Highly effective photoreduction of CO\textsubscript{2} to CO promoted by integration of CdS with molecular redox catalysts through metal–organic frameworks\textsuperscript{†}

Chunjun Chen,\textsuperscript{ab} Tianbin Wu,\textsuperscript{a} Haihong Wu,\textsuperscript{c} Huizhen Liu,\textsuperscript{ab} Qingli Qian,\textsuperscript{a} Zhimin Liu,\textsuperscript{ab} Guanying Yang\textsuperscript{b} and Buxing Han\textsuperscript{a} \textsuperscript{*ab}

We propose a strategy to synthesize ternary CdS/UiO-bpy/Co composites by integration of CdS and molecular redox catalysts through metal–organic framework (MOF) UiO-bpy. The CdS/UiO-bpy/Co composites were very effective for photocatalytic conversion of CO\textsubscript{2} to CO under visible light irradiation. The evolution rate of CO could reach 235 \textmu mol g\textsuperscript{-1} h\textsuperscript{-1}, which was a 10.2-fold improvement compared to that for the parent CdS, and selectivity for CO was 85%. The outstanding performance of the CdS/UiO-bpy/Co composites resulted from promoting the separation and migration of photo-induced charge carriers, enhancing the adsorption of CO\textsubscript{2} molecules, and offering abundant active sites for CO\textsubscript{2} reduction. As far as we know, this is the first study to integrate inorganic semiconductors and molecular redox catalysts through MOFs.

Introduction

Global energy demand and climate change have underpinned broad interest in the sustainable reduction of CO\textsubscript{2} into value added carbon products, such as CO, alcohols, and hydrocarbons. Photocatalytic reduction of CO\textsubscript{2} is regarded as an essential and important technology for mitigation of the greenhouse effect and generation of renewable transportation fuels.\textsuperscript{1–3} Although great effort has been devoted to exploring various photocatalysts,\textsuperscript{4–6} the photocatalytic efficiency is far from satisfactory, mainly because of fast recombination of the photogenerated electron–hole pairs, low specific surface area and confined active sites.\textsuperscript{7–9} Increasing the CO\textsubscript{2} adsorption ability of photocatalysts is an effective strategy to improve the CO\textsubscript{2} conversion efficiency.\textsuperscript{10,11} Due to their large surface area and well-ordered porous structure, metal–organic frameworks (MOFs) are considered to be promising candidates for gas adsorption, and can facilitate CO\textsubscript{2} adsorption.\textsuperscript{12,13} Nevertheless, the performance of MOFs in photocatalytic reduction of CO\textsubscript{2} is obstructed by the low efficiency of exciton generation and charge separation in MOFs, as well as inconsistencies between catalytic and adsorption sites.\textsuperscript{14,15}

Cadmium sulfide (CdS), with an excellent visible-light response and appropriately positioned conduction band, has been investigated extensively as a photocatalyst for photocatalytic reduction of CO\textsubscript{2}, whose applications have unfortunately been limited due to the fast recombination of photogenerated electron–hole pairs, the serious photocorrosion, the low CO\textsubscript{2} adsorption, and the lack of catalytic sites.\textsuperscript{16–18} It has been reported that the combination of CdS and MOFs can help to surmount their respective weak points by increasing the visible-light response and facilitating CO\textsubscript{2} adsorption.\textsuperscript{19} However, it is difficult for CdS/MOF composites to achieve high photocatalytic performance for CO\textsubscript{2} reduction due to the large size of CdS and lack of catalytic sites.\textsuperscript{20}

Some molecular redox catalysts containing metals such as Re, Co, Ni, and Fe are capable of reducing CO\textsubscript{2} to CO photochemically.\textsuperscript{21–22} However, they need to be fed with electrons by a photosensitizer. So far, a number of studies have been proposed to utilize inorganic semiconductors as photosensitizers,\textsuperscript{23–25} but the interaction between inorganic semiconductors and complexes is very poor, leading to difficulty in cycling experiments. It was reported that bipyridine cobalt complexes can efficiently promote the photoreduction of CO\textsubscript{2} to CO when combined with these inorganic semiconductors.\textsuperscript{26} However, bipyridine cobalt complexes can dissolve in the solution, which is unfavorable for recovery. Although a few studies have been reported on combining MOFs and molecular redox catalysts for
photocatalytic reduction of CO₂, either precious metals or complex ligands were used.²⁷,²⁸ UIO-bpy MOFs, with exceptional CO₂ adsorption capacity and stability,²⁹ have been constructed from linear 2,2’-bipyridine-5,5’-dicarboxylate (bpydc) bridging ligands and Zr₆[µ₃-O]₄[µ₁-OH]₄ SBUs. Although the bridging ligands of bpydc can coordinate with some transition metal (e.g., Co²⁺, Cu²⁺ and Ni²⁺), the CO₂ photoreduction is also obstructed by the low efficiency of visible light exciton generation.

In this work, we designed ternary CdS/UIO-bpy/Co composites. The inorganic semiconductors and the molecular redox catalysts were integrated through UIO-bpy. The CdS/UIO-bpy/Co composites showed outstanding performance for photocatalytic conversion of CO₂ to CO under visible light irradiation. To the best of our knowledge, there is no report on the combination of inorganic semiconductors and molecular redox catalysts through MOFs for photoreduction of CO₂.

Results and discussion

The method proposed to synthesize the ternary CdS/UIO-bpy/Co composites is shown schematically in Fig. 1. UIO-bpy was firstly prepared from ZrCl₄ and bpydc following a reported method.³⁰ Then, the vacuum-activated sample of UIO-bpy was treated with Cd(CH₃COO)₂.2H₂O and dimethylsulfoxide (DMSO) at 180°C for 12 h to form CdS/UIO-bpy. Because the bpydc bridging ligands can coordinate with Cd²⁺ in DMSO, and the Cd²⁺ can slowly react with DMSO to form CdS,³¹ small CdS nanoparticles were generated. The synthesized CdS/UIO-bpy samples and cobalt chloride were added into tetrahydrofuran (THF) solvent at room temperature and stirred for 12 h. Finally, the CoCl₂ was integrated into CdS/UIO-bpy by coordinating with bpydc and the ternary CdS/UIO-bpy/Co composites were obtained in this way.

The obtained CdS, UIO-bpy, CdS/UIO-bpy and CdS/UIO-bpy/Co composites were characterized using the N₂ adsorption--

Fig. 1 The route for the synthesis of the CdS/UIO-bpy/Co composites.

The contents of CdS and Co²⁺ were determined by ICP-AES (VISTA-MPX) and the results are shown in Table S1. The loading amount of Co in the CdS/UIO-bpy/Co composites was determined to be 6.18 wt%, indicating that 50.8% of the bpy units were metalated with Co in the UIO-bpy framework.

The scanning electron microscopy (SEM), transmission electron microscopy (TEM), and high-resolution TEM (HR-TEM) images of the as-synthesized UIO-bpy and CdS/UIO-bpy/Co composites are shown in Fig. 2. We can observe that the UIO-bpy is a regular octahedron, and the morphology of UIO-bpy was retained after the UIO-bpy was loaded with CdS and Co, as shown in Fig. 2A and B. It is found that the CdS nanoparticles with a diameter of about 10 nm were well dispersed on the UIO-68 framework in the CdS/UIO-bpy/Co composites, as shown in Fig. 2C and D. The lattice fringes of individual CdS particles with an interplanar spacing of ≈0.176 nm can be assigned to the (111) lattice plane of hawleyite CdS, which is in good agreement with XRD analysis (Fig. S1). Energy dispersive X-ray (EDX) elemental mapping of the CdS/UIO-bpy/Co composites in Fig. 2E shows that Cd, S, Co and Cl elements were dispersed uniformly in the composites.

The CdS, UIO-bpy, CdS/UIO-bpy and CdS/UIO-bpy/Co composites were characterized using the N₂ adsorption--
The coordination structure of the Co atom is consistent with the numbers of Co EXAFS. Similar to bpydc, biphenyl-4,4'-dicarboxylic acid can also react with ZrCl4 to produce UiO-67, and the morphology of UiO-67 was similar to that of the UiO-bpy (Fig. S3†). The preparation conditions for CdS/UiO-67 were the same as those for CdS/UiO-bpy. However, as shown in Fig. S5b†, the size of CdS particles in CdS/UiO-67 was about 50 nm, which was larger than that in CdS/UiO-bpy (10 nm, Fig. 2), mainly because biphenyl-4,4'-dicarboxylic acid does not coordinate with Cd2+. So we can conclude that the bridging ligands of bpydc played a key role in obtaining small size CdS nanoparticles. Furthermore, we also found that the amount of cadmium acetate had an important effect on the size of CdS, as shown in Fig. S6†.

In order to elucidate the local coordination environment of the Co atom in the CdS/UiO-bpy/Co composites, X-ray absorption near-edge structure (XANES) spectra and Fourier-transformed Co K-edge extended X-ray absorption fine structure (EXAFS) spectra were recorded. As shown in Fig. 3A, the absorption edge position of the CdS/UiO-bpy/Co composites is located between that of Co foil and Co3O4 and is close to that of CdS/UiO-bpy. Fourier transform of the EXAFS spectrum of CdS/UiO-bpy/Co composites decreased to 25.5 m2 g−1 due to high metal loading and the potential blocking of pore entrances by the sterically demanding CoCl2. Despite the fact that the BET surface area of the CdS/UiO-bpy/Co composites was smaller than that of pure CdS, the Co2+ adsorption amount was larger than that of pure CdS, as shown in Fig. S3,† which indicates that the UiO-bpy can facilitate CO2 adsorption.

The size of CdS in the CdS/UiO-bpy was smaller than that of the pure CdS, as can be seen from Fig. S4,† mainly resulting from the coordination of bpydc with Cd2+. In order to elucidate the role of bpydc, we replaced the bpydc with biphenyl-4,4'-dicarboxylic acid. Similar to bpydc, biphenyl-4,4'-dicarboxylic acid can also react with ZrCl4 to produce UiO-67, and the morphology of UiO-67 was similar to that of the UiO-bpy (Fig. S3a†). The preparation conditions for CdS/UiO-67 were the same as those for CdS/UiO-bpy. However, as shown in Fig. S5b,† the size of CdS particles in CdS/UiO-67 was about 50 nm, which was larger than that in CdS/UiO-bpy (10 nm, Fig. 2), mainly because biphenyl-4,4'-dicarboxylic acid does not coordinate with Cd2+. So we can conclude that the bridging ligands of bpydc played a key role in obtaining small size CdS nanoparticles. Furthermore, we also found that the amount of cadmium acetate had an important effect on the size of CdS, as shown in Fig. S6,†.

To evaluate the photocatalytic applications of CdS, UiO-bpy, CdS/UiO-bpy, and CdS/UiO-bpy/Co composites, their optical absorption properties were investigated by UV-vis diffuse reflectance spectroscopy, as shown in Fig. S8.† It is shown that the UiO-bpy can only absorb ultraviolet light. However, CdS, CdS/UiO-bpy, and CdS/UiO-bpy/Co composites exhibited strong optical absorption extending to the visible light region, suggesting that these materials can be excited by visible light irradiation to generate electron–hole pairs for redox reactions.

To gain a deeper understanding of the efficacy of photoexcited charge separation, the photo-current responses of CdS, CdS/UiO-bpy and CdS/UiO-bpy/Co composites were tested, as shown in Fig. 4A. The transient photocurrent results provide clear evidence for electron transfer within these catalysts. It is clear that the photocurrent intensity of CdS/UiO-bpy/Co was higher than that of both CdS and CdS/UiO-bpy under visible light irradiation (λ = 420 nm): CdS/UiO-bpy/Co (a), CdS/UiO-bpy (b), and CdS (c). (B) Yields of CO and H2 over CdS/UiO-bpy/Co. (C) Reusability of CdS/UiO-bpy/Co with a reaction time of 4 h for each cycle. (D) EPR spectra of CdS/UiO-bpy/Co under different conditions. Key: without light and N2 (a), with light and CO2 (b), with light and N2 (c).
light irradiation, indicating more effective charge separation in the CdS/UiO-bpy/Co composites.

The catalytic behaviors of the CdS/UiO-bpy/Co composites were examined for the photochemical reduction of CO₂ and compared with those of CdS and CdS/UiO-bpy. The CO₂ reduction was conducted under visible light irradiation, using triethanolamine (TEOA) as an electron donor and acetonitrile as the solvent. As shown in Fig. 4B, the amount of CO generated increased almost linearly with irradiation time. The CdS/UiO-bpy/Co composites showed a high CO evolution rate of 235 μmol g⁻¹ h⁻¹ under light illumination for 10 hours, which was higher than that of CdS (23 μmol g⁻¹ h⁻¹) and CdS/UiO-bpy (CO, 0 μmol g⁻¹ h⁻¹), as shown in Table 1. The turnover number of CO formation using the CdS/UiO-bpy/Co composites was 2.24. The CO evolution rate is 195 μmol g⁻¹ h⁻¹ for the CdS/UiO-bpy/Co composites under ambient pressure of CO₂ (Table 1). And the CO evolution rate was 110 μmol g⁻¹ h⁻¹ for CdS with the aid of Co-bpy as a cocatalyst, which was lower than that of the CdS/UiO-bpy/Co composites (Table 1). These results reveal that CdS/UiO-bpy/Co composites have comparable activity for CO₂ reduction compared with previously reported catalysts under comparative conditions, as can be seen from Table S3.† The yield and the morphology of the catalyst had no obvious change after five cycles, as shown in Fig. 4C and S11,† indicating the excellent stability of the CdS/UiO-bpy/Co composites. Our experiments showed that in the absence of photocatalysts or light illumination, no products were detected in the reaction, implying that CO was generated by the photocatalytic reduction of CO₂ under ambient pressure of CO₂ and the only detectable products were formed, as shown in Table 1.

In order to clarify the role of Co in the CdS/UiO-bpy/Co composites, we also loaded Ni atoms into the preformed CdS/UiO-bpy/Co composites to form ternary CdS/UiO-bpy/Co/Ni composites, which were similar to CdS/UiO-bpy/Co composites, and the results are shown in Fig. S14.† The CdS/UiO-bpy/Co/Ni composites displayed 2.1-fold higher photocatalytic activity for H₂ evolution compared with CdS/UiO-bpy, but no CO was generated, as shown in Table 1. This indicated that the Co in the composites was crucial for the photoreduction of CO₂ to CO. Electron paramagnetic resonance (EPR) spectroscopy was used to further study the function of Co atoms during the CO₂ photoreduction process. As shown in Fig. 4D(a), a strong signal for high-spin state Co²⁺ was observed for the CdS/UiO-bpy/Co composite without irradiation in a nitrogen atmosphere. However, the peak intensity of Co²⁺ was greatly weakened with light irradiation, which clearly indicates that the Co valence states transformed from high-spin state Co³⁺ to Co¹⁺ in a low-spin state (Fig. 4D(c)). The EPR signal corresponding to Co²⁺ was enhanced when CO₂ was introduced into the irradiated CdS/UiO-bpy/Co composites, implying that some of the Co⁻ was oxidized back to Co²⁺ species during the CO₂ photoreduction process (Fig. 4D(b)).† This kinetic behavior also indicates that photoexcited electrons are transferred to the Co center, and the photoreduction of CO₂ was enhanced by the valence transformation of the Co.

Conclusions

In conclusion, we demonstrate the design and fabrication of ternary CdS/UiO-bpy/Co composites, which combined inorganic semiconductors with molecular redox catalysts through MOFs using the function of the ligands. CdS nanoparticles of small size were obtained due to the presence of bridging ligands of bpydc. The ternary CdS/UiO-bpy/Co composites exhibited very high efficiency for photoreduction of CO₂, affording CO as the sole carbonaceous product with an evolution rate of 235 μmol g⁻¹ h⁻¹, and the selectivity was over 85%. We anticipate that the implantation strategy, combining inorganic semiconductors with molecular redox catalysts through MOFs, will be applicable for designing many other efficient photocatalysts for CO₂ reduction.

### Table 1

| Entry | CO (μmol g⁻¹ h⁻¹) | H₂ (μmol g⁻¹ h⁻¹) | Selectivity of CO (%) | AQYCO (%) |
|-------|------------------|------------------|----------------------|-----------|
| 1     | 235             | 41               | 85                   | 0.65      |
| 2     | 23              | 248              | 8                    | 0.063     |
| 3     | nd              | 275              | 0                    | —         |
| 4     | nd              | 580              | 0                    | —         |
| 5     | nd              | nd               | —                    | —         |
| 6     | nd              | nd               | 45                   | 0.302     |
| 7     | 110             | 132              | 45                   | 0.302     |
| 8     | 195             | 45               | 81                   | 0.54      |

* Reaction conditions: CdS/UiO-bpy/Co composites (10 mg), CH₃CN (3 mL), TEOA (0.5 mL), CO₂ (0.8 MPa), 300 W Xe lamp, λ = 420 nm, 10 h. Selectivity of CO = nCO/(nCO + nH₂). Apparent Quantum Yield (AQY): AQYCO = 2 × the number of evolved CO molecules/the number of incident photons. Using CdS instead of CdS/UiO-bpy/Co composites. Using CdS/UiO-bpy instead of CdS/UiO-bpy/Co composites. Using CdS/UiO-bpy/Ni composites instead of CdS/UiO-bpy/Co composites. In the dark. Using N₂ instead of CO₂; nd: not detected. Using CdS and Co-bpy instead of CdS/UiO-bpy/Co composites. The pressure of CO₂ was 0.1 MPa.

Comparable AQY was previously reported for CO₂ to CO conversion with a CdS-[Ni(terpyS)₃]²⁺ hybrid catalyst (0.28 ± 0.04%) and CO₂ to formate conversion with a Ru–Ag–TaON hybrid catalyst (0.48%). It is also found that higher efficiencies can be achieved by using a RuP/C₃N₄ hybrid catalyst with a suitable solvent of N,N-dimethylacetamide (5.7%). To further validate the source of the generated CO, an isotopic experiment using ¹³CO₂ as a substrate was performed under identical photocatalytic reaction conditions, and the products were analyzed by gas chromatography and mass spectrometry. As shown in Fig. S13, the peak at m/z = 29 could be assigned to ¹³CO, indicating that the carbon source of CO was the CO₂ used. When the CO₁ was replaced by N₂, no detectable products were formed, as shown in Table 1.
Conflicts of interest
There are no conflicts to declare.

Acknowledgements
The work was supported by the National Key Research and Development Program of China (2017YFA0403102 and 2017YFA0403003), National Natural Science Foundation of China (21733011, 21533011, and 21373230), and Chinese Academy of Sciences (QYZDY-SSW-SLH013).

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