

In₂S₃/Halloysite Nanocomposite as a Highly Efficient Photocatalyst for Oxidative Desulfurization of Dibenzothiophene in Fuel

PHAM Xuan Nui^{1,*}, TRAN Viet Hoang¹, HOANG Phuong Anh¹, NGUYEN T.-Thanh-Bao²

¹ Department of Chemical Engineering, Hanoi University of Mining and Geology, Ha Noi, Vietnam

² Institute of Physics, Vietnam Academy of Science and Technology, Hanoi, Vietnam

* Corresponding email: phamxuannui@humg.edu.vn

Abstract: Sulfur-containing organic compounds present in transportation fuels such as gasoline and diesel are a major source of environmental pollution, primarily due to the emission of sulfur oxides (SO_x) during combustion. In response, increasingly stringent global regulations have been implemented to limit sulfur content in fuels. In this study, a nanocomposite photocatalyst composed of indium sulfide (In₂S₃) and halloysite was synthesized via a two-step hydrothermal process. The structural, morphological, and optical properties of the resulting photocatalysts were characterized using X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FT-IR), scanning electron microscopy (SEM), and UV–Vis diffuse reflectance spectroscopy (UV–Vis DRS). The photocatalytic performance of the In₂S₃/Halloysite composite was evaluated through the oxidative desulfurization of dibenzothiophene (DBT) under visible light irradiation. Under optimized conditions—reaction temperature of 70 °C, 1.0 mL H₂O₂ as the oxidant, and 40 mg of photocatalyst—the DBT conversion reached 96% after 5 hours. These results highlight the potential application of In₂S₃/Halloysite nanocomposites as efficient visible-light-responsive photocatalysts for sulfur removal from fuels.

Keywords: Dibenzothiophene; In₂S₃; Halloysite; Photocatalysis; Oxidative desulfurization

1. Introduction

Currently, the application of sulfur removal technologies from fossil fuels has become a central focus in the petroleum refining and petrochemical industries. The elimination of sulfur-containing compounds is increasingly urgent to meet the recommended sulfur content of 10–15 ppm in diesel fuel, as established by the United States Environmental Protection Agency (USEPA) (Tanimu et al., 2019). Among these technologies, hydrodesulfurization (HDS) remains the only commercially implemented process and is considered the standard technology in modern refineries. However, HDS operates under harsh conditions, typically requiring high temperatures (300–400 °C) and elevated hydrogen pressures (150–250 psi H₂). Under such conditions, undesirable side reactions can occur, including hydrocracking and coke formation due to condensation reactions, which lead to catalyst deactivation. Furthermore, the presence of refractory sulfur-containing compounds such as thiophene (TH), benzothiophene (BT), dibenzothiophene (DBT), and their derivatives presents a significant challenge, as these compounds are chemically stable and require more severe operational conditions for effective removal (Castro et al. 2024).

The adsorptive desulfurization system (ADS) allows direct removal of sulfur compounds under mild conditions using adsorbents such as nanoporous carbons (Seredych et al., 2010), silica (Nasab et al., 2014), zeolites (Wang et al., 2009), metal–organic frameworks (MOFs) (Wang et al., 2015), natural clay minerals (Cuevas et al., 2019), and polymer-based compounds like polystyrene sulfonate or poly(allyldimethylammonium chloride) (Qin et al., 2009). Among these materials, clay minerals have garnered increasing attention due to their high adsorption capacity, non-toxic nature, wide availability, and low cost (Ramanayaka et al., 2020). A wide range of clays including kaolinite, bentonite, and zeolites have been extensively utilized for the adsorption of pollutants from aqueous media.

Halloysite nanotubes (HNTs), a naturally occurring aluminosilicate clay of the kaolinite group, exhibit a unique tubular morphology with the molecular formula Al₂Si₂O₅(OH)₄·nH₂O. The tubular structure of HNTs comprises an inner surface dominated by Al–OH groups and an outer surface rich in Si–O–Si groups. When dispersed in aqueous solutions over a broad pH range (pH 3–8), HNTs demonstrate distinct charge separation - possessing a positively charged inner lumen and a negatively charged outer surface. Zhang et al. reported the synthesis of an In₂O₃/HNT composite via a simple hydrothermal method, which was applied as an efficient adsorbent for tetracycline removal. However, the use of such adsorbents

for the removal of sulfur-containing compounds typically requires significant pre-treatment or modification to achieve desirable adsorption performance (Cavallaro et al., 2018).

Oxidative desulfurization (ODS) has emerged as a promising alternative to the conventional HDS process. ODS typically proceeds in two stages: (i) oxidation of sulfur-containing compounds and (ii) subsequent removal of the resulting oxidized products using suitable solvents or adsorbents. This method offers several advantages, including mild reaction conditions, shorter processing times, lower operational costs, and the absence of hydrogen usage. As a result, ODS is considered an environmentally benign approach for sulfur removal. In particular, photocatalytic oxidative desulfurization under visible light irradiation has gained attention as a sustainable process that utilizes light energy to activate photocatalysts for the oxidation of refractory sulfur species in fuels (Abdollahi et al., 2022). During photocatalytic ODS, sulfur compounds such as thiophenes are selectively oxidized to more polar products—sulfoxides and sulfones—which can be efficiently extracted or adsorbed from the reaction mixture using appropriate solvents or adsorbents (Hitam et al., 2019; Pham et al., 2020).

Indium sulfide (In_2S_3) is a well-known chalcogenide semiconductor photocatalyst belonging to the group III–VI compounds with n-type conductivity. Under atmospheric pressure, In_2S_3 crystallizes in three polymorphic forms: α , β , and γ . Among them, β - In_2S_3 exhibits a relatively narrow bandgap (2.0–2.4 eV), along with excellent photoresponsiveness, photoconductivity, and favorable chemical and physical properties. These characteristics make In_2S_3 an ideal candidate for use as a dopant or active component to enhance the photocatalytic performance of various framework materials (Gao et al., 2025; Li et al., 2017). In recent years, several In_2S_3 -based composite photocatalysts have been synthesized and demonstrated high photocatalytic activity under visible-light irradiation. Notable examples include $\text{In}_2\text{S}_3/\text{BiPO}_4$ heterojunctions (Zhang et al., 2018), $\text{In}_2\text{S}_3@\text{MIL-125}(\text{Ti})$ (Wang et al. 2016), and $\text{CdIn}_2\text{S}_4/\text{In}_2\text{S}_3$ heterojunctions (Hu et al., 2023).

Based on the distinctive structural features of In_2S_3 and the intrinsic advantages of halloysite nanotubes, a novel strategy is proposed in this study to synthesize a visible-light-active $\text{In}_2\text{S}_3/\text{HNT}$ nanocomposite photocatalyst for the oxidative desulfurization of dibenzothiophene in model fuels. The structural and optical properties of the synthesized nanocomposites were systematically characterized. Furthermore, the effects of various operational parameters, including catalyst dosage, DBT concentration, reaction temperature, and oxidant amount, were thoroughly investigated to evaluate the catalytic performance of the system.

2. Experimental

2.1. Materials

Indium(III) chloride tetrahydrate ($\text{InCl}_3 \cdot 4\text{H}_2\text{O}$, AR), thiourea ($(\text{NH}_2)_2\text{CS}$, AR), halloysite nanotubes (HNTs, AR), dibenzothiophene (DBT, AR), absolute ethanol ($\text{C}_2\text{H}_5\text{OH}$, AR), *n*-octane ($n\text{-C}_8\text{H}_{18}$, AR), and acetonitrile (CH_3CN , AR) were all purchased from Sigma-Aldrich and used without further purification.

2.2. Synthesis of In_2S_3

A 60 mL mixed aqueous solution containing 5 mM indium(III) chloride tetrahydrate ($\text{InCl}_3 \cdot 4\text{H}_2\text{O}$) and 25 mM thiourea ($(\text{NH}_2)_2\text{CS}$) was stirred magnetically for 1 h to obtain a homogeneous solution, followed by ultrasonication for 15 minutes. The resulting solution was transferred to a Teflon-lined stainless-steel autoclave and heated at 140 °C for 18 h. After natural cooling to room temperature, an orange precipitate of In_2S_3 was collected, washed several times with deionized water and absolute ethanol, and dried under vacuum at 70 °C for 12 h. The resulting powder was then ground and calcined in air at 400 °C for 3 h with a heating rate of 5 °C/min, yielding orange In_2S_3 powder.

2.3. Synthesis of 40% $\text{In}_2\text{S}_3/\text{HNTs}$ Composite

The 40% $\text{In}_2\text{S}_3/\text{HNTs}$ (w/w) composite was synthesized via a one-step hydrothermal method. In this process, 30 mL of 5 mM $\text{InCl}_3 \cdot 4\text{H}_2\text{O}$ solution was mixed with 30 mL of 25 mM thiourea solution under magnetic stirring. A predetermined amount of halloysite nanotubes was then added to the mixture under vigorous stirring. The final mixture was transferred to a Teflon-lined stainless-steel autoclave and maintained at 140 °C for 18 h. The resulting product was collected by centrifugation, washed several times with deionized water and ethanol, and dried at 70 °C for 12 h. The dried powder was subsequently ground and calcined in air at 400 °C for 3 h at a heating rate of 5 °C/min to obtain the 40% $\text{In}_2\text{S}_3/\text{HNTs}$ composite.

2.4. Characterization

The crystal structures of the synthesized samples were analyzed using X-ray Diffraction (XRD, Bruker D8 Advance diffractometer) with Cu K α radiation. Fourier-transform infrared (FT-IR) spectra were recorded using a Jasco 4700 spectrometer in the wavenumber range of 4000–400 cm⁻¹ using the KBr pellet technique. Surface morphology was observed via Scanning Electron Microscopy (SEM, HITACHI S-4800-3000F). UV–Vis Diffuse Reflectance Spectra (DRS) were collected using a UV-2600 spectrophotometer (Shimadzu) in the wavelength range of 200–800 nm.

2.5. Evaluation of photocatalytic activity of In₂S₃/HNTs

In each photocatalytic desulfurization experiment, 40 mg of the catalyst was dispersed in 40 mL of a DBT solution (250 ppm of dibenzothiophene dissolved in *n*-octane). The resulting suspension was magnetically stirred at 600 rpm in the dark for 1 hour to achieve adsorption–desorption equilibrium between DBT and the catalyst surface. After the dark adsorption phase, the suspension was irradiated under visible light ($\lambda > 400$ nm). A 300 W xenon lamp equipped with a 400 nm cutoff filter with light intensity of 106 mW/cm² as the visible-light source. The light source was positioned 15 cm above the surface of the reaction solution. The photocatalytic reactions were carried out under different conditions, including variations in catalyst type, oxidant dosage, and reaction temperature.

At regular intervals (every 1 h), 4 mL aliquots of the reaction mixture were withdrawn to determine the DBT concentration. Prior to analysis, each sample was centrifuged at 4000 rpm for 10 minutes and filtered through a 0.22 μ m PTFE membrane filter to remove solid particles. Following centrifugation and filtration, acetonitrile (used as the extracting solvent) was introduced to carry out the extraction. The concentration of DBT was measured using UV–Vis spectroscopy at its maximum absorption wavelength of 325 nm. The DBT conversion was calculated using the following equation:

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

where C_0 is the initial concentration of DBT and C_t is the concentration at time t .

3. Results and Discussion

3.1. Materials Characterization

The composition and purity of the catalyst materials were investigated by XRD. As shown in Fig. 1, the XRD patterns of HNTs and In₂S₃ correspond well with the standard reference patterns (JCPDS No. 09-0453 for HNTs and JCPDS No. 32-0456 for In₂S₃). The diffraction peaks observed at 12.2°, 20°, 24.9°, 32.5°, and 62.7° are attributed to the (001), (100), (002), (110), and (300) lattice planes of HNTs, respectively. For In₂S₃, characteristic peaks appear at 27.6°, 33.2°, and 47.8°, corresponding to the (311), (400), and (440) crystal planes of the cubic β -In₂S₃ phase (Fu et al., 2010).

Compared to pure In₂S₃, several additional peaks at 12.2°, 20°, and 24.9°, which are characteristic of HNTs, are observed in the In₂S₃/HNTs composite. Although the peak intensities in the composite are lower than those in the pure In₂S₃ and HNTs samples, all diffraction peaks are well matched with the individual crystalline phases of In₂S₃ and HNTs. This confirms the successful synthesis of the In₂S₃/HNTs composite material.

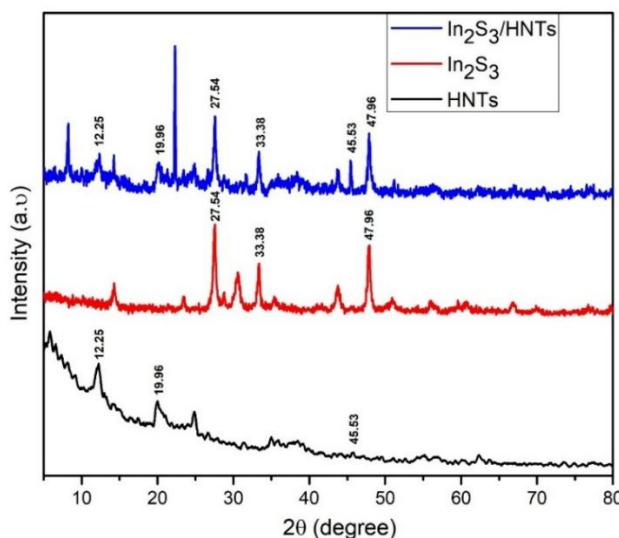


Fig. 1. XRD patterns (a) HNTs and (b) In_2S_3 và composite 40% In_2S_3 /HNTs composite

The FT-IR spectra of In_2S_3 , HNTs, and 40% In_2S_3 /HNTs were recorded to further confirm the coexistence of In_2S_3 and HNTs, as illustrated in Fig. 2. The characteristic peak at 848 cm^{-1} corresponds to the vibrational mode of In-S bonds (Parhizkar et al. 2020; Timoumi et al., 2020), while the peak observed at 487 cm^{-1} is attributed to the In-O stretching vibration. The presence of the In-O vibration is ascribed to the interaction between In^{3+} ions and oxygen-containing surface functional groups (Yuan et al., 2019).

For HNTs, the absorption bands at 3689 and 3623 cm^{-1} are attributed to the O-H stretching vibrations of Al-OH groups. The bands at 1021 and 1118 cm^{-1} correspond to the stretching vibrations of Si-O bonds. Additionally, the bands at 904 and 510 cm^{-1} are associated with the bending vibration of Al-OH and the deformation vibration of Al-O-Si, respectively. In the FT-IR spectrum of 40% In_2S_3 /HNTs, the characteristic peak at 848 cm^{-1} corresponds to the In-O bond, confirming the presence of In_2S_3 . Notably, the characteristic peaks of Si-O and Al-OH shift from 1118 and 904 cm^{-1} to 1123 and 907 cm^{-1} , respectively, indicating interfacial interactions between In_2S_3 and HNTs (Zhang et al., 2021).

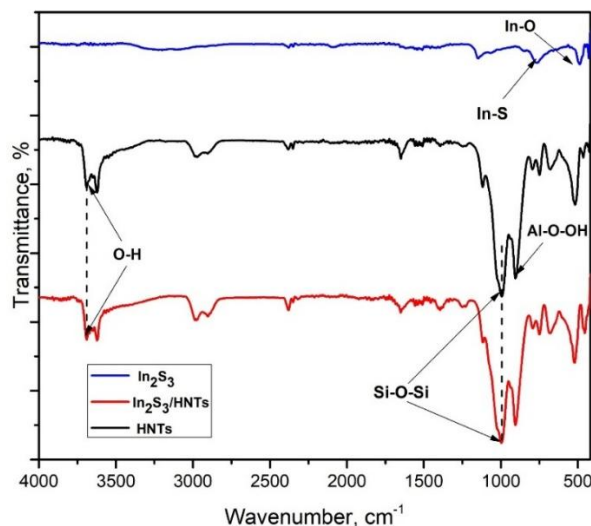


Fig. 2. FT-IR spectra of the pure In_2S_3 , HNTs, 40% In_2S_3 /HNTs composite

Fig. 3 presents the morphological structures of HNTs, In_2S_3 , and 40% In_2S_3 /HNTs as observed by scanning electron microscopy (SEM). As shown in Fig. 3a,b the HNTs exhibit a tubular structure with characteristic hollow interiors. Fig. 3c,d illustrates the SEM image of pure In_2S_3 , revealing an irregularly stacked nanosheet-like morphology. In the SEM image of 40% In_2S_3 /HNTs (Fig. 3e,f), the In_2S_3 nanosheets are closely attached to the HNTs, and the incorporation of HNTs significantly improves the dispersion of In_2S_3 sheets. This enhanced dispersion is beneficial for improving photocatalytic efficiency under visible light irradiation.

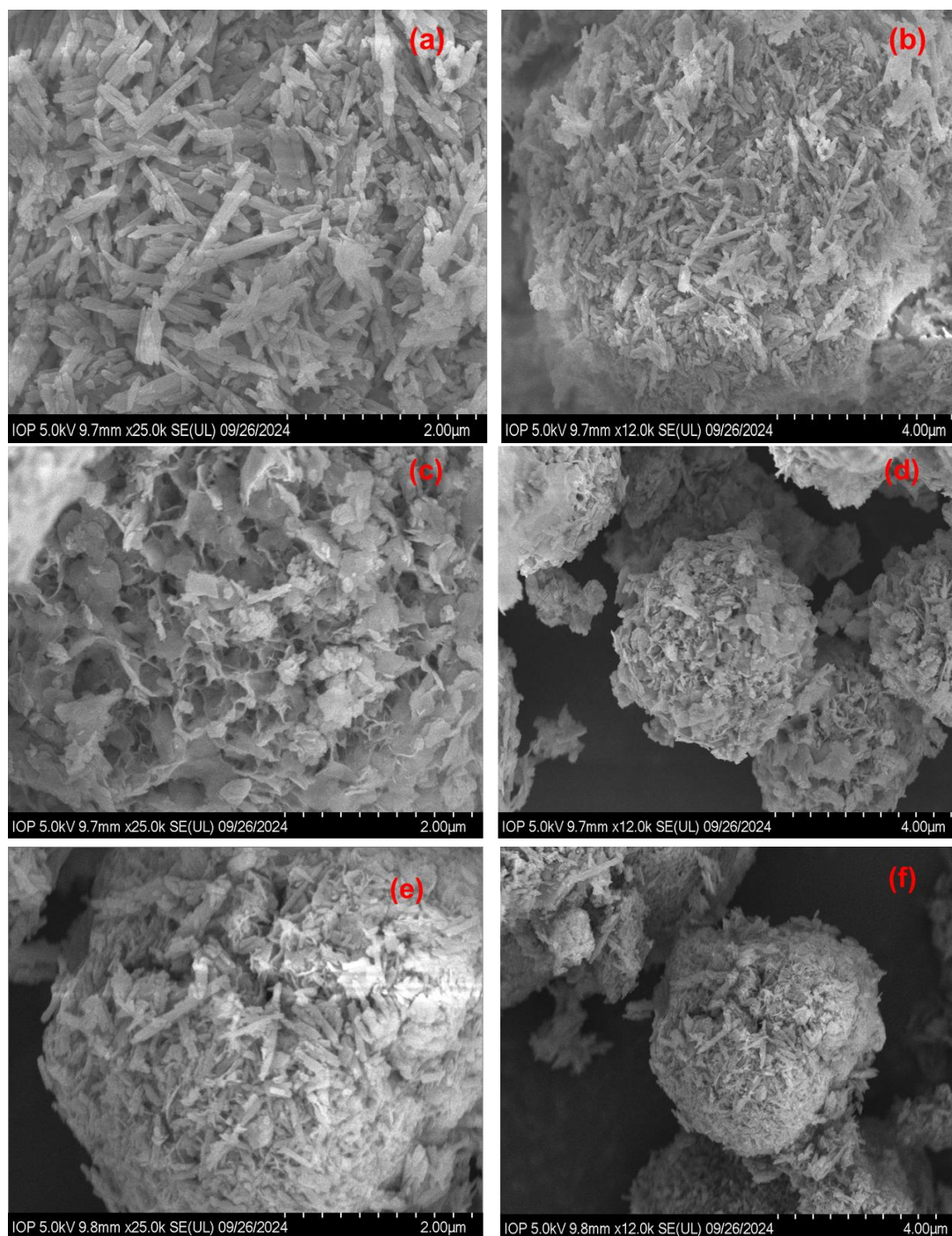


Fig. 3. SEM images of (a,b) HNTs, (c,d) In_2S_3 , and (e,f) $\text{In}_2\text{S}_3/\text{HNTs}$ composite

Fig. 4 displays the optical absorption spectra of In_2S_3 and $\text{In}_2\text{S}_3/\text{HNTs}$ materials, recorded in the wavelength range of 300–900 nm using UV–Vis diffuse reflectance spectroscopy (DRS). The bandgap energy (E_g) of the synthesized materials was determined using Tauc plots of $(\alpha h\nu)^{1/m}$ versus $h\nu$, where α is the absorption coefficient (taken as 10^4 cm^{-1}), h is Planck's constant, and ν is the photon frequency. The electronic transition was identified as a direct transition ($m=1/2$). The bandgap energies were estimated by extrapolating the linear portion of the $(\alpha h\nu)^2$ versus $h\nu$ plot to the energy axis (Figure 5). The calculated bandgap energies of In_2S_3 and $\text{In}_2\text{S}_3/\text{HNTs}$ were approximately 1.83 eV and 1.74 eV, respectively.

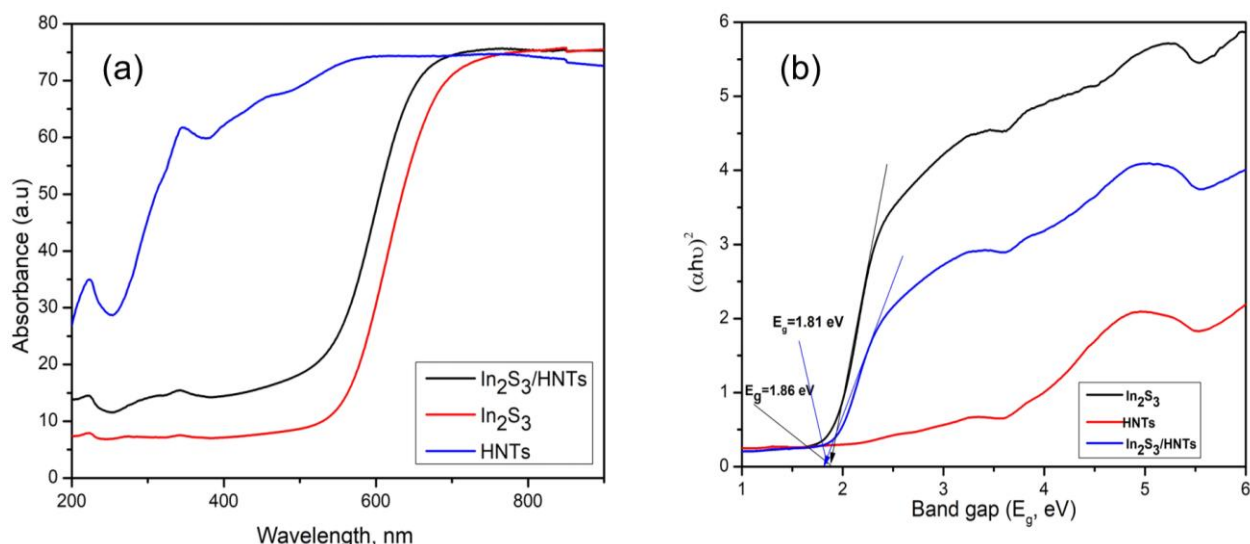


Fig. 4. (a) UV–Vis DRS spectrum, (b) band gap energies of HNTs, In₂S₃, and 40%In₂S₃/HNTs composite

3.2. Photocatalytic activity for DBT degradation

To investigate the photocatalytic performance of In₂S₃, HNTs, and 40%In₂S₃/HNTs, photocatalytic oxidative degradation reactions of DBT were conducted. Both In₂S₃ and 40%In₂S₃/HNTs exhibited high photocatalytic activity in the oxidation of DBT, achieving conversion efficiencies of 89% and 92.01%, respectively, after 5 hours of visible light irradiation. In contrast, HNTs were capable of adsorbing a small amount of DBT (46%) within the first hour, and this adsorption efficiency remained stable throughout the 5-hour reaction period (Fig. 5).

Interestingly, the 40%In₂S₃/HNTs composite demonstrated significantly enhanced photocatalytic performance compared to pure In₂S₃. This improvement indicates the synergistic effect of the HNTs support, which facilitates better dispersion of the active In₂S₃ phase both inside and outside the nanotubes. This, in turn, enhances visible light absorption and promotes the generation of reactive radicals essential for DBT degradation.

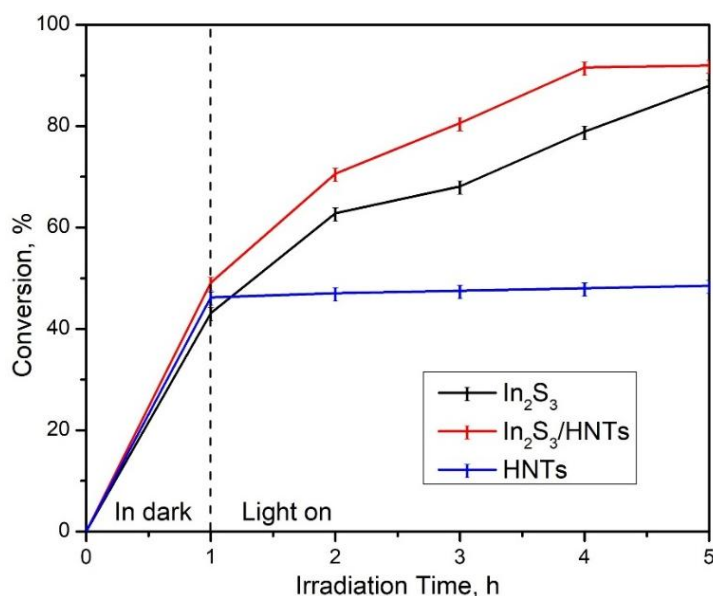


Fig. 5. Effect of the different photocatalysts on removal efficiency of DBT (Photocatalyst loading 40 mg, and 30 mg.L⁻¹ DBT)

Moreover, the presence of the oxidizing agent H₂O₂ significantly influences the photocatalytic process. Fig. 6 illustrates the DBT conversion on the 40%In₂S₃/HNTs catalyst after 5 hours of reaction with the addition of H₂O₂. The DBT conversion reached only 92% with the addition of 1.0 mL H₂O₂, and the conversion efficiency increased only marginally when 2.0 mL was added. Interestingly, a slight decrease in conversion was observed when 3.0 mL of H₂O₂ was used. As is well known, H₂O₂ is a strong oxidizing agent that can generate hydroxyl radicals (HO[•]) under light irradiation. However, an excessive

amount of H_2O_2 may lead to surface poisoning of the $\text{In}_2\text{S}_3/\text{HNTs}$ catalyst, resulting in adverse side reactions during the photocatalytic process (Zhang et al., 2021). Therefore, the optimal generation of $\text{HO}^\bullet$ radicals for DBT oxidation was achieved with the addition of 1.0 mL H_2O_2 .

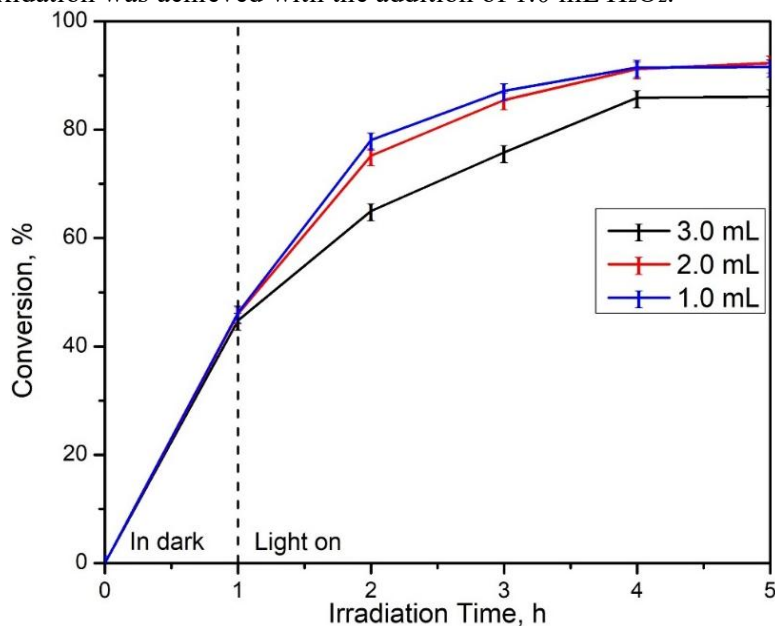


Fig. 6. Effect of the different oxidizing agent on removal efficiency of DBT (Photocatalyst loading 40 mg, and 30 mg.L^{-1} DBT)

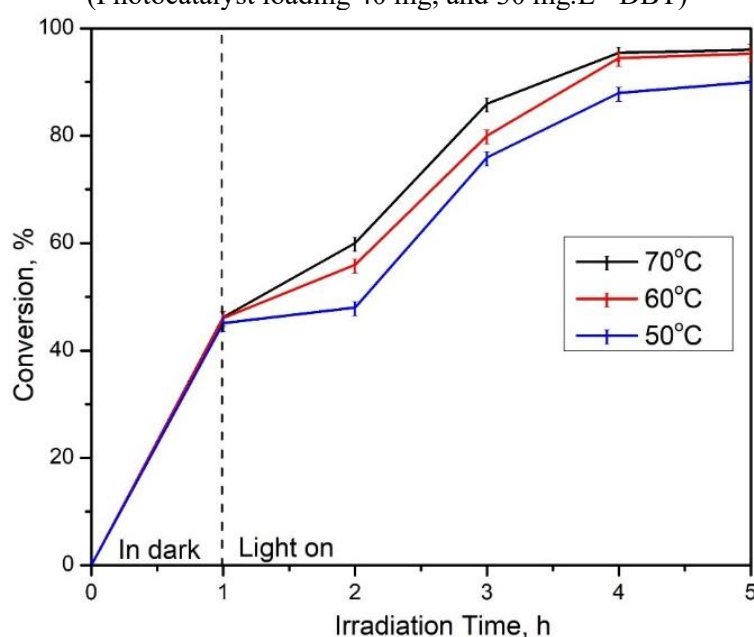


Fig. 7. Effect of the different temperature on removal efficiency of DBT (Photocatalyst loading 40 mg, and 30 mg.L^{-1} DBT)

The influence of temperature on photocatalytic degradation efficiency is particularly evident during the adsorption process in the absence of light. Therefore, the photocatalytic activity of the $\text{In}_2\text{S}_3/\text{HNTs}$ catalyst was evaluated for DBT degradation under dark conditions, and the results are presented in Fig. 7. Experimental results indicated that no photocatalytic reaction occurred in the absence of sunlight, even in the presence of the catalyst. Only approximately 44% of DBT was adsorbed at temperatures of 50 °C, 60 °C, and 70 °C over 5 hours. This once again confirms that the photocatalytic activity is primarily attributed to the generation of hydroxyl radicals ($^\bullet\text{OH}$) under solar irradiation, which leads to the decomposition of DBT. Furthermore, it was observed that the DBT conversion efficiency increased with rising reaction temperature. A maximum DBT conversion of 96% was achieved at 70 °C after 5 hours of reaction. At this temperature, the rate of $^\bullet\text{OH}$ radical formation is significantly higher than at 50 °C, enabling more effective DBT oxidation on the catalyst surface. Additionally, DBT

oxidation is kinetically limited at temperatures below 50 °C due to diffusion constraints, which hinder the overall reaction rate.

3.3. Photocatalyst Stability

To evaluate the reusability of the 40%In₂S₃/HNTs photocatalyst, recycling experiments were conducted for the photocatalytic degradation of DBT. After each reaction cycle, the catalyst was collected, centrifuged, thoroughly washed with deionized water and ethanol, then dried before being reused in subsequent cycles. As shown in Fig. 8, the photocatalytic efficiency under sunlight irradiation decreased slightly from 96% to 92% after five consecutive cycles. This slight reduction indicates that the 40%In₂S₃/HNTs catalyst maintains good photocatalytic stability and reusability over multiple uses.

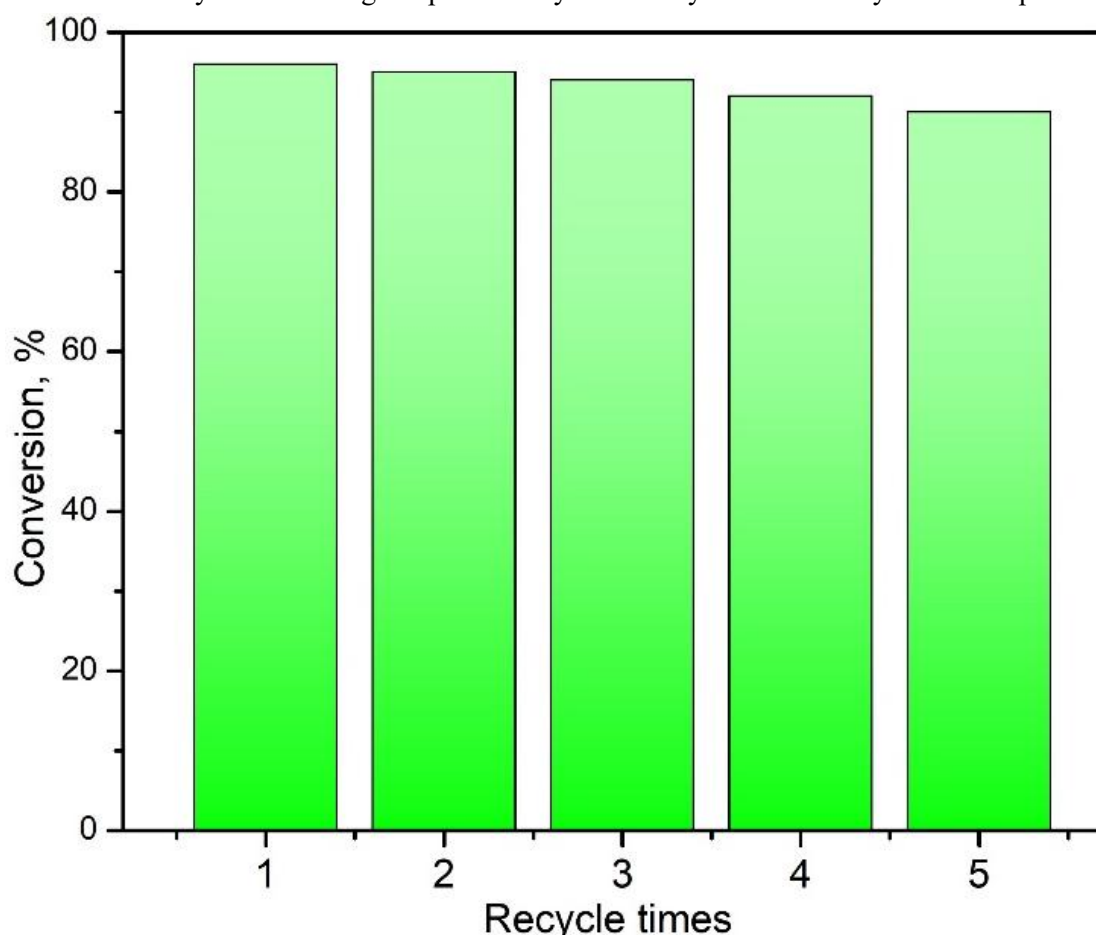


Fig. 8. Recycling tests of 40%In₂S₃/HNTs composite for removal of DBT

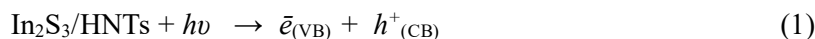
Table 2 illustrates the superior characteristics of the 40%In₂S₃/HNTs composite material. It not only demonstrates outstanding photocatalytic efficiency but also exhibits excellent catalytic stability, a simple synthesis procedure, and reduced usage of oxidizing agents for sulfur removal in fuels. These advantages position it as a promising candidate for large-scale industrial applications.

Tab. 2. Comparison of photocatalytic performance for DBT degradation using various photocatalysts

Materials	DBT conc. (ppm)	Light source	Band gap (eV)	Removal time (h)	Removal efficiency (%)	Refs.
40%In ₂ S ₃ /HNTs	250	300 W Xe lamp	1.84	5	96	This work (Hanh et al., 2024)
AgInS ₂	250	160 W high pressure Hg	2.04	8	98	
Cu-TiO ₂ -SBA-16	600	35 W UV light	2.95	5	79	(Pham et al., 2018)
Ag-AgBr/Al-MCM-41	300	125 W high pressure Hg	1.52	6	98	(Saeed et al., 2022)
Ni-WO ₃ @g-C ₃ N ₄	100	-	-	3	98	

Based on the results obtained above, Figure 9 summarizes the proposed mechanism of the In₂S₃/HNTs composite for DBT removal in this study. Under visible light irradiation, electrons are excited from the valence band to the conduction band of In₂S₃, leaving corresponding holes in the valence band. The photogenerated holes can directly oxidize DBT molecules. Since HNTs are inactive under visible light and cannot generate charge carriers by themselves, no direct photocatalytic activity is expected from them. However, the presence of HNTs facilitates the dispersion of active catalytic sites, enhancing the composite's ability to harvest visible light. This leads to an accelerated generation of reactive radicals such as superoxide anions (O₂^{•-}) and holes (h⁺), which effectively oxidize DBT.

Overall, the photocatalytic mechanism was significantly enhanced through electron transfer and consumption processes in the In₂S₃/HNTs photocatalyst, thereby improving the redox efficiency for DBT photocatalysis via the generation of active superoxide anions (O₂^{•-}) and hydroxyl radicals (HO•) (Pham et al., 2020).



The overall mechanistic pathway can be illustrated as follows:

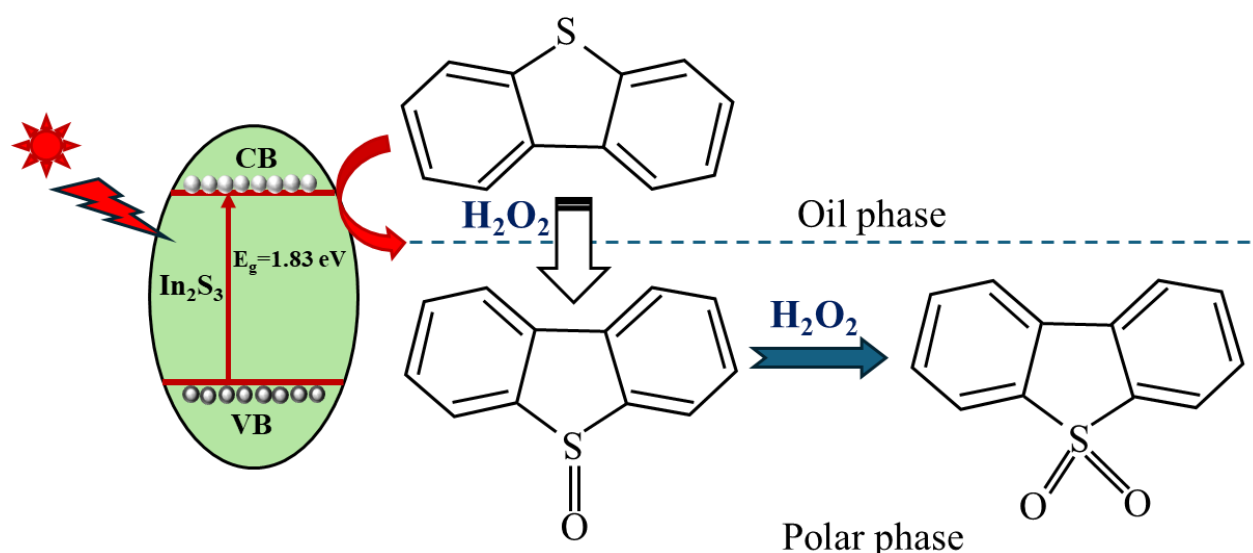


Fig. 9. Schematic diagram of the oxidative photodegradation mechanism for DBT on the In₂S₃/HNTs catalyst

4. Conclusion

In this study, the In₂S₃/HNTs composite was successfully synthesized via a one-step hydrothermal method and demonstrated to be an efficient photocatalyst for the redox degradation of DBT. The 40%In₂S₃/HNTs catalyst exhibited superior photocatalytic activity compared to pure In₂S₃. The interfacial interaction between HNTs and In₂S₃ in the composite played a crucial role in enhancing the photocatalytic performance by providing a larger surface area for reactant adsorption and improving visible light absorption. This work not only presents a highly active photocatalytic system but also confirms the catalyst's stability during the DBT removal process in fuel.

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