

Article

Amorphization-Enabled Direct Z-Scheme CdSe/MoSe_x Heterojunction for Enhanced Photocatalysis

Lie Tian ¹, Rong Wu ^{1,*}, HaiYang Liu ¹, Dong Zhang ¹ and Xiangqian Shen ^{1,2,*}

¹ Xinjiang Key Laboratory of Solid State Physics and Devices, School of Physical Science and Technology, Xinjiang University, Urumqi 830046, China; tianlie@xju.edu.cn (L.T.); 008093@xju.edu.cn (H.L.); keepdoing50@163.com (D.Z.)

² State Key Laboratory of Metal Matrix Composites, Shanghai Jiao Tong University, Shanghai 200240, China

* Correspondence: wurongxju@sina.com (R.W.); sxqlyq@sjtu.edu.cn (X.S.)

Abstract

Organic pollutant degradation demands photocatalysts that couple efficient charge separation with strong redox capability. Direct Z-scheme heterojunction architectures fulfill these requirements and are consequently considered promising candidates. However, the rational design of such systems continues to present a major obstacle. Herein, a CdSe/amorphous MoSe_x (CdSe/a-MoSe_x) direct Z-scheme heterojunction was successfully synthesized via a simple solid-state grinding and low-temperature hydrothermal method. Within 120 min under visible-light irradiation, methylene blue (MB, 40 mg/L) was degraded to 97.3% efficiency by the CdSe/a-MoSe_x heterojunction, whose photocatalytic activity markedly exceeded that of pristine CdSe and a-MoSe_x. Intimate interfacial contact between CdSe and a-MoSe_x enables photogenerated carriers to separate and migrate more efficiently, underpinning the observed performance enhancement. Moreover, the suitable band alignment derived from valence-band (VB) XPS and Mott-Schottky measurements supports the formation of a direct Z-scheme charge-transfer pathway. The Z-scheme mechanism effectively inhibits electron-hole recombination while maintaining the robust oxidation and reduction capabilities of the photogenerated carriers. This study offers a straightforward approach for fabricating CdSe/a-MoSe_x direct Z-scheme heterojunctions for the efficient photocatalytic degradation of organic pollutants.

Keywords: amorphous MoSe_x; CdSe; Z-scheme heterojunction; photocatalytic degradation; organic pollutant removal; charge separation



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1. Introduction

Heavy fossil fuel consumption and the sustained emission of industrial and domestic pollutants have aggravated current energy and environmental problems, with adverse implications for human health and ecosystem sustainability [1,2]. Conventional treatment processes struggle to fully eliminate recalcitrant organic pollutants—dyes, antibiotics, pharmaceutical residues, and endocrine-disrupting compounds—owing to their inherent resistance to degradation [3]. Solar-driven semiconductor photocatalysis has drawn significant interest in recent years—largely because it taps into sunlight to break down pollutants under mild, ambient conditions, with minimal energy input and no added environmental burden [4,5]. Photocatalytic materials encompass several broad classes, including conventional and doped metal oxides, bismuth-based oxides and oxyhalides, metal chalcogenides, and emerging multicomponent high-entropy oxides [6–8]. Despite their potential, the

use of single-component photocatalysts in practical photocatalytic systems remains restricted. While poor light absorption and slow surface reactions certainly matter, the rapid recombination of photogenerated carriers constitutes the most critical bottleneck [9]. Semiconductor heterojunction engineering has thus emerged as a widely adopted strategy for addressing charge separation bottlenecks and boosting photocatalytic efficiency [10,11]. Z-scheme and S-scheme heterojunctions have recently emerged as important architectures for photocatalytic applications. By enabling efficient interfacial charge transfer without sacrificing strong oxidation and reduction capabilities, they offer considerable potential for the degradation and mineralization of persistent organic pollutants [12–17].

Within the broad class of photocatalytic materials, layered transition metal dichalcogenides (TMDs, MX_2 ; $\text{M} = \text{Mo}$ or W , $\text{X} = \text{S}$ or Se) have emerged as particularly promising candidates. Their appeal is largely associated with band structures that can be modulated, high specific surface areas, and distinctive physicochemical characteristics [18]. MoSe_2 , as a representative TMD material, possesses a tunable narrow band gap of approximately 1.33–1.72 eV, enabling broad visible-light absorption [19,20]. In addition, MoSe_2 exhibits relatively good resistance to photocorrosion, which has been attributed to d-state-related optical transitions and the metal-chalcogen antibonding character of the VB states [18,21]. These characteristics make MoSe_2 a promising candidate for photocatalytic degradation. For instance, MoSe_2 -based composite photocatalysts have demonstrated higher degradation efficiency for common dyes and antibiotics, mainly because they broaden visible-light harvesting, facilitate interfacial charge transport, and limit the recombination of photogenerated carriers [22–24]. Nevertheless, pristine crystalline MoSe_2 usually suffers from restacking/agglomeration, insufficiently exposed edge active sites, and sluggish charge transfer, which greatly restrict its photocatalytic activity [21].

Amorphous TMDs diverge from crystalline counterparts: laden with unsaturated atoms, defects, and disordered coordination motifs, they effectively expose a higher density of catalytically accessible sites [25–27]. Yu et al. demonstrated that two-dimensional amorphous sulfides harbor a high concentration of catalytically active edge sites, a feature they attributed to the prevalence of coordinatively unsaturated sulfur atoms at the material periphery [26]. Jaramillo et al. provided further evidence that disulfide bonds within amorphous MoS_3 function as active sites, and catalytic efficiency appears to depend strongly on how many such sites remain accessible [28,29]. Subsequent investigations have further attributed the pronounced photocatalytic hydrogen evolution activity of amorphous MoS_x to the disordered arrangement of coordinatively unsaturated atomic sites within its structure [26]. Regarding molybdenum selenides, Feng et al. found that incorporating Co into amorphous MoSe_x resulted in a narrower band gap. In a separate study, Yang et al. synthesized amorphous MoSe_x via a straightforward hydrothermal route and reported that the amorphous material exhibited stronger photocatalytic degradation performance than its crystalline counterpart [27,30]. These findings suggest that the presence of abundant coordinatively unsaturated Se atoms and defect-enriched amorphous structures contributes to enhanced surface adsorption and more favorable interfacial reaction processes. Nevertheless, pristine amorphous MoSe_2 remains constrained by sluggish carrier transport and rapid recombination of photoexcited electron-hole pairs. Consequently, integrating amorphous MoSe_2 with a compatible semiconductor to form a well-matched heterojunction offers a practical strategy for boosting its photocatalytic activity. CdSe was selected as the coupling semiconductor because of its narrow band gap, strong visible-light absorption, and potentially complementary band-edge positions with amorphous MoSe_2 , which are favorable for constructing a direct Z-scheme heterojunction with efficient charge separation and preserved redox capability.

In recent years, MoSe₂-based semiconductor heterojunctions have been widely explored for photocatalytic pollutant degradation. Most MoSe₂/semiconductor composites documented to date operate via type-II charge-transfer pathways, yielding only modest gains in spatial separation of photoexcited carriers [31]. However, the spatial migration of electrons and holes in conventional type-II heterojunctions usually weakens the redox ability of the retained charge carriers, thereby limiting the generation of highly active species. Unlike conventional designs, Z-scheme heterojunctions trap electrons within the more negative CB while confining holes to the more positive VB, a separation that sustains strong redox capabilities [12]. Wang et al. reported that MoSe₂/g-C₃N₄ Z-scheme heterojunctions exhibited a narrow band gap, abundant active sites, enhanced photocurrent response, and improved degradation performance toward organic pollutants [32]. Recently, g-C₃N₄@MoSe₂ composites have shown excellent visible-light-driven tetracycline degradation performance, which was attributed to promoted charge separation and increased generation of $\bullet\text{O}^{2-}$ and h^+ species [24]. Additionally, hollow S-scheme MoSe₂/Fe₂O₃ heterojunctions have been fabricated as visible-light-responsive peroxymonosulfate activators capable of removing ciprofloxacin, which underscores why controlling interfacial charge dynamics and catalytic-site accessibility matters [16,17]. These advances suggest that the design of MoSe₂-based heterojunctions is shifting from simply broadening light absorption to precisely controlling charge-transfer pathways, built-in electric fields, and reactive-species generation.

Despite notable progress, rationally engineering amorphous MoSe₂-based Z-scheme heterojunctions remains nontrivial. In this work, a CdSe/a-MoSe_x heterojunction was synthesized via solid-state grinding of CdSe followed by low-temperature hydrothermal assembly with a-MoSe_x. Under visible-light irradiation, the composite achieved 97.3% MB degradation at an initial concentration of 40 mg/L within 120 min, delivering markedly superior photocatalytic performance relative to pristine CdSe and a-MoSe_x. Intimate interfacial contact and appropriate band alignment enable efficient charge separation via a direct Z-scheme pathway—accounting for the markedly improved photocatalytic activity. This work presents a scalable strategy for amorphous MoSe₂-based Z-scheme photocatalysts and can be extended to other semiconductor–pollutant systems.

2. Materials and Methods

2.1. Materials

Ammonium molybdate tetrahydrate ((NH₄)₆Mo₇O₂₄·4H₂O), hydrazine hydrate (N₂H₄·H₂O, 98%), Se powder with a purity of 99.9%, sodium selenite (Na₂SeO₃, 99.5%), sodium hydroxide (NaOH, 99%), absolute ethanol (C₂H₅OH, ≥99.8%), sodium sulfite (Na₂SO₃, 99.5%), and cadmium acetate dihydrate (Cd(CH₃COO)₂·2H₂O, 99.7%) were obtained from Alfa Aesar (Ward Hill, MA, USA). MB was purchased from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China). Each reagent was used directly in its analytical-grade form, with no additional purification step.

2.2. Synthesis of CdSe Nanoparticles

CdSe was synthesized using a solid-state chemical method. Typically, Se powder (0.592 g), Na₂SO₃ (1.525 g), and NaOH (0.600 g) were placed in an agate mortar and ground vigorously at room temperature for 20 min until a black viscous paste was formed. Subsequently, Cd(CH₃COO)₂·2H₂O (1.998 g) was added, and the mixture was continuously ground for another 30 min. The resulting product was rinsed repeatedly with deionized water and absolute ethanol before being oven-dried at 60 °C for 14 h.

2.3. Synthesis of CdSe/a-MoSe_x Heterojunction Composite

CdSe/a-MoSe_x composites were prepared using a hydrothermal route. For solution A, Na₂SeO₃ (0.487 g) was introduced into hydrazine hydrate (10 mL) and magnetically stirred for 30 min until dissolved. In a separate step, (NH₄)₆Mo₇O₂₄·4H₂O (0.464 g) was dissolved in deionized water (25 mL), with stirring continued for 1 h to yield solution B. The two precursor solutions were then combined, and the obtained mixture was stirred for 1 h to produce solution C. Finally, the as-prepared CdSe (0.230 g) was dispersed into solution C under 30 min of continuous stirring. The resulting suspension was loaded into a 50 mL Teflon-lined stainless-steel autoclave and hydrothermally treated at 140 °C for 17 h. After natural cooling to room temperature, the precipitate was collected, washed sequentially with deionized water and absolute ethanol (≥99.8%), and vacuum-dried at 60 °C for 8 h, yielding the final CdSe/a-MoSe_x composite.

For comparison, a-MoSe_x was synthesized using the same hydrothermal procedure as that employed for the CdSe/a-MoSe_x composite, except that CdSe was not added. Briefly, solutions A and B were prepared and mixed as described above, and the resulting solution was hydrothermally treated at 140 °C for 17 h. After natural cooling, the precipitate was washed sequentially with deionized water and absolute ethanol and vacuum-dried at 60 °C for 8 h.

2.4. Methods of Characterization

The morphology and microstructure of the as-prepared samples were characterized by scanning electron microscopy (SEM, LEO 1430VP, LEO Electron Microscopy Ltd., Cambridge, UK), transmission electron microscopy (TEM, JEM-2100, JEOL Ltd., Akishima, Japan), and high-resolution TEM (HRTEM, JEM-2100, JEOL Ltd., Akishima, Japan). The SEM observations were performed at an accelerating voltage of 20.0 kV and a working distance of 10 mm, and the representative SEM image was acquired at a magnification of 10,000×. The TEM and HRTEM observations were conducted at an accelerating voltage of 200 kV, and the representative TEM image was acquired at a direct magnification of 50,000×. Crystalline structure and phase constitution were determined from X-ray diffraction measurements performed on a D/MAX-2500 diffractometer over a 2θ range of 10–80°. Elemental information, including both compositional makeup and elemental mapping, was acquired via energy-dispersive X-ray spectroscopy (EDS). To examine the surface chemistry in greater detail, X-ray photoelectron spectroscopy (XPS, VG-ESCALAB-LK, VG Scientific Ltd., East Grinstead, UK) was employed to identify the chemical composition at the surface and the corresponding elemental valence states. The XPS survey spectra were recorded over a binding-energy range from −9.92 to 1350.08 eV. Photoluminescence spectroscopy (PL, Hitachi F-4500, Hitachi Ltd., Tokyo, Japan) was further used to assess the separation efficiency and recombination tendency of photogenerated electron-hole pairs. The PL spectra were recorded at room temperature using an excitation wavelength of 500 nm under identical measurement conditions. Mott-Schottky plots were recorded for the samples on a CHI 660E electrochemical workstation over an applied-potential range from −0.76 to 0.74 V with a potential interval of 0.01 V, and the obtained curves were used to identify the semiconductor type and derive the corresponding flat-band potential. GenAI (DeepSeek-V3.1-Terminus) has been used in the paper to generate Figure 7.

2.5. Photocatalytic Experiment

Visible-light degradation of MB served as the probe reaction for assessing photocatalytic activity. A 350 W Xe lamp fitted with a cutoff filter (λ > 400 nm) supplied the irradiation. The distance between the light source and the surface of the reaction solution was approximately 10 cm, and the irradiation intensity at the solution surface was

approximately 300 mW cm^{-2} . For each test, 10 mg of photocatalyst was added to 50 mL of an aqueous methylene blue trihydrate solution with a concentration of 40 mg L^{-1} , corresponding to approximately $1.07 \times 10^{-4} \text{ mol L}^{-1}$, and the mixture was homogenized by stirring. Before illumination, magnetic agitation was continued for 30 min in darkness to allow adsorption–desorption equilibrium to be reached. The reaction mixture then underwent visible-light irradiation for 120 min while stirring was maintained. At every 30 min interval, a portion of the suspension was collected and centrifuged to separate the solid catalyst. The remaining MB concentration in the supernatant was quantified with a UV–Vis spectrophotometer (Hitachi U-3010, Hitachi High-Technologies Corporation, Tokyo, Japan) by measuring the characteristic absorption at 664 nm. Photocatalytic experiments using CdSe, a-MoSe_x, and CdSe/a-MoSe_x were conducted under otherwise identical conditions. Each experiment was repeated four times to confirm the reproducibility of the observed photocatalytic trends.

3. Results and Discussion

3.1. Phase Structure and Morphology

As shown in Figure 1, the XRD pattern of a-MoSe_x exhibits no sharp diffraction peaks. Instead, a broad diffuse scattering feature is observed over the approximate 2θ range of $20\text{--}35^\circ$, indicating the absence of long-range crystalline order. Because the obtained MoSe_x is amorphous and non-stoichiometric, this diffuse feature cannot be indexed to specific crystallographic planes of crystalline 2H-MoSe₂ or directly matched with its standard PDF pattern. For pristine CdSe, the peaks at $2\theta = 25.35^\circ$, 42.09° , and 49.69° are indexed to the (111), (220), and (311) planes of cubic sphalerite CdSe. These peak positions agree well with the calculated reference diffraction pattern of cubic sphalerite CdSe (PDF No. 97-004-1528, derived from ICSD No. 41528) [33,34]. Following coupling with a-MoSe_x, the CdSe/a-MoSe_x composite still displays the characteristic diffraction peaks of cubic CdSe, with no discernible peak displacement. In particular, the broad major diffraction feature observed near $24\text{--}25^\circ$ is centered at approximately 25.35° and is assigned to the (111) plane of cubic CdSe rather than to an unidentified impurity phase. Its apparent broadening may also arise from overlap with the diffuse scattering contribution of amorphous MoSe_x in the $20\text{--}35^\circ$ region. No additional sharp reflections attributable to crystalline molybdenum selenide phases are detected, suggesting that the MoSe_x component remains X-ray amorphous or highly dispersed in the composite. Therefore, the XRD patterns confirm the coexistence of crystalline CdSe and amorphous MoSe_x in the CdSe/a-MoSe_x composite. Accordingly, the composite should not be described as a single phase-pure crystalline material; rather, it consists of crystalline cubic CdSe and amorphous or highly dispersed MoSe_x, with no detectable crystalline impurity phases within the detection limit of XRD. The interfacial heterostructure should be further verified by microscopic and surface chemical analyses.

As shown in Figure 2a, a-MoSe_x exhibits an irregular nanoparticle-aggregated morphology with a rough surface. After coupling with CdSe, the CdSe/a-MoSe_x composite still maintains an aggregated nanoparticle-like morphology, while the surface becomes relatively compact and smoother (Figure 2b). The TEM image in Figure 2c further shows that the CdSe/a-MoSe_x composite consists of irregularly aggregated nanoparticles with sizes of several tens of nanometers. The HRTEM image in Figure 2d reveals the coexistence of ordered lattice fringes and disordered regions in the CdSe/a-MoSe_x composite, suggesting the presence of crystalline CdSe and amorphous MoSe_x domains.

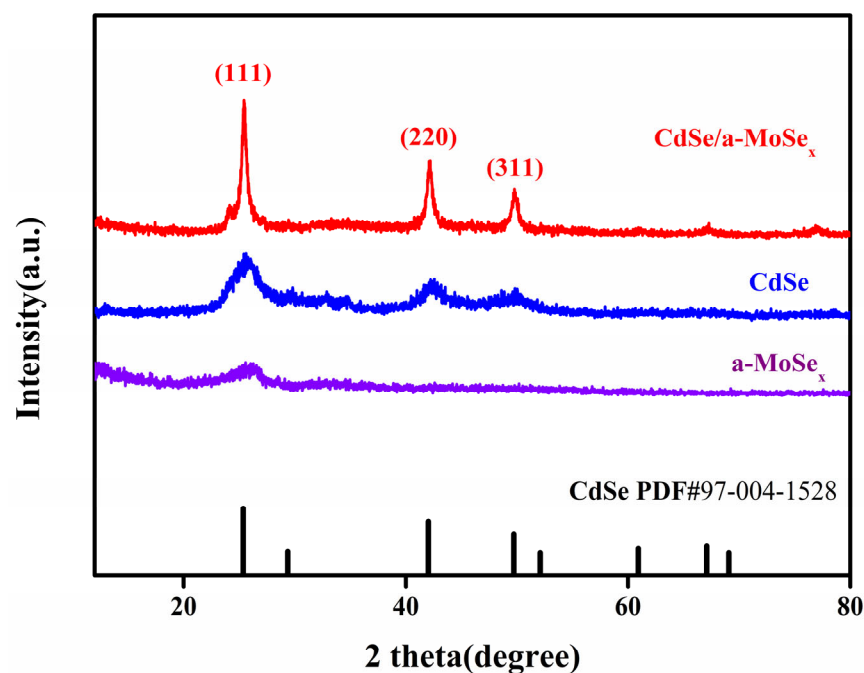


Figure 1. XRD patterns of a-MoSe_x, CdSe, and CdSe/a-MoSe_x, together with the calculated reference pattern of cubic CdSe (PDF No. 97-004-1528).

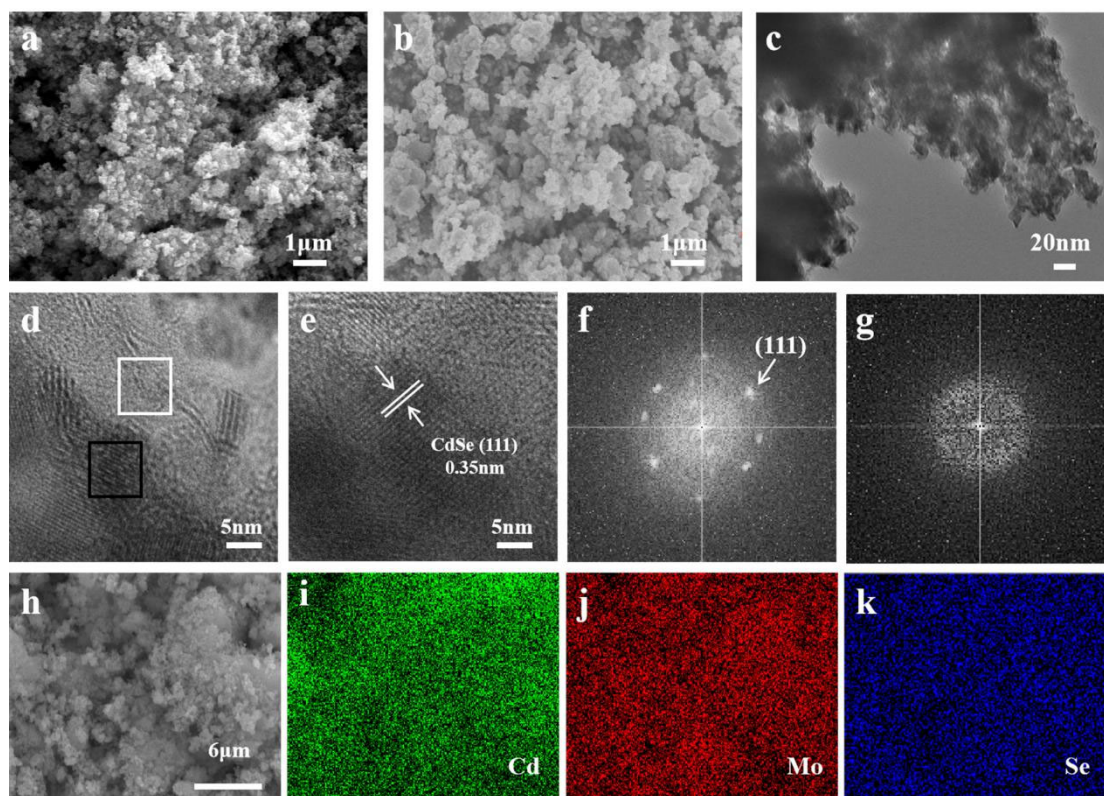


Figure 2. SEM micrographs of (a) a-MoSe_x and (b) CdSe/a-MoSe_x, together with (c) a TEM micrograph of CdSe/a-MoSe_x; (d,e) HRTEM images of CdSe/a-MoSe_x; (f) FFT pattern of the black-framed region in (d), corresponding to crystalline CdSe; (g) FFT pattern of the white-framed region in (d), corresponding to amorphous MoSe_x; (h–k) EDS elemental mapping images of CdSe/a-MoSe_x for Cd, Mo, and Se.

Figure 2e displays well-resolved lattice fringes with an interplanar spacing of 0.351 nm, matching the (111) plane of cubic zinc blende CdSe [35]. Local structural information was

further extracted from the FFT analysis of selected regions in Figure 2d. The FFT pattern from the black-framed area contains discrete diffraction spots, verifying the crystalline character of the CdSe domain. By contrast, the white-framed region gives an FFT pattern dominated by a broad diffuse halo rather than sharp diffraction spots. This response confirms the amorphous nature of MoSe_x and agrees with the XRD patterns [36]. The elemental maps obtained from EDS analysis, shown in Figure 2h–k, reveal a homogeneous spatial distribution of Cd, Mo, and Se across the examined area. This co-localization supports the close interfacial contact between CdSe and a-MoSe_x. Quantitative EDS analysis gives a Mo/Se atomic ratio of approximately 1:2.16, indicating that the amorphous MoSe_x phase deviates from ideal stoichiometry. These results confirm the coexistence of crystalline CdSe and amorphous MoSe_x and support the successful construction of the CdSe/a-MoSe_x heterostructure.

3.2. Surface Chemical States and Band Structure

XPS was employed to examine the surface chemistry of CdSe/a-MoSe_x, with particular attention to its elemental composition and valence-state information. From the survey XPS data in Figure 3a, signals of Cd, Mo, and Se are clearly identifiable, indicating that CdSe and a-MoSe_x have been combined successfully. Pristine CdSe exhibits two Cd 3d peaks at 404.5 and 411.2 eV (Figure 3b), attributed to Cd 3d_{5/2} and Cd 3d_{3/2} of Cd²⁺ [35]. For CdSe/a-MoSe_x, these two peaks shift to 405.2 and 411.9 eV, indicating a changed electronic environment of Cd after coupling with a-MoSe_x. Figure 3c displays the Se 3d region of pristine CdSe, where two signals emerge at 53.4 and 54.5 eV; these correspond, respectively, to the Se 3d_{5/2} and Se 3d_{3/2} peaks originating from Se^{2−} [37]. For a-MoSe_x, the analogous Se 3d_{5/2} and Se 3d_{3/2} components appear at 54.5 and 55.6 eV, respectively. Upon formation of CdSe/a-MoSe_x, the Se 3d signals originating from a-MoSe_x move toward smaller binding-energy values, appearing at 54.1 and 55.2 eV. This shift points to interfacial electron redistribution between CdSe and a-MoSe_x. Figure 3d presents the high-resolution Mo 3d spectra. In the case of a-MoSe_x, the spectral features centered at 228.6 and 231.9 eV correspond to the Mo⁴⁺ 3d_{5/2} and Mo⁴⁺ 3d_{3/2} components, respectively. After coupling with CdSe, the corresponding Mo⁴⁺ peaks shift to 228.2 and 231.5 eV. The components located at 230.0 eV for a-MoSe_x and 229.6 eV for CdSe/a-MoSe_x are assigned to the Se 3s signal overlapping with the Mo 3d region. The higher-binding-energy components at 233.5 and 236.2 eV for a-MoSe_x, and at 233.1 and 235.8 eV for CdSe/a-MoSe_x, are attributed to higher-valence Mo species associated mainly with partial surface oxidation [38]. To quantify the different Mo species, the background-subtracted integrated areas of the Mo 3d_{5/2} and Mo 3d_{3/2} components belonging to the same Mo species were summed and normalized to the total fitted Mo 3d area. The overlapping Se 3s contribution was fitted separately and excluded from the calculation. The quantitative fitting results indicate that a-MoSe_x contains approximately 67.2% Mo⁴⁺ and 32.8% higher-valence Mo species, whereas CdSe/a-MoSe_x contains approximately 74.8% Mo⁴⁺ and 25.2% higher-valence Mo species. Thus, the relative Mo⁴⁺ content increases by approximately 7.6 percentage points after coupling with CdSe. The coexistence of Mo⁴⁺ and higher-valence Mo species indicates the non-stoichiometric and defect-rich nature of amorphous MoSe_x [27]. The decrease in the relative proportion of higher-valence Mo species after coupling also suggests a modified surface chemical environment of the MoSe_x component. The positive shift in the Cd 3d peaks in CdSe/a-MoSe_x relative to pristine CdSe is consistent with electron depletion at the Cd sites. In contrast, the negative shifts in Mo 3d and Se 3d peaks associated with a-MoSe_x suggest increased electron density around the MoSe_x component. These opposite binding-energy shifts are consistent with interfacial electron redistribution from CdSe toward a-MoSe_x, which may be attributed to Fermi-level equilibration after

intimate contact between the two semiconductors. This interfacial charge redistribution promotes an internal electric field that drives spatial separation and transport of photogenerated carriers [39]. Therefore, the XPS analysis provides strong evidence for electronic interaction between CdSe and a-MoSe_x and supports the formation of a mediator-free CdSe/a-MoSe_x heterostructure.

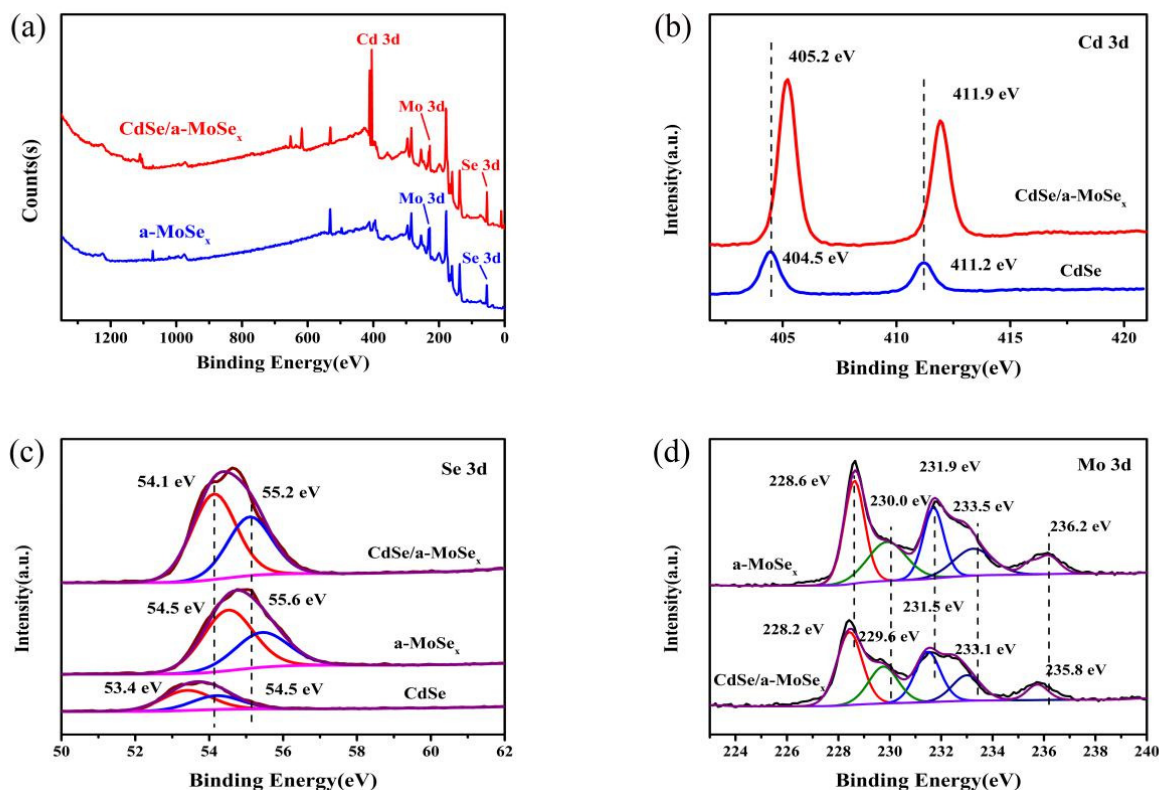


Figure 3. XPS spectra: (a) survey scans of a-MoSe_x and CdSe/a-MoSe_x; high-resolution regions of (b) Cd 3d (CdSe, CdSe/a-MoSe_x), (c) Se 3d (CdSe, a-MoSe_x, CdSe/a-MoSe_x), and (d) Mo 3d (a-MoSe_x, CdSe/a-MoSe_x).

UV–Vis absorption spectroscopy, VB XPS, and Mott–Schottky analysis were carried out to probe the band alignment and interfacial charge-transfer behavior of the CdSe/a-MoSe_x heterojunction. Figure 4a shows that a-MoSe_x and CdSe/a-MoSe_x both respond strongly across the 300–800 nm spectral region, demonstrating their effective capacity to harvest visible light. Compared with pristine a-MoSe_x, the CdSe/a-MoSe_x composite displays marginally stronger visible-light absorption. The UV–Vis absorption spectrum of CdSe (Figure 4b) shows that its absorption edge extends to approximately 650 nm, confirming its visible-light response. According to Tauc’s formula: $(\alpha h\nu)^n = A(h\nu - E_g)$, the band gaps of a-MoSe_x, CdSe, and CdSe/a-MoSe_x are derived as 1.47, 1.69, and 1.38 eV, respectively [40,41]. The narrowed band gap of CdSe/a-MoSe_x is beneficial for improving visible-light absorption.

The VB XPS spectra in Figure 4c indicate that the energy gaps separating the VB maximum from the Fermi level are 0.36 eV for a-MoSe_x and 1.27 eV for CdSe. Figure 4d reveals that the Mott–Schottky plots exhibit positive slopes, implying n-type semiconducting behavior for both a-MoSe_x and CdSe [41,42]. Mott–Schottky analysis yields flat-band potentials of roughly −0.33 V for a-MoSe_x and −0.12 V for CdSe (vs. NHE at pH 7). Coupling these values with the VB–XPS data gives VB edges of approximately 0.03 V and 1.15 V vs. NHE for a-MoSe_x and CdSe, respectively. Using the bandgap values, the conduction-band (CB) edges are derived as approximately −1.44 V for a-MoSe_x and −0.54 V for CdSe.

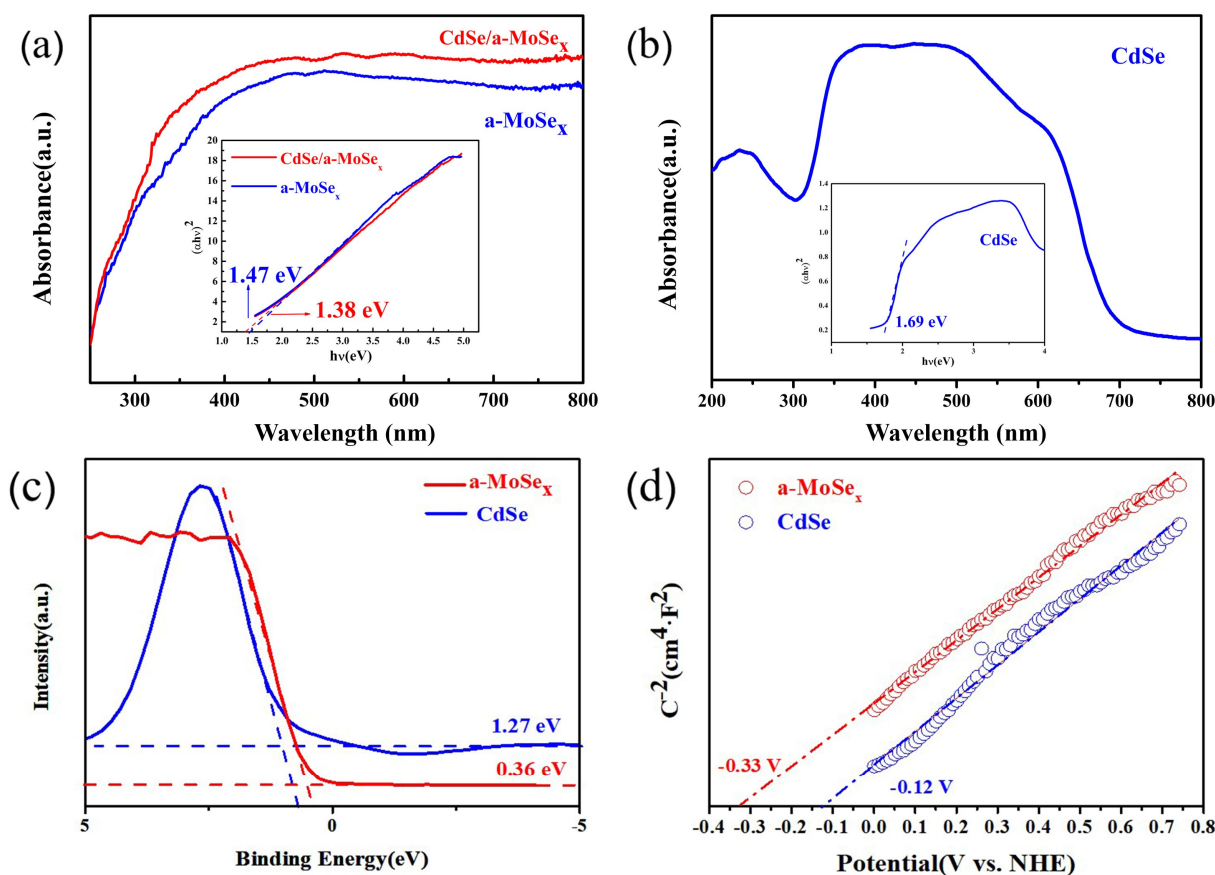


Figure 4. Optical and electronic structure characterization: (a) UV-Vis spectra of a-MoSe_x and CdSe/a-MoSe_x, with the corresponding bandgaps shown in the inset; (b) UV-Vis absorption spectrum of CdSe photocatalyst, with its bandgap displayed in the inset; (c) VB XPS spectra of a-MoSe_x and CdSe; and (d) Mott-Schottky plots of a-MoSe_x and CdSe.

The band alignment discussed above supports the construction of a direct Z-scheme carrier-transport mechanism in the CdSe/a-MoSe_x heterojunction. Upon visible-light irradiation, the photoexcited electrons generated on CdSe are consumed at the interface through recombination with holes from a-MoSe_x. This process leaves the highly reductive electrons on the a-MoSe_x CB (−1.44 V), while the oxidation-active holes are maintained on the CdSe VB (1.15 V). This interfacial pathway promotes faster charge separation while retaining the redox capability of the photoinduced carriers, thereby providing the reactive driving force required for efficient methylene blue degradation.

According to a conventional type-II pathway, electrons would depart the a-MoSe_x CB and migrate into that of CdSe, while holes would leave the CdSe VB and move into that of a-MoSe_x [12,39,43]. Although this CB/VB configuration may spatially separate charge carriers, it inevitably places electrons and holes on energetically less favorable sites, weakening their reduction and oxidation capacity. Such attenuation of the redox capability is inconsistent with the marked photocatalytic enhancement observed for the CdSe/a-MoSe_x heterojunction, indicating that the type-II mechanism alone is insufficient to explain the reaction behavior.

3.3. Photocatalytic Performance, Stability, and Active-Species Analysis

The photocatalytic performance of CdSe, a-MoSe_x, and CdSe/a-MoSe_x was probed through visible-light-driven degradation of MB. Figure 5a indicates that CdSe/a-MoSe_x delivers markedly enhanced photocatalytic performance compared with CdSe and a-MoSe_x alone. Following 120 min of visible-light exposure, CdSe, a-MoSe_x, and CdSe/a-MoSe_x

achieved MB removal rates of 58.8%, 67.0%, and 97.3%, respectively. CdSe/a-MoSe_x achieves a degradation efficiency roughly 1.65-fold greater than CdSe and 1.45-fold greater than amorphous MoSe_x, demonstrating that forming the CdSe/a-MoSe_x heterojunction significantly improves photocatalytic degradation performance.

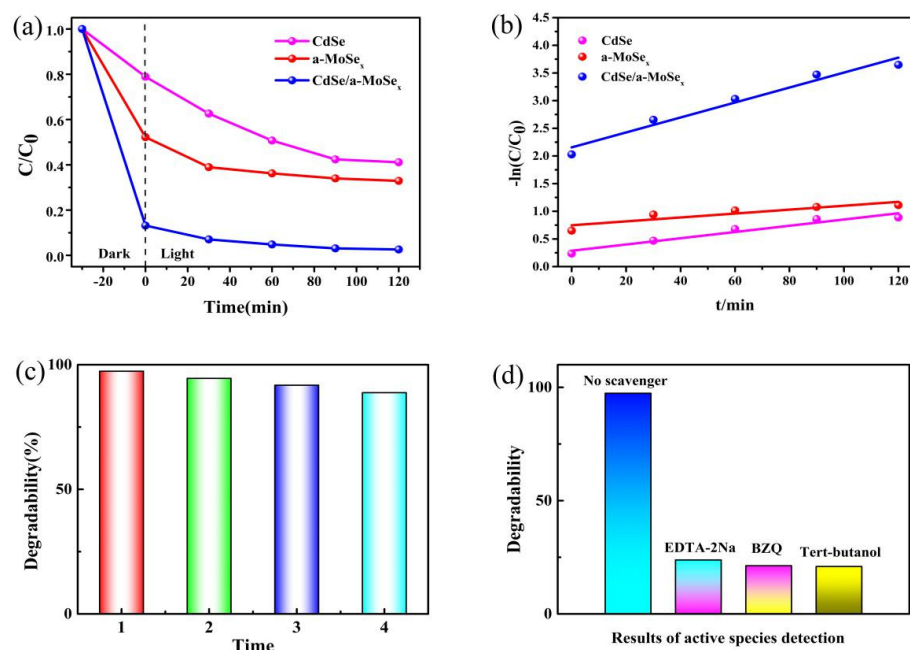


Figure 5. (a) Photocatalytic degradation rate of MB; (b) fitting the first-order kinetic curve of photocatalytic degradation of MB; (c) cyclic test of CdSe/a-MoSe_x photocatalytic activity; (d) active species trapping tests of CdSe/a-MoSe_x.

To better characterize pollutant degradation, the pseudo-first-order fit $\ln(C/C_0) = -kt$ was applied to the measured data. Here, C_0 is the MB concentration measured after dark adsorption equilibrium, $\ln(C/C_0)$ is the concentration recorded at irradiation time t , and k corresponds to the apparent rate constant [44,45]. According to Figure 5b, the apparent rate constant for CdSe/a-MoSe_x reaches 0.0135 min^{-1} , which is 2.37-fold greater than that of CdSe (0.0057 min^{-1}) and 3.86-fold greater than that of a-MoSe_x (0.0035 min^{-1}). This finding further confirms that the CdSe/a-MoSe_x heterojunction can effectively accelerate MB degradation. For broader context, a qualitative comparison of the photocatalytic performance of CdSe/a-MoSe_x with representative reported photocatalysts under different experimental conditions is provided in Table S1 of the Supporting Information.

The stability of CdSe/a-MoSe_x was evaluated through repeated cycling tests. Figure 5c shows that after four successive cycles, the photocatalytic degradation efficiency remains at 88.7%, which is only 8.6 percentage points lower than the initial value of 97.3%, indicating satisfactory durability. The XRD pattern of the catalyst recovered after four cycles is presented in Figure S1. Compared with the fresh catalyst, no obvious changes in the characteristic diffraction peaks or peak positions are observed, and no additional crystalline phases appear, indicating that the principal crystalline phase and structural characteristics of CdSe/a-MoSe_x are well preserved after cycling. Rather than intrinsic deactivation, the minor performance decline is more likely associated with catalyst loss during recovery and washing.

Scavenger tests were conducted to determine the dominant active species, with EDTA-2Na used for h^+ , p-benzoquinone (BQ) for $\bullet O_2^-$, and tert-butanol (TBA) for $\bullet OH$. As presented in Figure 5d, introducing EDTA-2Na, BQ, or TBA reduces the degradation

efficiency to 21.3%, 23.8%, and 20.9%, respectively, indicating that h^+ , $\bullet O_2^-$, as well as hydroxyl radicals, contribute to the photocatalytic degradation of MB [46].

Band-structure calculations suggest that CdSe/a-MoSe_x operates via a direct Z-scheme pathway for charge transfer. When exposed to visible light, CdSe and a-MoSe_x both produce electron–hole pairs. At the interface, photoexcited electrons from CdSe’s CB recombine with VB holes on a-MoSe_x, whereas the CB of a-MoSe_x preserves strongly reducing electrons and the VB of CdSe holds strongly oxidizing holes. Electrons residing in the CB of a-MoSe_x are capable of converting dissolved O₂ into $\bullet O_2^-$, whereas holes located in the VB of CdSe directly oxidize MB molecules. Although the VB potential of CdSe lacks sufficient oxidative strength to convert H₂O/OH[−] directly into $\bullet OH$, the scavenger tests indicate that $\bullet OH$ may be produced through an indirect oxygen-reduction pathway. In this process, $\bullet O_2^-$ formed on a-MoSe_x can undergo protonation and/or disproportionation to generate H₂O₂, which is then further transformed into $\bullet OH$ through electron-driven or surface-mediated reactions [46,47]. This at least suggests that CdSe/a-MoSe_x performs better mainly because its Z-scheme heterojunction separates carriers more effectively while retaining high oxidation-reduction capacity.

PL spectroscopy (Figure 6) was used to examine how photoexcited carriers recombine in a-MoSe_x, CdSe, and the CdSe/a-MoSe_x composite. The PL spectra were recorded at room temperature using an excitation wavelength of 500 nm under identical measurement conditions. All samples exhibit broad emission bands centered at around 750 nm. The emission maxima of the three samples are located at approximately 751–752 nm. The similar emission positions do not necessarily indicate identical band structures or the same radiative transition. For CdSe, the emission near 752 nm is close to its band-edge energy, whereas the broad emission of amorphous and defect-rich a-MoSe_x may involve localized defect or disorder-related states. The broad spectral bands from the two components overlap in the same wavelength region. Notably, after baseline correction, the PL intensity follows the order CdSe > a-MoSe_x > CdSe/a-MoSe_x, which is inversely correlated with their photocatalytic performance. The much lower PL intensity of CdSe/a-MoSe_x suggests more effective carrier separation and reduced electron–hole recombination, allowing photoinduced charges to move more readily toward the catalyst surface and participate in the photocatalytic reaction [48]. This observation is consistent with the superior MB degradation efficiency achieved by the CdSe/a-MoSe_x heterojunction.

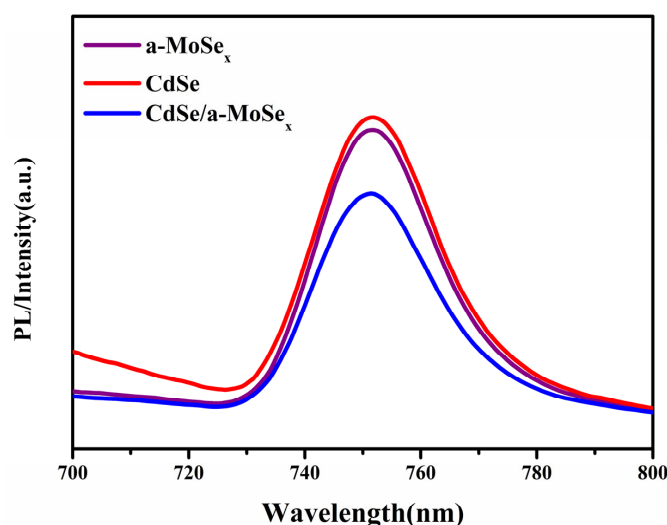


Figure 6. Baseline-corrected room-temperature PL emission spectra of a-MoSe_x, CdSe, and CdSe/a-MoSe_x recorded under excitation at 500 nm.

3.4. Charge-Carrier Dynamics and Photocatalytic Mechanism

Therefore, the proposed photocatalytic degradation mechanism of MB over the direct Z-scheme CdSe/a-MoSe_x heterojunction structure is illustrated in Figure 7. Relative to pristine CdSe and amorphous MoSe_x, the direct Z-scheme CdSe/a-MoSe_x heterojunction shows markedly improved photocatalytic degradation performance for MB dye during visible-light exposure. For semiconductor photocatalysts, overall activity is typically determined by light absorption capacity, charge-carrier separation and mobility, and interfacial redox processes. Several factors discussed below likely account for the marked boost in photocatalytic behavior. Firstly, the introduction of amorphous MoSe_x into the CdSe/MoSe_x system effectively enhances the visible-light absorption intensity and narrows the band gap to 1.38 eV, thereby improving visible-light utilization. Moreover, the rough surface and defect-rich nature of a-MoSe_x provide abundant active sites for photocatalytic reactions. Secondly, Z-scheme heterojunctions retain the high photogenerated electron-hole separation efficiency characteristic of conventional type-II heterojunctions while overcoming their inherent limitation of weakened redox capability, thereby enhancing the interfacial redox ability of the material. In a conventional type-II heterojunction, photogenerated electrons and holes preferentially accumulate at the lower-energy band positions upon light excitation. Although this arrangement promotes charge separation, it also reduces the driving force for redox reactions. In the direct Z-scheme CdSe/a-MoSe_x heterojunction, CdSe-derived CB electrons (−0.54 V) undergo interfacial recombination with VB holes from amorphous MoSe_x (0.03 V), as illustrated by the red dashed pathway in Figure 7. This process preserves the reductive electrons on a-MoSe_x and the oxidative holes on CdSe, leading to improved photocatalytic performance.

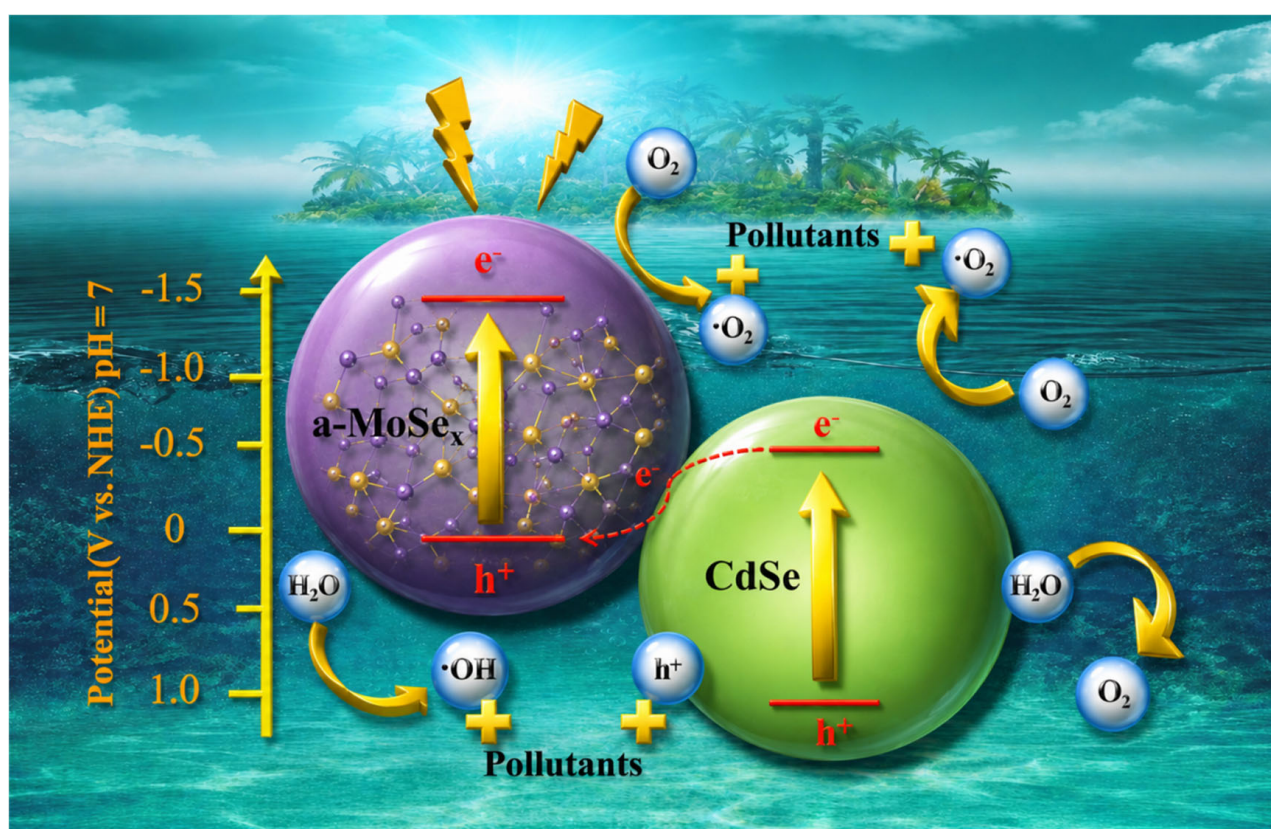
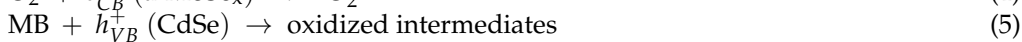
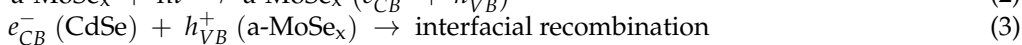
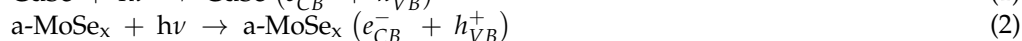


Figure 7. Direct Z-scheme electron transfer channel and photocatalytic reaction scheme for the CdSe/a-MoSe_x heterojunction.

The proposed photoexcitation, charge-transfer, reactive-species generation, and MB degradation processes can be expressed as follows:



Consequently, highly reductive electrons in the CB of MoSe_x drive the conversion of O₂ to $\bullet\text{O}_2^-$, whereas the oxidative holes retained in the valence band of CdSe directly oxidize adsorbed MB molecules. Hydroxyl radicals may subsequently be generated through secondary reactions involving $\bullet\text{O}_2^-$ and H₂O₂ intermediates. These reactive species jointly promote the degradation and mineralization of MB. Finally, combining amorphization with a direct Z-scheme interface improves light absorption, exposes more active sites, and promotes interfacial charge separation while preserving strong redox ability. Evidence for the proposed mechanism is internally consistent rather than merely correlative: the CdSe/a-MoSe_x composite delivers 97.3% photocatalytic degradation of methylene blue, while XPS resolves a positive shift in the Mo⁴⁺ peaks and higher-valence Mo species, implicating a defect-rich, non-stoichiometric surface with a greater density of catalytically accessible sites. Driven by the interfacial potential difference, charge recombination is kinetically suppressed, as corroborated by PL quenching; active-species trapping assigns h⁺, $\bullet\text{O}_2^-$, and $\bullet\text{OH}$ as the principal oxidative contributors in the degradation pathway. These results substantiate an amorphization-enabled direct Z-scheme CdSe/a-MoSe_x heterojunction and define amorphization engineering as a rational route toward efficient organic-pollutant degradation.

4. Conclusions

In summary, the amorphization-enabled CdSe/a-MoSe_x heterojunction achieves 97.3% MB removal upon visible-light irradiation, reaching an apparent rate constant of 0.0135 min^{−1} and markedly surpassing the individual components. The amorphous MoSe_x component introduces band-tail and defect states that not only inhibit electron-hole recombination but also promote Z-scheme interfacial charge transfer a mechanism supported by the narrowed band gap (1.38 eV), interfacial electron redistribution, and the synergistic participation of h⁺, $\bullet\text{O}_2^-$, and $\bullet\text{OH}$. This work demonstrates that amorphization engineering is a viable and scalable strategy for constructing efficient Z-scheme photocatalytic systems. Although the repeated experiments confirmed consistent photocatalytic trends, a more comprehensive statistical evaluation was not included in the present preliminary study. Future work will incorporate systematic statistical analyses, including standard deviations, error bars, confidence intervals, and significance testing, to further assess the reproducibility and quantitative reliability of the photocatalytic performance. Future studies will focus on extending this approach to other pollutant systems and optimizing the amorphous-crystalline interface for long-term stability. This work establishes amorphization engineering as a feasible route to construct direct Z-scheme heterojunctions, and future efforts will extend this strategy to other crystalline amorphous semiconductor systems, with particular emphasis on long-term cycling stability and practical application in real wastewater treatment.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/cryst16080495/s1>, Figure S1: XRD patterns of the fresh CdSe/a-MoSe_x photocatalyst and the catalyst recovered after four successive photocatalytic degradation cycles; Table S1: Qualitative comparison of the photocatalytic degradation performance of CdSe/a-MoSe_x with representative reported photocatalysts under different experimental conditions [49–52].

Author Contributions: L.T. proposed the research concept and design, was responsible for data collection and experimental implementation, and completed the writing and revision of the manuscript. R.W. supervised the research design, guided the data analysis methods, and conducted critical reviews and revisions of the paper content. H.L., D.Z., and X.S. assisted in data organization and result verification, proofread the paper format, reviewed the citation norms of references, and coordinated the submission process. R.W. and X.S. collaborated to revise the manuscript. All authors are responsible for the authenticity of the research data and the integrity of the paper content. All authors have read and agreed to the published version of the manuscript.

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