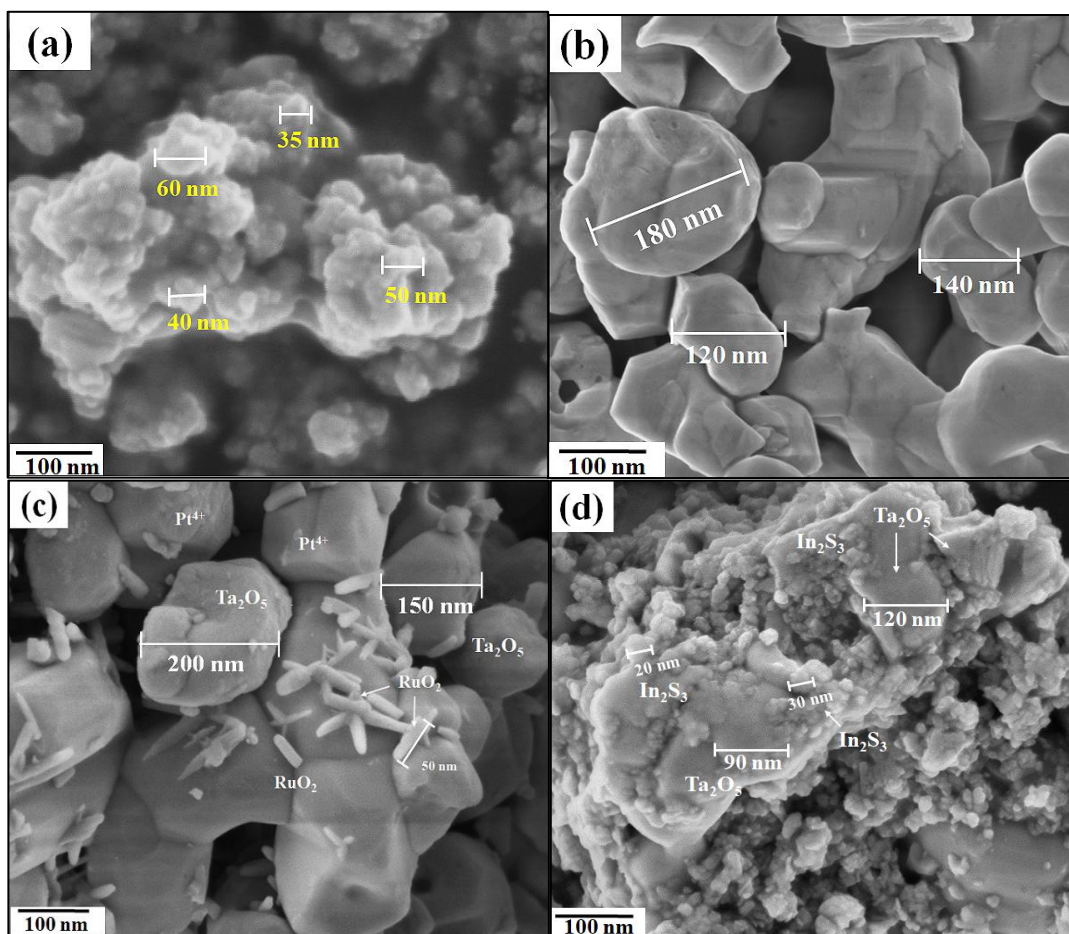


Fig. 6 FE-SEM images of (a) In₂S₃, (b) Ta₂O₅, (c) Pt,Ru-IT-0%, and (d) Pt,Ru-IT-75%.

The Pt,Ru-IT-0% sample (Figure 6c) shows a morphology comparable to that of Ta₂O₅, although the particle size is reduced to approximately 180–200 nm. Previous studies [30] have reported the presence of rod-like features on the Ta₂O₅ surface, which are attributed to the formation of RuO₂ species during annealing. The Pt,Ru-IT-75% composite (Figure 6d) exhibits a uniform distribution of fine plate-like In₂S₃ (20–40 nm) structures over the Ta₂O₅ (90–120 nm), resulting in a more dispersed and interconnected morphology. The intimate interfacial contact between In₂S₃ and Pt,Ru–Ta₂O₅ leads to a reduced particle size and improved dispersion. Such structural integration is expected to enhance the accessible surface area and increase the density of active sites while facilitating charge transport across the heterointerface. These structural features are closely associated with the enhanced photocatalytic degradation efficiency of RhB [31].

The microstructure and morphology of Ta_2O_5 , In_2S_3 , and the Pt,Ru-IT-75% nanocomposite were investigated using HR-TEM combined with EDS. While SEM provides a general estimation of the particle size, TEM enables a more precise characterization of the internal structure and morphology. The corresponding TEM and EDS results for the Pt,Ru-IT-75% sample are presented in Figure 7 and 8. As shown in Figure 7a–c, In_2S_3 exhibits a plate-like morphology, with particle sizes in the range of 15–30 nm and a relatively uniform distribution. In contrast, Ta_2O_5 displays a hexagonal morphology with a larger average particle size of approximately 120 nm. The composite sample (Fig. 7c) reveals that In_2S_3 is uniformly dispersed and firmly anchored onto the Pt,Ru– Ta_2O_5 , indicating the formation of a well-defined heterojunction interface between the two components. The SAED patterns (Figure 7d–f) provide insights into the crystallinity of the samples. Ta_2O_5 exhibits diffuse diffraction rings, indicating relatively low crystallinity, whereas In_2S_3 displays sharp and well-defined rings corresponding to the polycrystalline cubic $\beta\text{-In}_2\text{S}_3$ phase. The SAED patterns of the Pt,Ru-IT-75% composite (Figs. 7j and k) show distinct diffraction rings that can be indexed to both In_2S_3 —(220), (311), (222)—and Ta_2O_5 —(001), (100), (200)—planes, confirming the coexistence of both phases and the successful formation of the heterostructure [32].

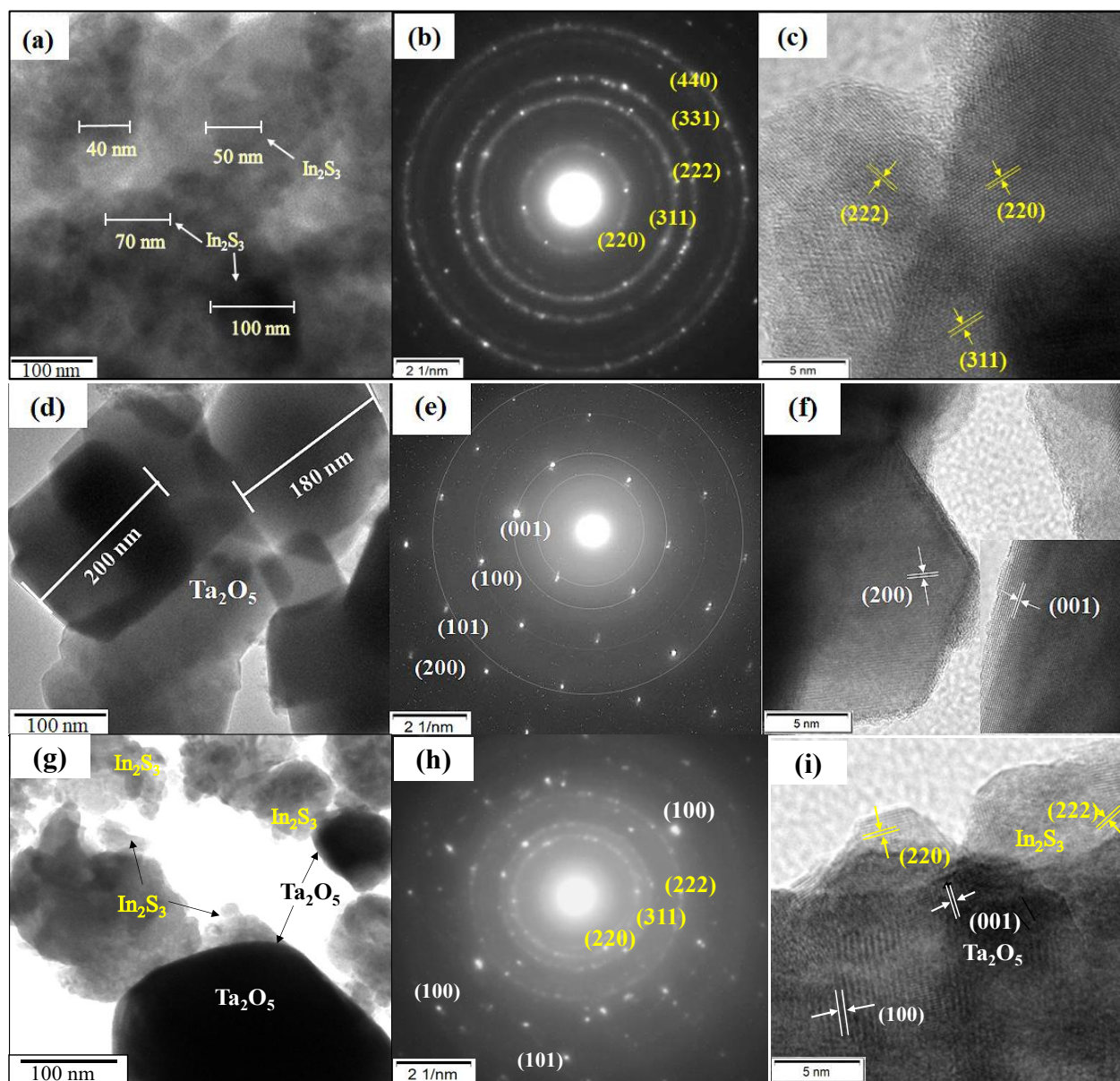


Fig. 7 HR-TEM micrographs with SAED images and lattice fringes of (a–c) In_2S_3 , (d–f) $\text{Pt,Ru-Ta}_2\text{O}_5$, and (g–i) 75% $\text{In}_2\text{S}_3/\text{Pt,Ru-Ta}_2\text{O}_5$.

The elements composition and spatial distribution within the composite were additionally verified by EDS analysis and elemental mapping (Figure 8). The elemental mapping photos indicate the uniform distribution of In and S (from In_2S_3) and Ta and O (from Ta_2O_5) throughout the sample, whereas the EDS spectrum shows the presence of Ta, O, In, S, Pt, and Ru in the Pt,Ru-IT-75% sample. These results confirm the formation of a well-defined $\text{In}_2\text{S}_3/\text{Pt,Ru-Ta}_2\text{O}_5$ heterostructure with a uniform elemental distribution, which is expected to facilitate efficient interfacial charge transfer and enhance the photocatalytic performance [33].

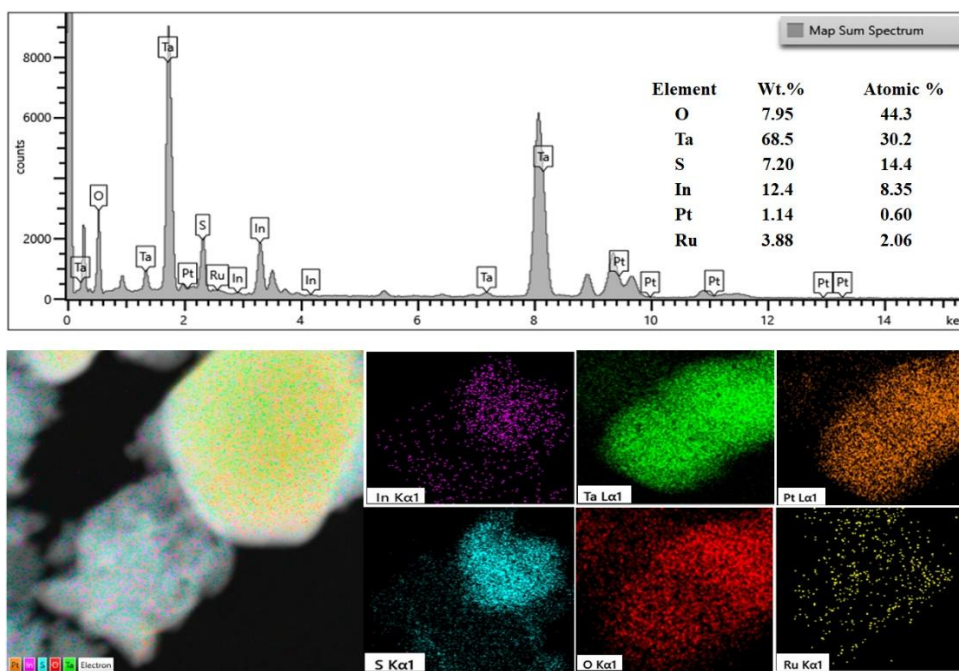


Fig. 8 EDS spectra and elemental mapping of the 75% $\text{In}_2\text{S}_3/\text{Pt,Ru-Ta}_2\text{O}_5$ heterojunction photocatalyst.

Figure 9 presents the XPS survey spectrum of the Pt,Ru-IT-75% nanocomposite, confirming the presence of Ta, O, In, S, Pt, and Ru on the surface. The elemental composition is consistent with the EDS results, verifying the successful formation of the composite system.

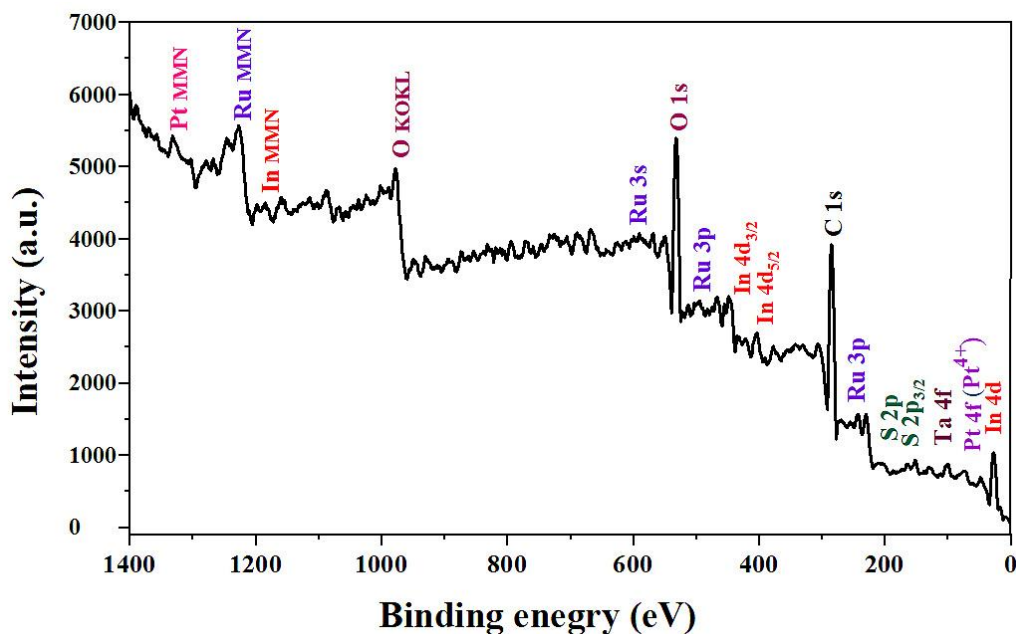


Fig. 9 XPS survey scan spectrum of the Pt,Ru-IT-75% nanocomposite.

The high-resolution Ta 4f spectrum (Figure 10a) exhibits two characteristic peaks at binding energies of 26.88 and 28.74 eV, corresponding to Ta 4f_{7/2} and Ta 4f_{5/2} of Ta₂O₅, respectively. Regarding the Pt,Ru-IT-75% composite, both peaks shift positively to 26.92 and 28.81 eV, indicating a change in the local electronic environment of the Ta species. This shift is attributed to the incorporation of Pt and Ru and the formation of Pt–O–Ta linkages, suggesting strong metal–support interactions and lattice distortion within the Ta₂O₅ framework [34]. The Ru 3p spectrum displays a characteristic peak at approximately 453.7 eV, assigned to Ru³⁺ species, confirming the incorporation of Ru into the Ta₂O₅ lattice. No significant shift in the binding energy is observed relative to the reference sample, indicating that the oxidation state of Ru remains largely unchanged [35]. Nevertheless, the homogeneous distribution of Ru, as evidenced by TEM-EDS mapping (Figure 8b), suggests its role in facilitating interfacial charge transfer. The O 1s spectrum (Figure 10b) shows a slight negative shift to approximately 529.6 eV relative to pristine Ta₂O₅, indicating an increased electron density around oxygen atoms [36]. In contrast, the Pt,Ru-IT-75% composite exhibits a positive shift, reflecting electron redistribution induced by strong interfacial interactions. These changes are attributed to the formation of Pt–O bonds and the possible substitution of Ta⁵⁺ by Pt⁴⁺, leading to lattice distortion and the generation of oxygen vacancies [37]. Such defect states are known to enhance charge separation and the efficiency of charge transfer. The In 3d spectrum (Figure 10c) exhibits two peaks at 444.7 and 452.3 eV, corresponding to In 3d_{5/2} and

In $3d_{3/2}$, respectively [38]. Both peaks exhibit positive shifts compared with pristine In_2S_3 , indicating that indium remains in the In^{3+} oxidation state while experiencing a modified electronic environment within the composite [39]. This behavior further supports interfacial charge transfer between In_2S_3 and the $\text{Pt,Ru-Ta}_2\text{O}_5$. The S 2p spectrum (Figure 10d) displays two main peaks at approximately 162.35 and 163.6 eV, corresponding to the S $2p_{3/2}$ and S $2p_{1/2}$ spin-orbit doublet of the sulfide species in In_2S_3 , respectively [40]. A weak shoulder peak at approximately 161.35 eV can also be observed. The dominant peak at 162.35 eV is attributed to disulfide (S_2^{2-}) species, whereas the lower binding energy component is assigned to monosulfide species. The observed shifts in the binding energy (+0.20 and -0.15 eV) indicate electron redistribution and lattice distortion arising from strong interfacial interactions between In_2S_3 and the $\text{Pt,Ru-modified Ta}_2\text{O}_5$ framework [41]. These results demonstrate that the shifts in the Ta 4f, O 1s, In 3d, and S 2p spectra are associated with strong interfacial electronic coupling and charge transfer within the composite system. The incorporation of Pt and Ru induces lattice distortion, electron redistribution, and oxygen vacancy formation, which collectively promote the efficient separation of photogenerated electron-hole pairs. These synergistic effects contribute to the enhanced photocatalytic degradation performance of RhB [42,43]. The Pt 4f spectrum provides further insights into the electronic state of the Pt species. The characteristic Pt $4f_{7/2}$ and Pt $4f_{5/2}$ peaks can be distinctly observed, with a slight positive shift (+0.1 eV) in the Pt $4f_{5/2}$ component, indicating that the Pt species predominantly exist in partially oxidized states. It is proposed that Pt^{4+} species act as electron acceptors and are partially reduced to Pt^{2+} , implying the coexistence of multiple oxidation states within the composite [44]. The positive shift in the binding energy suggests electron depletion at the Pt sites due to electron transfer from Pt to the Ta_2O_5 support, indicating strong metal-support interactions. Such interactions play a key role in modulating the electronic structure and facilitating charge separation. Consequently, the separation efficiency of photogenerated electron-hole pairs is enhanced, leading to improved photocatalytic activity [45]. In addition, the XPS spectra of both the pristine and composite samples show that the Ru $3p_{3/2}$ peak at approximately 453.7 eV exhibits no significant shift, indicating that the electronic state of Ru remains largely unchanged upon composite formation. Nevertheless, Ru^{3+} species are uniformly distributed throughout the material, as evidenced by TEM-EDS mapping (Fig. 8b), suggesting their contribution to interfacial charge transfer processes. Therefore, incorporating Pt and Ru species into the Ta_2O_5 synergistically enhances the photocatalytic performance by promoting efficient charge separation and interfacial charge transfer [46].

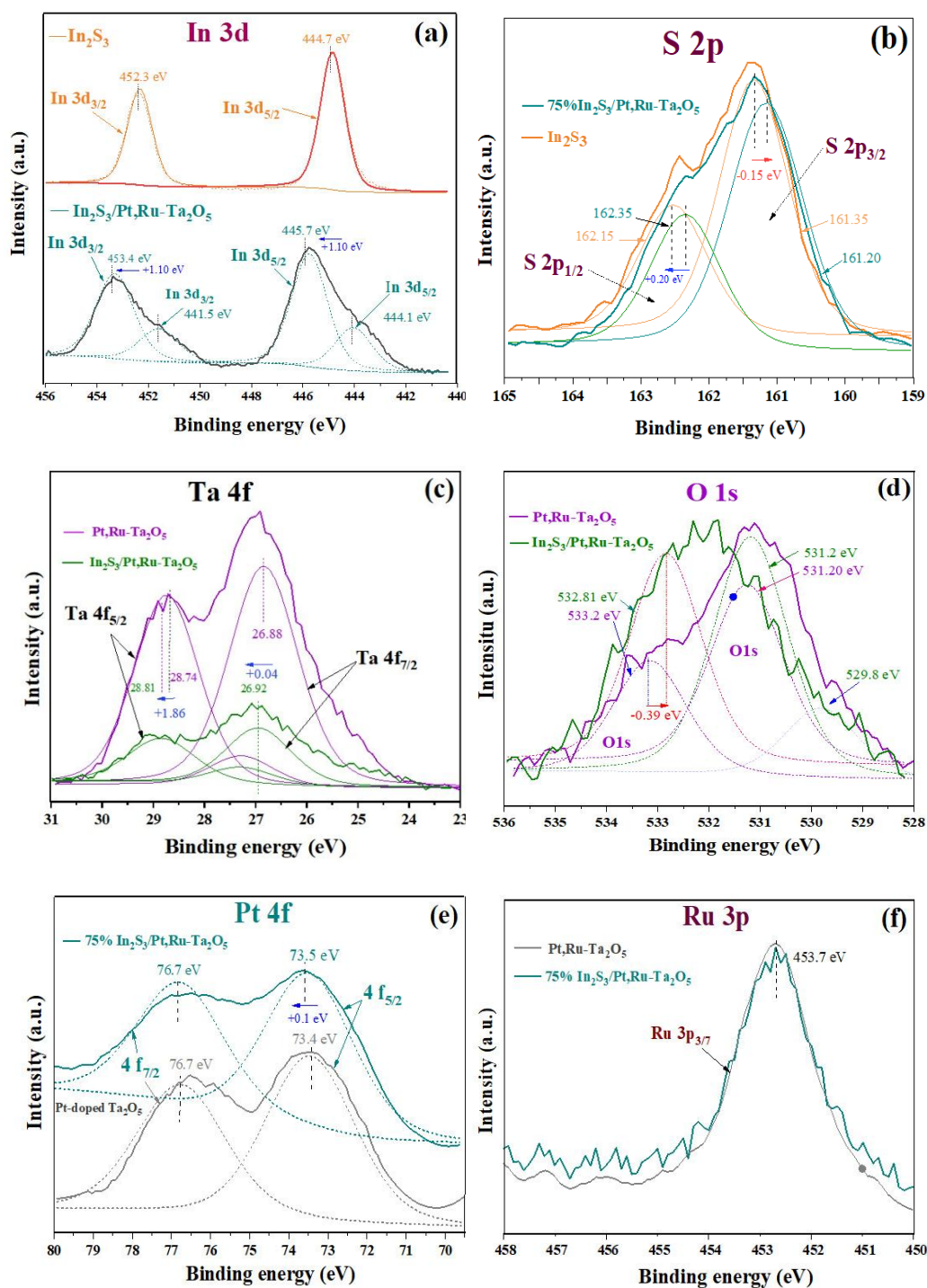


Fig. 10 XPS spectra of (a) In 3d, (b) S 2p, (c) Ta 4f, (d) O 1s, (e) Pt 4f, and (f) Ru 3p orbitals for the Pt,Ru-Ta₂O₅ and 75% In₂S₃/Pt,Ru-Ta₂O₅ composite photocatalysts.

Photocatalytic degradation of methylene blue (MB) activity

Figure 11 presents the photocatalytic degradation behavior of a 20 mg L⁻¹ methylene blue (MB) solution over various photocatalyst films under visible light irradiation, including pristine Ta₂O₅, pure In₂S₃, Pt,Ru-Ta₂O₅, and the In₂S₃/Pt,Ru-Ta₂O₅ heterostructures with different In₂S₃ loadings. Among the samples, the 75% In₂S₃/Pt,Ru-Ta₂O₅ composite exhibits the highest photocatalytic degradation efficiency (~92%) after irradiation for 120 min, significantly outperforming the pristine and single-component photocatalysts. The enhanced photocatalytic activity of the optimal heterostructure can be attributed to the synergistic integration of bandgap engineering, heterointerface formation, and noble-metal-induced electronic modulation [47]. UV-Vis diffuse reflectance analysis revealed a pronounced redshift and a narrowed bandgap, enabling enhanced visible light absorption and improved photon utilization [48]. Simultaneously, the XRD results indicated reduced crystallinity and lattice distortion after Pt/Ru incorporation, suggesting defect generation and structural disorder favorable for charge separation. SEM and TEM morphological investigations further demonstrated the formation of well-dispersed plate-like In₂S₃ nanostructures anchored onto the Pt,Ru-Ta₂O₅, providing enhanced interfacial contact and shortened charge migration pathways [49]. Furthermore, XPS analysis confirmed strong electronic interactions among In₂S₃, Pt/Ru species, and Ta₂O₅ through binding-energy shifts and electron redistribution [50]. The formation of oxygen vacancies and partially oxidized Pt species is believed to facilitate electron trapping and suppress electron-hole recombination. Such interfacial electronic coupling effectively promotes charge carrier separation and accelerates the generation of reactive oxygen species during photocatalysis [51,52].

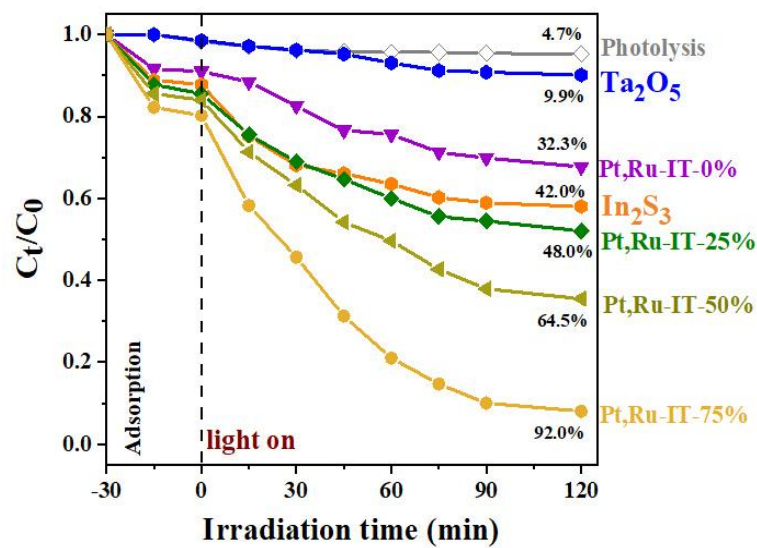


Fig. 11 Photocatalytic RhB degradation efficiencies of various catalyst films under visible light irradiation in comparison with the photolysis process.

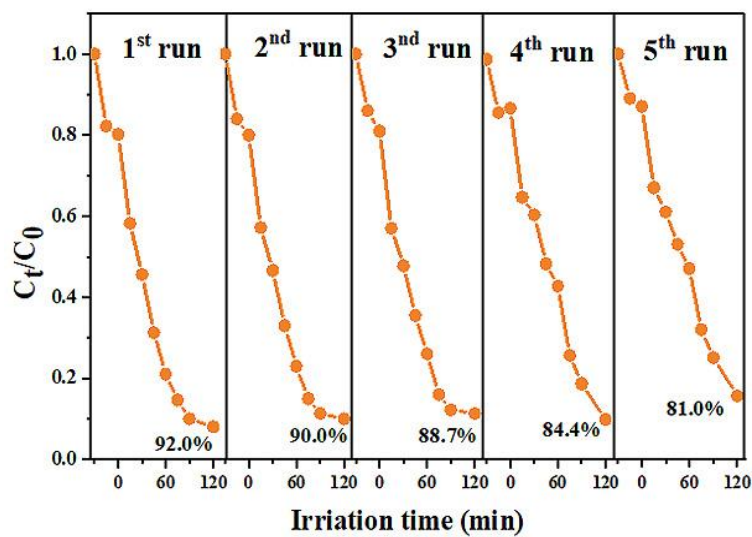


Fig. 12 Cycling performance of the synthesized Pt,Ru-IT-75% catalyst film for MB photodegradation over five consecutive runs.

Figure 12 illustrates the cycling stability of the optimal Pt,Ru-IT-75% photocatalyst film over five successive photocatalytic degradation runs under visible light irradiation. The degradation efficiency exhibits only a slight decline from 92.0% to 81.0% after five cycles, indicating favorable structural stability and good resistance against photocorrosion during prolonged photocatalytic operation [53]. The retained photocatalytic performance suggests that the strong interfacial interaction between In_2S_3 and Pt,Ru- Ta_2O_5 effectively preserves the integrity of the heterostructure and suppresses catalyst deactivation [54]. These results demonstrate that the rationally designed $\text{In}_2\text{S}_3/\text{Pt,Ru-Ta}_2\text{O}_5$ heterojunction possesses excellent photocatalytic durability and considerable potential for visible-light-driven environmental remediation applications [55]. To further investigate the photocatalytic mechanism of the optimal catalyst, scavenger experiments were conducted to identify the dominant reactive species involved in MB degradation, as shown in Figure 13. *p*-BZQ, *t*-BuOH, and EDTA were employed as scavengers for h^+ , $\cdot\text{O}_2^-$, and $\cdot\text{OH}$, respectively [56]. The addition of scavengers exhibits no significant impact on the adsorption process; however, a considerable reduction in the photocatalytic degradation efficiency was detected. The addition of *p*-BZQ significantly reduces the MB breakdown efficiency, from 92.0% to 34.1%, suggesting that $\cdot\text{O}_2^-$ radicals are the most reactive species in the photocatalytic process [57]. This highlights that Pt and Ru species effectively trap photogenerated electrons in the conduction band, allowing electron transport to dissolved oxygen and increasing the generation of $\cdot\text{O}_2^-$ radicals while suppressing electron-hole recombination [58]. Furthermore, the addition of *t*-BuOH and EDTA reduces the degradation efficiency to approximately 67.5% and 47.2%, respectively, confirming that h^+ and $\cdot\text{OH}$ species also contribute to MB degradation. These results demonstrate that multiple reactive species participate in the photocatalytic process, although $\cdot\text{O}_2^-$ radicals play the dominant role. The enhanced photocatalytic activity can, therefore, be attributed to the synergistic interaction between In_2S_3 and Pt,Ru- Ta_2O_5 , which promotes efficient charge separation and electron migration [59, 60]. Incorporating In_2S_3 broadens the visible light absorption and improves photogenerated electron production. Additionally, Pt and Ru act as electron-trapping centers, facilitating the production of reactive oxygen species [61]. Simultaneously, photogenerated holes remaining in the valence band participate in oxidation reactions associated with photocatalytic degradation, collectively contributing to the efficient decomposition of MB molecules [62].

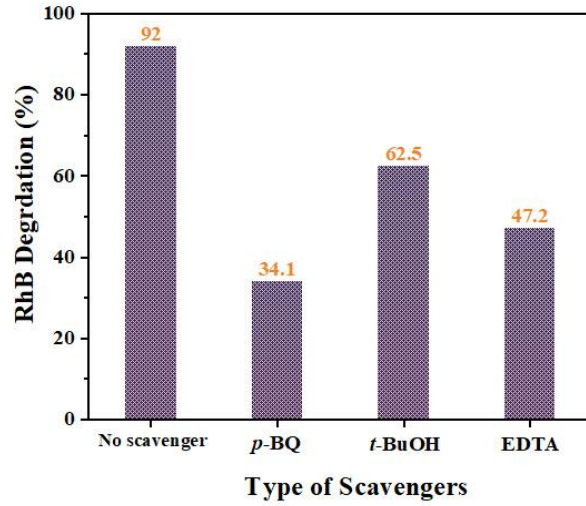


Fig. 13 Effect of different scavengers on the photocatalytic degradation of MB over the optimal photocatalyst.

Figure 14 presents the proposed interfacial charge separation and transport mechanism of the Pt,Ru-doped In₂S₃/Ta₂O₅ heterojunction upon visible light irradiation. The conduction band (CB) and valence band (VB) potentials of Ta₂O₅ and In₂S₃ can be calculated using Equation 5 [63]:

$$E_{CB}^0 = \chi - E^C - \frac{1}{2} E_g \quad (5)$$

The absolute electronegativities (χ) of Ta₂O₅ and In₂S₃ were taken as approximately +5.72 and +4.82 eV, respectively. In the band edge calculations, E^C represents the energy of free electrons on the hydrogen scale relative to the absolute vacuum scale (AVS), where 0 V versus NHE corresponds to +4.5 eV versus AVS [64]. The bandgap energies (E_g) of Ta₂O₅ and In₂S₃, as determined from the present study, are +3.45 and +1.94 eV, respectively. The calculated CB and VB edge potentials of Ta₂O₅ are approximately −0.51 and +2.95 V versus NHE, respectively, whereas In₂S₃ exhibits CB and VB positions of −0.65 and +1.29 V, respectively. Since the CB potential of In₂S₃ is more negative than that of Ta₂O₅, the photogenerated electrons in In₂S₃ can be readily transferred to the CB of Ta₂O₅ through the close interfacial contact between the two semiconductors, which is consistent with the SEM and TEM morphological observations [65].

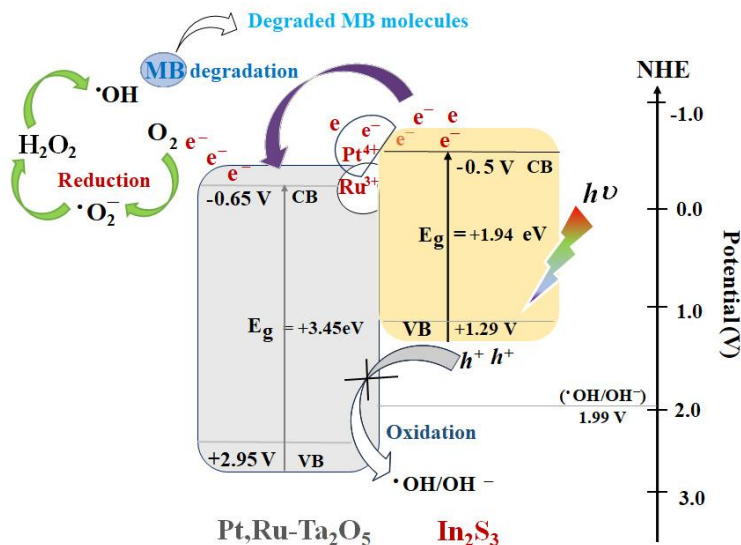


Fig. 14. Proposed Z-scheme heterojunction charge-transfer mechanism of the $\text{In}_2\text{S}_3/\text{Pt,Ru-Ta}_2\text{O}_5$ photocatalyst under visible light irradiation.

The improved photocatalytic performance of the Pt,Ru-modified $\text{In}_2\text{S}_3/\text{Ta}_2\text{O}_5$ heterostructure is strongly associated with the synergistic effect of heterojunction formation and the presence of Pt and Ru species acting as electron-trapping sites. These metallic species effectively capture photogenerated electrons, suppressing the rapid recombination of electron-hole pairs and promoting interfacial charge separation. The accumulated electrons can subsequently react with dissolved oxygen molecules to generate reactive oxygen species, particularly $\cdot\text{O}_2^-$, which are considered the dominant active species responsible for the degradation of MB molecules. In addition, the generated $\cdot\text{O}_2^-$ radicals may further participate in secondary oxidation processes, contributing to the formation of highly reactive oxidative species during photocatalysis. These reactive species can effectively decompose MB into smaller intermediate compounds and ultimately mineralize them into harmless end products, such as CO_2 and H_2O . However, the valence band potential of In_2S_3 (+1.29 V vs. NHE) is less positive than the standard oxidation potential of $\cdot\text{OH}/\text{OH}^-$ (+1.99 V vs. NHE) [66], indicating that the photogenerated holes in the VB of In_2S_3 are thermodynamically incapable of directly oxidizing OH^- or H_2O to generate $\cdot\text{OH}$ radicals. Therefore, the enhanced photocatalytic degradation efficiency observed in this work is primarily governed by reduction reactions occurring at the heterojunction interface rather than by direct hole oxidation pathways. The efficient migration and separation of photogenerated charge carriers within the heterostructure play a key role in enhancing the visible-light photocatalytic degradation of MB.

Conclusions

In this study, a visible-light-responsive $\text{In}_2\text{S}_3/\text{Pt,Ru-Ta}_2\text{O}_5$ heterostructured catalyst film was fabricated and systematically investigated. The optimized $\text{In}_2\text{S}_3/\text{Pt,Ru-Ta}_2\text{O}_5$ film, with a 75% In_2S_3 content, exhibits superior photocatalytic performance, achieving approximately 92.0% RhB degradation within 120 min under visible light irradiation. This enhanced activity is attributed to the synergistic effects of heterojunction formation, noble metal incorporation, and an increased surface area. Structural and morphological analyses confirm the formation of a well-defined interface, which facilitates efficient interfacial charge transfer. Optical characterization reveals a narrowed bandgap and enhanced visible light absorption, promoting the effective generation of electron-hole pairs. XPS further highlights strong metal-support interactions and electron redistribution, leading to the formation of oxygen vacancies and an improved charge separation efficiency. Scavenger experiments confirm that $\cdot\text{O}_2^-$ radicals are the dominant reactive species, highlighting the critical role of electron transfer pathways in the photocatalytic process. In addition, the catalyst film exhibits good stability and reusability over 5 cycles. The rational integration of In_2S_3 with Pt,Ru- Ta_2O_5 provides an effective strategy for designing high-performance visible-light-driven photocatalysts. These findings offer valuable insights into heterostructure engineering and electronic structure modulation for advanced photocatalytic systems. In addition, further optimization of the defect density and noble metal dispersion is expected to enhance the electronic structure and charge carrier dynamics of the composite system. Scaling up the catalyst film architecture and improving its stability under long-term operation also remain important considerations for practical applications.

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Competing interests The authors declare no competing interests.

Data Availability Statement

All generated and analyzed data in this research included within the article are free of ethical and usage constraints.

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