

Interfacial Charge Separation in ZrO₂-Coupled Bismuth Sulfobromide Photocatalysts for CO₂-to-CH₄ Conversion

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Abstract

Photocatalytic CO₂ reduction requires efficient charge separation and suitable surface sites for multi-electron transfer. Herein, ZrO₂-coupled bismuth sulfobromide heterojunctions were synthesized through a solvothermal method and applied to gas-phase CO₂ photoreduction. XRD, TEM, XPS, UV-vis diffuse reflectance spectroscopy, photoluminescence, and transient photocurrent measurements confirmed the formation of a strongly coupled interface between ZrO₂ and bismuth sulfobromide. The optimized heterojunction exhibited a CH₄ evolution rate of 28.7 μmol g⁻¹ h⁻¹, which was 3.4 times higher than that of pristine bismuth sulfobromide. Isotope labeling with ¹³CO₂ confirmed that methane originated from CO₂ reduction rather than surface carbon contamination. The enhanced activity was attributed to improved electron transfer from the sulfobromide phase to ZrO₂ and increased stabilization of CO₂-derived intermediates at interfacial oxygen sites.

Keywords: CO₂ photoreduction; methane production; ZrO₂ heterojunction; bismuth sulfobromide; charge separation; photocatalysis

1. Introduction

Photocatalytic CO₂ conversion provides a promising approach for coupling solar-energy utilization with carbon recycling by transforming CO₂ into energy-rich chemicals and fuels. Among the possible reduction products, CH₄ is particularly attractive because of its high energy density, compatibility with existing natural-gas infrastructure, and potential for long-term chemical energy storage. Nevertheless, selective CO₂ photoreduction to CH₄ remains challenging because the overall process involves eight-electron transfer together with multiple protonation and C–O bond-cleavage steps. These elementary processes compete with kinetically simpler pathways leading to CO, H₂, formate, and methanol. In addition, the chemical stability of CO₂, rapid recombination of photogenerated electrons and holes, insufficient visible-light utilization, and sluggish surface reaction kinetics collectively limit the rate of deep reduction to methane. Recent studies have therefore focused on heterojunction construction, defect engineering, cocatalyst deposition, and surface modification to improve both charge-carrier utilization and surface

reaction kinetics [1,2]. These results demonstrate that increasing the lifetime or density of photogenerated electrons alone is insufficient to achieve high CH₄ selectivity. Efficient photocatalysts must simultaneously provide suitable adsorption configurations for CO₂, stabilize key carbon-containing intermediates, and sustain sequential proton-coupled electron-transfer steps. Although developed for thermocatalytic CO₂ hydrogenation rather than photocatalysis, recent ensemble modeling of structurally diverse inverse ZrO₂/Cu interfaces showed that variations in local interfacial configuration can substantially alter reaction mechanisms, intrinsic reactivity, and deactivation behavior [3]. This observation emphasizes a broader principle relevant to photocatalytic systems: the catalytic function of ZrO₂-containing interfaces cannot be understood solely from their nominal composition, because local coordination and interfacial electronic structure may strongly influence the stabilization and conversion of CO₂-derived intermediates. Similar relationships between interfacial charge transfer, intermediate adsorption, and product selectivity have been observed in In₂S₃/In₂O₃, Cu–TiO₂/WO₃, and CuInSnS₄ photocatalytic systems [4,5].

Bismuth-based semiconductors have attracted extensive interest for photocatalytic CO₂ reduction because their electronic structures, optical absorption, and surface chemistry can be regulated through anion composition, vacancy formation, and heterointerface construction. Considerable attention has been given to BiOBr, BiOCl, and related bismuth oxyhalides, whose layered structures facilitate the formation of internal electric fields and provide opportunities for defect-mediated charge separation. Vacancy-rich BiOBr, WO₃/BiOBr, g-C₃N₄/BiOBr, and Bi–BiOCl/TiO₂ systems have exhibited improved carrier separation and enhanced CO₂ photoconversion compared with the corresponding individual components [6,7]. However, improved overall conversion does not necessarily result in efficient methane formation. Many bismuth-based photocatalysts predominantly generate CO, formate, or methanol because these products require fewer electron-transfer and protonation events, whereas CH₄ formation demands prolonged retention of carbon intermediates on the catalyst surface and continuous delivery of reducing equivalents. Premature desorption of CO or insufficient stabilization of highly reduced intermediates can therefore terminate the reaction before complete reduction to methane [8,9]. Consequently, designing a photocatalyst for selective CH₄ generation requires not only favorable band alignment and rapid interfacial charge separation but also surface sites capable of promoting successive hydrogenation and C–O bond cleavage. ZrO₂ offers several properties that make it a potentially useful interfacial component in such systems. It is chemically and thermally stable and contains Lewis acidic Zr centers and surface oxygen species that can participate in CO₂ adsorption and activation. Surface hydroxyl groups, coordinatively unsaturated Zr sites, and oxygen-deficient environments may alter the adsorption geometry and electronic state of CO₂-derived species [10,11]. Nevertheless, bulk ZrO₂ possesses a wide band gap, weak visible-light absorption, and relatively poor charge transport, which restrict its use as an independent visible-light photocatalyst. ZrO₂ is therefore more commonly introduced as a surface modifier, electron-accepting component,

adsorption-enhancing phase, or heterojunction partner. Studies involving Pt/ZrO₂, Mn/ZrO₂, and ZrO₂/ZnS have shown that coupling ZrO₂ with catalytically or photoactively complementary phases can enhance CO₂ adsorption, modify interfacial electron transport, and promote methane formation [12,13]. These findings suggest that the contribution of ZrO₂ extends beyond simple physical support. Properly constructed ZrO₂ interfaces may redistribute photogenerated charge, create additional adsorption environments, and modify the binding strength of carbon-containing intermediates. Such effects are especially relevant to methane formation because deep reduction requires that intermediates remain sufficiently stable to undergo successive proton-coupled electron-transfer reactions without being trapped irreversibly or released prematurely as partially reduced products. Bismuth sulfobromide represents another promising class of visible-light-responsive bismuth chalcogenide materials because the simultaneous presence of sulfide and bromide anions can produce electronic structures different from those of conventional bismuth oxyhalides. Its relatively strong visible-light response and adjustable band structure have stimulated investigations in photodetection, photovoltaic conversion, and photocatalytic pollutant degradation [14,15]. However, its potential for gas-phase CO₂ photoreduction, particularly for selective CH₄ formation, has received substantially less attention. This gap is important because gas-phase CO₂ conversion imposes requirements different from those encountered in pollutant degradation or liquid-phase photocatalysis. Charge carriers must reach accessible gas–solid interfacial sites, CO₂ and water-derived species must be adsorbed in suitable configurations, and carbon intermediates must undergo repeated electron and proton transfer without assistance from soluble sacrificial donors. Coupling bismuth sulfobromide with ZrO₂ may therefore provide complementary functions: the bismuth-containing semiconductor can serve as the principal visible-light absorber, whereas the ZrO₂-containing interface can regulate electron distribution and provide additional sites for adsorption and subsequent conversion of CO₂-derived intermediates. A properly formed heterointerface may also generate a directional driving force for charge migration, suppress bulk electron–hole recombination, and increase the probability that long-lived electrons participate in multi-electron surface reduction rather than being consumed through competing processes. Mechanistic evaluation of such heterojunction photocatalysts requires more than measurements of apparent product yield. Many reported CO₂ photoreduction studies are performed in liquid media containing sacrificial reagents, which can enhance carrier consumption but do not fully represent direct gas-phase conversion using CO₂ and water [16,17]. Reported catalytic rates are also difficult to compare because catalyst loading, irradiation intensity, spectral distribution, reactor volume, CO₂ pressure, humidity, reaction time, and product-normalization methods vary considerably among studies. More importantly, methane detection alone cannot unambiguously establish photocatalytic reduction of CO₂. Carbon-containing solvents, organic precursors, synthesis residues, adsorbed carbonate species, and adventitious surface contaminants may produce carbonaceous products under irradiation and lead to overestimation of genuine CO₂ conversion. Isotope labeling using ¹³CO₂ is therefore particularly valuable for verifying the carbon

origin of methane. Conventional photoluminescence and photocurrent measurements can provide evidence for altered recombination behavior and carrier transport, but they cannot independently establish the actual interfacial charge-transfer pathway or the chemical origin of enhanced selectivity. Their interpretation should be combined with crystallographic, microscopic, spectroscopic, and surface-chemical analyses to determine whether improvements originate from heterojunction formation, modified electronic states, increased light absorption, or changes in surface reaction sites [18,19].

Against this background, the present study develops ZrO₂-coupled bismuth sulfobromide heterojunctions through a solvothermal strategy and investigates their performance in direct gas-phase photocatalytic CO₂ reduction to CH₄. The material design is intended to integrate visible-light harvesting by the bismuth sulfobromide phase with the interfacial and surface functions provided by ZrO₂, thereby addressing charge recombination and sluggish deep-reduction kinetics within the same catalytic architecture. X-ray diffraction and transmission electron microscopy are used to determine phase composition, crystallinity, morphology, and interfacial contact, while X-ray photoelectron spectroscopy is employed to probe surface chemical states and changes in the local electronic environment associated with heterojunction formation. UV-vis diffuse reflectance spectroscopy, photoluminescence spectroscopy, and transient photocurrent measurements are combined to evaluate light absorption and photogenerated charge-carrier behavior. Particular emphasis is placed on connecting these electronic and structural characteristics with CH₄ formation rather than treating enhanced charge separation as an isolated performance descriptor. A ¹³CO₂ isotope-labeling experiment is further employed to verify that the detected methane originates from the supplied CO₂, providing direct evidence for the carbon-conversion pathway and excluding major contributions from residual carbon species. Through these complementary analyses, this study aims to clarify how the ZrO₂-containing interface alters charge separation, interfacial electron utilization, CO₂ adsorption, and the stabilization of intermediates required for sequential reduction toward CH₄. Establishing these relationships is expected to provide a mechanistic basis for understanding deep CO₂ photoreduction on bismuth-based heterojunctions and to offer a rational strategy for designing visible-light photocatalysts in which charge-transfer efficiency and surface multi-electron reaction kinetics are optimized simultaneously.

2. Materials and Methods

2.1. Catalyst Samples and Reaction Conditions

Six catalyst samples were prepared, including pure BiSBr, pure ZrO₂, and four ZrO₂/BiSBr composites containing 2, 5, 10, and 15 wt% ZrO₂. BiSBr was prepared from bismuth nitrate, thiourea, and potassium bromide by a solvothermal method. The mixed solution was placed in a Teflon-lined autoclave and heated at 160 °C for 12 h. The obtained solid was washed with ethanol and water and dried at 60 °C. ZrO₂ nanoparticles were added during synthesis to form direct

contact with BiSBr. Gas-phase CO₂ reduction was conducted in a 120 mL quartz reactor at room temperature and atmospheric pressure. For each test, 50 mg of catalyst was spread on a glass plate. Ultrapure water (5 mL) was placed at the bottom of the reactor to provide water vapour. The reactor was filled with high-purity CO₂ for 30 min before light exposure. A 300 W xenon lamp with a 420 nm cut-off filter was used. Each catalyst was tested three times, giving 18 independent tests.

2.2. Experimental Design and Control Tests

The four ZrO₂/BiSBr samples were compared with pure BiSBr and pure ZrO₂ under the same reaction conditions. The ZrO₂ content was changed to determine the amount needed for charge transfer without blocking visible-light absorption. A physical mixture of ZrO₂ and BiSBr was also tested. Its composition was the same as that of the best heterojunction sample. This control was used to separate the interface effect from simple particle mixing. Blank tests were carried out in the dark, without a catalyst, and under N₂ instead of CO₂. These tests were used to check thermal reactions, reactor contamination, and carbon products from surface residues. Before each test, the catalyst was heated at 100 °C under flowing N₂ for 1 h. This treatment removed weakly adsorbed carbon compounds. The stability of the best catalyst was tested over five cycles. Fresh CO₂ was added before each cycle.

2.3. Characterization and Quality Control

Crystal phases were identified by X-ray diffraction with Cu K α radiation. Particle shape, lattice spacing, and interface contact were examined by transmission electron microscopy and high-resolution transmission electron microscopy. Surface elements and chemical states were measured by X-ray photoelectron spectroscopy. The C 1s peak at 284.8 eV was used for energy correction. Light absorption was measured by UV–vis diffuse reflectance spectroscopy. Charge recombination was studied by steady-state photoluminescence spectroscopy. Transient photocurrent was measured in a three-electrode cell under repeated light on–off cycles. Gas samples were collected every 30 min and analysed by gas chromatography. CH₄ was measured with a flame-ionization detector and a methanizer. CO and H₂ were measured with a thermal-conductivity detector. Calibration curves were prepared with certified gas standards before each test series. Blank injections were used to check background signals. The calibration error was kept below 3%. A test was repeated when the difference among three runs was greater than 5%. Signals detected in the dark, catalyst-free, or N₂ tests were removed from the final results.

2.4. Data Processing and Calculation Methods

Gas concentrations were converted into molar amounts using the reactor volume, temperature, and pressure. The CH₄ evolution rate was calculated as

$$r_{\text{CH}_4} = \frac{n_{\text{CH}_4}}{m_{\text{cat}}t},$$

where r_{CH_4} is the CH₄ evolution rate in $\mu\text{mol g}^{-1} \text{h}^{-1}$, n_{CH_4} is the amount of CH₄ formed, m_{cat} is the catalyst mass, and t is the reaction time. The apparent quantum yield for CH₄ formation was calculated as

$$\text{AQY(\%)} = \frac{8N_{\text{CH}_4}}{N_{\text{photon}}} \times 100,$$

where N_{CH_4} is the number of CH₄ molecules and N_{photon} is the number of incident photons. The factor of eight represents the eight electrons needed to convert one CO₂ molecule into CH₄. Product formation rates were obtained from the slope of the concentration–time curves. Results were reported as mean values with standard deviations. Differences among catalyst groups were tested by one-way analysis of variance followed by Tukey's test. A value of $p < 0.05$ was considered significant.

2.5. Isotope Tracing and Interface Analysis

The carbon source of CH₄ was checked using ¹³CO₂ under the same conditions as the normal photocatalytic tests. Gas products were measured by gas chromatography–mass spectrometry. The formation of ¹³CH₄ was confirmed by the signal at $m/z=17$. The signal at $m/z=16$ was also recorded to check carbon contamination. Surface reaction species were studied by in situ diffuse reflectance infrared Fourier-transform spectroscopy under CO₂ and water-vapour flow. Spectra were recorded before light exposure and at fixed reaction times. Changes in carbonate, bicarbonate, formate, and carbonyl bands were compared among pure BiSBr, the physical mixture, and the best ZrO₂/BiSBr sample. XPS shifts, photoluminescence intensity, and photocurrent density were examined together to determine the direction of electron transfer. These results were used to explain the relation among charge separation, oxygen-site adsorption, and CH₄ formation.

3. Results and Discussion

3.1. Phase Structure and Interface Formation

The XRD patterns confirmed the formation of pure BiSBr and ZrO₂ phases. Both sets of diffraction peaks were present in the ZrO₂/BiSBr samples, and no clear impurity peaks were observed. The weak ZrO₂ peaks at low loading were related to its small content and particle size. TEM images showed that ZrO₂ nanoparticles were evenly attached to the BiSBr surface rather than forming large separate particles. The HRTEM image in Fig. 1 showed clear lattice fringes from both phases and a close contact region at the interface. This contact provided a path for electron transfer between BiSBr and ZrO₂. The shifts in the Bi, S, Br, Zr, and O XPS peaks also

indicated charge movement after coupling. Changes in the O 1s spectrum showed that oxygen-containing sites were present near the interface. These sites may improve CO₂ adsorption and help stabilize carbon-containing intermediates [20]. Wu et al. (2026) reported that BiSBr has suitable band positions for CO₂ reduction, but rapid charge recombination can limit its activity. The ZrO₂/BiSBr interface reduced this problem by providing an additional electron-transfer path. Yang et al. (2026) also found that surface oxygen sites improved CO₂ adsorption and affected product formation during photocatalytic reduction.

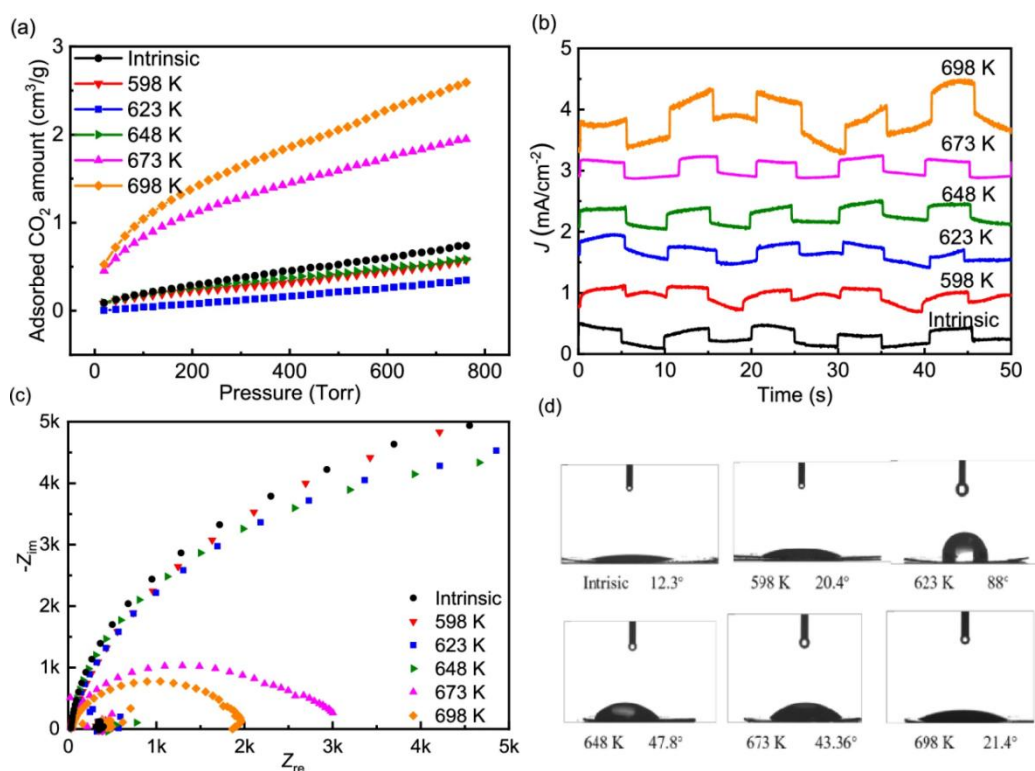


Fig. 1. Structure and charge transfer of BiSBr and ZrO₂/BiSBr photocatalysts.

3.2. Light Absorption and Charge Separation

Pure BiSBr showed strong absorption in the visible-light region, while ZrO₂ mainly absorbed ultraviolet light. The ZrO₂/BiSBr samples retained the visible-light response of BiSBr. However, high ZrO₂ loading caused a small decrease in light absorption because part of the BiSBr surface was covered. As shown in Fig. 1, the optimized sample had the lowest photoluminescence intensity and the highest transient photocurrent density. The lower photoluminescence signal showed that electron-hole recombination was reduced. The higher photocurrent response indicated that more photoexcited electrons reached the catalyst surface. These results support electron transfer from BiSBr to ZrO₂. BiSBr acted as the main light absorber, while ZrO₂ accepted electrons and supplied surface oxygen sites. This structure increased the number and lifetime of electrons available for CO₂ reduction. The effect depended on ZrO₂ loading. Low loading produced too few interface sites, whereas high loading blocked light absorption and active surface regions. Similar trends have been reported for WO₃/BiOBr and other bismuth-based

heterojunctions, in which a moderate oxide content gave the best charge separation [21,22]. Wahba et al. (2026) also reported that fast electron transfer across several interfaces improved CO₂ reduction. In comparison, the binary ZrO₂/BiSBr system provides a simpler structure for studying interface effects.

3.3. Photocatalytic CO₂-to-CH₄ Performance

Pure ZrO₂ showed little activity under visible light because of its wide band gap. Pure BiSBr produced CH₄ at a lower rate because many photoexcited charges recombined before reaching the surface. All ZrO₂/BiSBr samples showed higher CH₄ production than the two single materials. As shown in Fig. 2, the optimized heterojunction reached a CH₄ evolution rate of 28.7 $\mu\text{mol g}^{-1} \text{h}^{-1}$, which was 3.4 times that of pure BiSBr. The physical mixture with the same composition showed lower activity than the solvothermally prepared sample. This result indicates that simple particle contact was not enough to improve CH₄ formation. A well-formed interface was needed for efficient electron transfer and surface reduction. The activity first increased and then decreased as the ZrO₂ content rose. The initial increase was caused by the formation of more interface sites. The later decrease resulted from surface coverage and weaker light absorption. No clear CH₄ signal was detected in the dark, without a catalyst, or under N₂. These tests ruled out thermal reactions and common carbon contamination. The optimized sample also retained most of its activity after five cycles. However, longer tests are still needed to confirm its stability. Al Zoub et al. (2026) reported that oxygen-rich surface sites promoted the conversion of CO₂ to CH₄ by improving reactant adsorption and electron use. The present results show that interfacial oxygen sites can produce a similar effect in ZrO₂/BiSBr.

3.4. Carbon Source and Reaction Pathway

The isotope test confirmed the carbon source of methane. Under normal CO₂, the main methane signal appeared at $m/z=16$. After CO₂ was replaced by ¹³CO₂, the main signal shifted to $m/z=17$, as shown in Fig. 2. This change confirmed that CH₄ was produced from CO₂ rather than from solvents, catalyst residues, or surface carbon. The in situ infrared spectra showed carbonate and bicarbonate bands after CO₂ entered the reactor. Formate and carbonyl-related bands became stronger during light exposure. These results suggest that CO₂ was first adsorbed on oxygen-containing sites and then reduced through several electron- and proton-transfer steps [23,24]. Under light irradiation, BiSBr generated electrons and holes. The electrons moved to ZrO₂ and took part in CO₂ reduction. The holes remained mainly in BiSBr and supported water oxidation. The ZrO₂ surface also helped retain CO₂-derived intermediates, which allowed further reduction to CH₄. Rana et al. (2025) linked stronger CO₂ and H₂O adsorption with higher CH₄ production, while WU et al. (2026) used ¹³CO₂ tests to confirm the carbon source of methane. The present study combines these effects in one ZrO₂/BiSBr interface. However, the proposed pathway is mainly based on XPS, photocurrent, isotope, and infrared results. Operando EPR and X-ray

absorption measurements are needed to identify short-lived intermediates and confirm the electron-transfer route. Tests under natural sunlight and continuous-flow conditions are also needed before practical use.

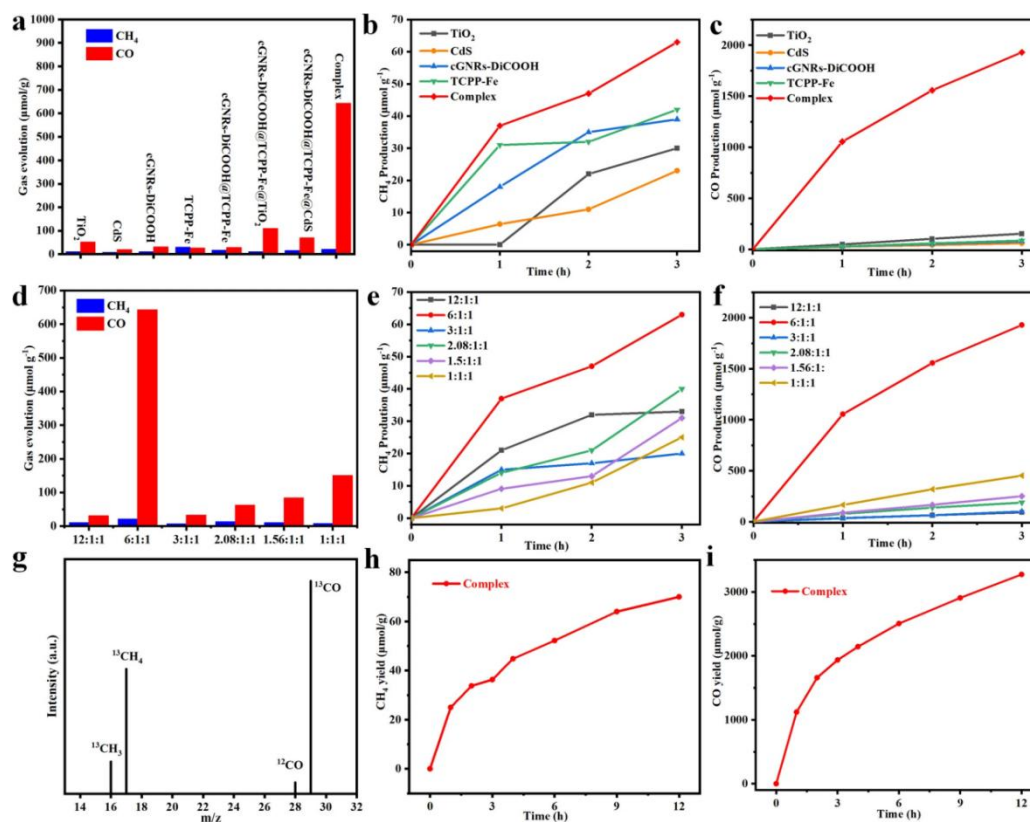


Fig. 2. CO₂-to-CH₄ activity and reaction pathway of the ZrO₂/BiSBr photocatalyst.

4. Conclusion

ZrO₂/BiSBr heterojunctions were prepared for gas-phase CO₂ reduction to CH₄. The best sample reached a CH₄ production rate of 28.7 μmol g⁻¹ h⁻¹, which was 3.4 times higher than that of pure BiSBr. Structural and surface analyses confirmed close contact between ZrO₂ and BiSBr. This interface reduced charge recombination and promoted electron transfer from BiSBr to ZrO₂. Oxygen sites on ZrO₂ also improved CO₂ adsorption and supported the formation of key reaction intermediates. The ¹³CO₂ test confirmed that CH₄ came from CO₂ rather than surface carbon residues. The main value of this study lies in combining interface charge transfer with surface oxygen sites to improve the multi-electron reduction of CO₂ to CH₄. The catalyst may be useful for solar-fuel production and gas-phase carbon conversion. However, the tests were carried out in a small batch reactor under controlled light. Longer stability tests, in situ measurements, natural-sunlight tests, and continuous-flow experiments are still needed to confirm the reaction process and practical performance.

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