Boosting photocatalytic efficiency of MoS$_2$/CdS by modulating morphology

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Abstract
CdS-based composites as the highly efficient photocatalyst have been extensively investigated in recent years due to the suitable band gap and high photocatalytic efficiency. In this study, the effect of various factors (pH, U(VI) concentration, contents, and types of photocatalyst) on photocatalytic reduction of U(VI) by MoS$_2$/CdS composite was investigated. The optimized experimental conditions (e.g., pH 7.0, 20 mg/g U(VI), and 1.0 g/L photocatalyst) was obtained by batch techniques. Approximately 97.5% of U(VI) was photo-catalytically reduced into U(IV) by 2.5 wt% MoS$_2$/CdS composite within 15 min. After 5 cycles, 2.5 wt% MoS$_2$/CdS composite still exhibited the high removal efficiency of U(VI) under 50-min irradiation, indicating the good stability. The photo-reduction mechanism of U(VI) on MoS$_2$/CdS composite was attributed to the O$^-$ radicals by quenching experiments, ESR, and XPS analysis. The findings indicate that CdS-based catalyst has a great potential for the photocatalytic reduction of uranyl in actual environmental remediation.

Keywords MoS$_2$ · CdS · Photocatalytic reduction · U(VI)

Introduction
With the development of the economy and society, mankind’s demand for energy is seriously increasing year by year (Arunachalam and Fleischer 2008; Chu and Majumdar 2012; Jacobson 2009; Jacobson and Delucchi 2011). Nuclear energy, as one of the most promising energy resources, has been widely used in countries around the world due to its high energy density and clean energy (Graves et al. 2011). Uranium has extensively been used in nuclear-related industries, which exhibits a great threat to environmental diversity and human health (Andrews and Cahill 2013). Therefore, it is very important to remove these pollutants before releasing the environment. Various methods, including chemical precipitation (B. et al. 1998), ultrafiltration (Misra et al. 2009), photocatalysis (Chang et al. 2015), and adsorption (Dolatyari et al. 2016), have been widely used to remove uranium from aqueous solutions (Xie et al. 2013). The photocatalytic reduction of U(VI) into U(IV) by various photocatalysts is regarded as a promising method due to its environmentally friendly, low cost, and high removal efficiency (Chang et al. 2015; Qin et al. 2017; Wu et al. 2017).

CdS, as a common photocatalyst, has been widely investigated to remove various environmental pollutants by photocatalysis due to the feasible band gap (~2.4 eV) and high photocatalytic activity (Liu et al. 2013). CdS exhibit the different energy band structures due to the different crystal phases such as hexagonal and cubic structures, which determines the light absorption and redox ability of photogenerated charge carriers (Yin et al. 2016; Zhang et al. 2014). However, the poor stability of CdS extends the light time prone to corrosion, which limits the actual environmental application (Chen et al. 2015; Li et al. 2019; Wu et al. 2017). Various metal disulfides (e.g., MoS$_2$) have been extensively employed to increase electron transport, which is conducive to effectively separating electron-hole pairs and decreasing band gaps (Basu et al. 2019, Haseen and Ahmad 2020; Sabaraya et al. 2021; Yin et al. 2016). For instance, MoS$_2$/CdS composites have been widely used in energy storage (e.g., hydrogen/oxygen evolution reaction) and environmental cleanup (e.g., degradation of organics and photocatalytic removal of heavy metals and radionuclides) (Chen et al. 2015; Jiang et al. 2015; Zong et al. 2008).
However, few investigations concerning the photocatalytic reduction of U(VI) on MoS2/CdS composites are available. The objectives of this study are (1) to synthesize MoS2/CdS composites and characterize them using TEM, XRD, FTIR, UV-visible diffuse reflectance spectroscopy (DRS), photoluminescence (PL) spectra, and electrochemical impedance spectroscopy (EIS) techniques; (2) to investigate the photocatalytic reduction of U(VI) by MoS2/CdS under different environmental conditions; (3) to demonstrate the interaction mechanism of U(VI) on MoS2/CdS using quenching experiments, electron spin resonance (ESR), and XPS techniques. These findings are very important for environmental scientists and material engineers to design metal sulfide-containing materials with high chemical stability and excellent removal performance toward various pollutants in actual environmental remediation.

**Experimental**

**Materials**

Cadmium acetate (Cd(CH3COOH)2·2H2O, Aladdin, 99.99% purity), ammonium molybdate ((NH4)6Mo7O24·4H2O, Aladdin, 99.99%), thiourea ((NH2)2CS, Aladdin, 99% purity), N,N-dimethylformamide (DMF), and ethanol were purchased from Tianjin Damao Chemical reagent Co., Ltd. Methanol (Me), tert-butanol (TBA), and p-b enzoquinone (p-BQ) were obtained from Aladdin (Shanghai, China). All reagents (analytical grade) were used without further purification.

**Synthesis of MoS2/CdS nanoparticles**

MoS2/CdS composites were prepared by the solvothermal method (Li et al. 2019; Wu et al. 2017; Yin et al. 2016). CdS nanospheres were firstly prepared by the hydrothermal method (Chen et al. 2015). Briefly, 0.8529 g Cd(CH3COOH)2·2H2O and 1.2179 g (NH2)2CS were dissolved in 40 mL deionized water under vigorous stirring and at room temperature for 30 min. The mixture was added to a 50-mL Teflon-lined stainless-steel autoclave and was heated at 140 °C for 24 h and then cooled to room temperature. The orange CdS was obtained by drying it at 80 °C for 12 h after washing with ethanol and deionized water several times. Additionally, 5 wt% MoS2/CdS (Figure S1b) and 10 wt% MoS2/CdS (Figure S1c) composites were also fabricated by adding two- and fourfold amounts, respectively, of (NH4)6Mo7O24·4H2O and (NH2)2CS.

**Characterization**

Transmission electron microscope (JEOL, JEM-2100F, Japan) and scanning electron microscope (Hitachi, s4800) was employed to determine morphological features. Fourier transferred infrared spectrometer (IRTracer-100 Shimadzu Corporation) were conducted using KBr disc method. Brewer-Emmett-Taylor (BET) surface area of samples was determined by TriStar II. XPS spectra were taken on an X-ray photoelectron spectroscope (Escalab 250xi instrument, Thermo ESCALAB) using monochromatic Al Kα radiation (225 W, 15 mA, 15 kV). The crystallinity of as-prepared MoS2/CdS was characterized using X-ray diffractometer (Rigaku Giegerflex D/Max B diffractometer with Cu-Kα radiation). EIS measurement was carried out on the CHI-660D electrochemical workstation. In the three-electrode system, Pt counter electrode in saturated Na2SO4 and a standard calomel reference electrode are used. PL spectra were conducted using RF-6000 photoluminescence emission spectrometer at 380 nm. The optical properties were measured by Shimadzu Corporation UV-2700 UV-visible light photometer and UV-visible diffuse reflectance spectrooscope (DRS, barium sulfate as the reflection standard).

**Photoreduction of U(VI)**

The photoreduction experiments were conducted in a photocatalytic reaction box (CEL-HXF 300, China Education Au-Light) and a xenon lamp (150 W, as the light source) with a double-walled beaker with circulated water to keep the constant temperature. Briefly, 0.05 g of MoS2/CdS was added to 50 mL 20 mg/L UO22+ solution with a small amount of methanol. The pH of the mixture was adjusted by adding small volumes of NaOH and HNO3 solutions. The suspension was pre-introduced firstly N2 2 h and then reacted in the dark for 30 min to remove dissolved oxygen from water and to achieve the adsorption equilibrium. After adsorption equilibrium, the mixtures were exposed to a photocatalytic reaction box at different time intervals (15, 30, 60, 90, and 120 min).

In the recycle experiment, the samples after photocatalytic reaction for 60 min were washed several times with deionized water, and dried it in vacuum at 60 °C for 12 h, and then the dried powder was collected to conduct the photocatalytic experiment again and repeated five times.
In the quenching experiment, photoreduction experiments were conducted by separately adding methanol, TBA, and p-BQ solution, which were used as radical scavengers for holes, OH and \( \text{O}_2^- \) radicals, respectively. After the reaction, the suspension was centrifuged at 4000 r/min for 10 min, and the concentration of uranium in the supernatant was measured by UV-vis spectrophotometer at 650 nm.

**Results and discussion**

**Characterization**

The morphology of as-prepared MoS\(_2\)/CdS was characterized by SEM and TEM images. Figure 1a and 1b shows SEM image of CdS and TEM image of 2.5 wt% MoS\(_2\)/CdS, respectively. The olive-like CdS had an irregular block-shape, rough surface and obvious aggregation (Chai et al. 2018; Chen et al. 2012). For 2.5 wt% MoS\(_2\)/CdS composite, the irregular sheet-like MoS\(_2\) was anchored at the edge of CdS, which increased the catalytic edge sites (Xu and Cao 2015). With the increase of the ratio of MoS\(_2\), the color of MoS\(_2\)/CdS composite changed from bright orange to dark orange (Fig. S1c and d of Supporting Information).

XRD analysis was used to characterize the crystal structure of MoS\(_2\)/CdS (Fig. 1c). The diffraction peaks at \( 2\theta = 24.9, 26.5, 28.3, 43.8, 48, \) and 52° corresponded to the (100), (002), (101), (110), (103), and (112) planes of cubic CdS, respectively (Alomar et al. 2019). The high-diffraction peak intensity of the (101) plane indicated the growth of CdS along the (101) crystal plane (Wu et al. 2017). The large and sharp diffraction peak intensity of 2.5 wt% MoS\(_2\)/CdS showed good crystallization. Compared to CdS, no obvious shift in the diffraction peak of MoS\(_2\)/CdS composites indicated the loading of MoS\(_2\) rather than doping. However, no diffraction peak of MoS\(_2\) may be due to the small content (Li et al. 2019). No significant change in diffraction peaks after photocatalytic reaction indicated 2.5 wt% MoS\(_2\)/CdS composite with strong stability.

Figure 1d shows the FT-IR spectra of 2.5 wt% MoS\(_2\)/CdS nanocomposites before and after the reaction in the frequency range of 400–4000 cm\(^{-1}\). The IR bands at 428 and 508 cm\(^{-1}\) correspond to the stretching vibration of the Mo–S and \((\text{S}–\text{S})^2^-\) bond, respectively (Habibi and Rahmati 2014). The stretching vibration of the Cd–S bond and the bending vibration of H\(_2\)O were observed at 630 and 1572 cm\(^{-1}\), respectively. The broad peak in the range of 3200–3500 cm\(^{-1}\) was related to the vibration of O–H bond (Khawula et al. 2016). After the reaction, no new bands and little shift indicated that main interaction mechanism of U(VI) on MoS\(_2\)/CdS composite was photoreduction rather than adsorption (Yan et al. 2009; Yuan et al. 2018).

Figure S2 shows nitrogen adsorption-desorption isotherms and corresponding pore size distribution curves of MoS\(_2\)/CdS.
CdS and 2.5 wt% MoS$_2$/CdS composite. The IV-type adsorption isotherms showed an $H_3$ hysteresis loop, indicating the aggregation of plate-like particles on slit-like pores (Misra et al. 2009; Sing et al. 1985). The main size distribution (~2 and 85 nm) suggested the formation of mesoporous and microporous 2.5 wt% MoS$_2$/CdS composite. From table S1, BET specific surface area of MoS$_2$/CdS composite (~8.8 m$^2$/g) was slightly higher than CdS (3.8 m$^2$/g) due to the increase of mesopore by adding MoS$_2$, which is beneficial to improve the catalytic performance due to accommodation of more surface active sites and promotion of the transport of charge carriers (Alomar et al. 2019).

To study the light absorption characteristics, UV-vis DRS, and PL spectra (at 500–700 nm) of 2.5 wt% MoS$_2$/CdS composite are shown in Fig. 2a and 2b, respectively. For CdS, a strong absorption band edge at ~ 530 nm corresponded to the inherent band gap absorption of hexagonal CdS (Li et al. 2019; Yin et al. 2016). It is worth noting that the gradual increase of light absorption intensities in 580–800 nm with the increase of MoS$_2$ may be due to the color shift from bright orange into dark orange MoS$_2$/CdS composite (Zhang et al. 2014). According to the calculation of Tauc plots (Supporting information), the band gap of 2.5 wt% MoS$_2$/CdS composite (2.31 eV) was significantly lower than CdS (2.43 eV, inset of Fig. 2a) owing to charge transfer from the CV of MoS$_2$ to CdS.

PL spectrum as an important tool provides important information of photo-generated electron-hole separation.

Fig. 2 Spectroscopic and electrochemical properties of MoS$_2$/CdS. a UV-vis absorbance spectra of CdS, 2.5, 5, and 10 wt% MoS$_2$/CdS, and inset of band gap by Kubelka–Munk model; b, c, and d PL spectra, transient photocurrent responses, and EIS Nyquist plots, respectively.
efficiency. The high PL intensity of CdS (Fig. 2b) indicated the high recombination centers due to trap-related states (Wu et al. 2017). The low PL intensity of 2.5 wt% MoS₂/CdS composite indicated low recombination of photogenerated electrons and holes (Yuan et al. 2018), which also reduced the photo-corrosion of CdS (Zong et al. 2008). The effective transfer of photogenerated electrons from CB of CdS into CB of MoS₂ indicated that the introduction of MoS₂ inhibited recombination of photogenerated electrons and holes, which significantly improved the photocatalytic activity (Zhang et al. 2014).

To further understand the charge separation, the photocurrent responses of CdS and 2.5 wt% MoS₂/CdS under several intermittent full-light irradiation is shown in Figure 2c. The transient photocurrent of all samples quickly reached a constant value under continuous illumination (Jiang et al. 2015), whereas the photocurrent reached to 0 after close of irradiation. Compared to CdS (4.76 μA/cm²), the high photocurrent density of 2.5 wt% MoS₂/CdS composite (31.13 μA/cm²) indicated the effective inhibit recombination of photogenerated electrons and holes due to the formation of heterostructure (Chen et al. 2015).

As shown by the Nyquist diagram of the EIS spectrum in Fig. 2d, the semicircle related to charging transfer resistance (Rct) is connected in parallel with a sub-layer capacitor (CPE). Compared to CdS, the small semicircle of 2.5 wt% MoS₂/CdS indicated the fast electron transfer due to the excellent conductivity of the introduced MoS₂ (Qin et al. 2017). The characterization results reflected that the MoS₂/CdS heterojunction improved the separation and transfer efficiency of photon-generated carriers (Xu and Cao 2015; Zhang et al. 2014).

Effect of different photocatalysts and pH

As shown in Fig. 3a, the no removal of U(VI) was observed in the absence of photocatalysts and light irradiation, which ruled out the possibility of self-photolysis of U(VI). Under the dark conditions, the adsorption of U(VI) on photocatalysts slightly increased (from 11.3 to 16.7%) with increasing doping amount of MoS₂, indicating no obvious difference in the adsorption capacity of photocatalysts for U(VI). For CdS, the low photocatalytic efficiency of CdS (i.e., ~0.6% after 20-min irradiation) was mainly due to the rapid recombination of photogenerated electrons and holes. Within 15 min irradiation, the photocatalytic efficiency of 2.5 wt% MoS₂/CdS (97.51%) was remarkably higher than 10 wt% MoS₂/CdS (65%) due to the reduction of active sites by adding aggregated MoS₂ (Zhang et al. 2020). Figure 3b shows the effect of pH on
the photoreduction efficiency of U(VI) on 2.5 wt% MoS2/CdS. Almost no removal of U(VI) at pH 3.0 may be due to the competition of H+ and U(VI). The highest removal rate of U(VI) (99.64%) was observed at pH 7.0 due to the fast transfer of generated charges of 2.5 wt% MoS2/CdS under neutral conditions (Dai et al. 2021), which limited the recombination of photogenerated electrons and holes.

**Effect of U(VI) concentration and dosage**

Figure 3c and d shows the effect of U(VI) concentrations (10, 20, and 30 ppm) and dosage (0.5, 1.0, and 2.0 mg/mL) on U(VI) photoreduction, respectively. Within 5 min irradiation, the highest removal of U(VI) at 20 ppm was consistent with the previous studies. Similarly, the highest removal efficiency of U(VI) was observed at 1.0 g/L under light irradiation. The further increase of U(VI) concentration and photocatalyst dosage leads to the decrease of U(VI) photoreduction due to the occurrence of the shielding effect after exceeding the optimal concentration and dosage. It is demonstrated that excessive MoS2 may block the light absorption of CdS (Ji et al. 2015).

**Regeneration and quenching experiments**

Figure 4a and b shows the regeneration and quenching experiments of U(VI) photoreduction on 2.5 wt% MoS2/CdS, respectively. After 5 times of recycling, 2.5 wt% MoS2/CdS still exhibited the high photocatalytic removal efficiency (>97%, Fig. 4a). In addition, no change in microstructure and crystal structure was observed after the photocatalytic reduction process (Fig. 1c and d). The regeneration results indicated that 2.5 wt% MoS2/CdS had high photoreduction efficiency, good chemical stability, and excellent reusability. The photoreduction mechanism of U(VI) on 2.5 wt% MoS2/CdS composites was investigated by quenching experiments. TBA, p-BQ, and Me were used as electron acceptors to remove ·OH, ·O2−, and holes, respectively (Wang et al. 2019). As shown in Fig. 4b, only 20% of U(VI) was photoreduced after adding p-BQ into the reaction system, indicating the significant inhibition of photocatalytic activity. The little effect of TBA and Me on U(VI) photoreduction was observed. This experiment confirmed that ·O2− radicals were responsible for U(VI) photocatalytic reduction (Yuan et al. 2018).

In order to further clarify the reaction mechanism, an electron spin resonance spin trap experiment was performed, which can detect the generation of OH and O2− in the light
experimental system. As displayed in Figs. 4c and S3, no peak and characteristic peaks of DMPO-OH and DMPO-O$_2^-$ were observed under the dark and light conditions, respectively, indicating that OH and O$_2^-$ were generated at $\lambda > 420$ nm. Compared to DMPO-OH, the intensities of DMPO-O$_2^-$ were significantly weakened after adding U(VI), which can be deduced that dissolved oxygen acts as an electron shuttle between 2.5 wt% MoS$_2$/CdS.

**XPS analysis**

XPS analysis was used to determine the photocatalytic reduction of U(VI) on CdS and 2.5 wt% MoS$_2$/CdS composite. Apart from Cd 3d and S 2p, the appearance of a low Mo 3d peak showed the successful synthesis of the MoS$_2$/CdS composite (Fig. 5a). In addition, the U 4f peak was also found for 2.5 wt% MoS$_2$/CdS composite after the reaction. As shown in Fig. 5b, the two peaks of Mo 3d at 226.0 and 232.5 eV corresponded to Mo$^{4+}$ 3d$_{5/2}$ and Mo$^{4+}$ 3d$_{3/2}$, respectively (Alomar et al. 2019). After the reaction, Mo$^{4+}$ 3d$_{5/2}$ was shifted by +0.2 eV, while Mo$^{4+}$ 3d$_{3/2}$ had no obvious shift. Similarly, S 2p spectra at 161.8 and 163.0 eV corresponded to S 2p$_{3/2}$ and S 2p$_{1/2}$, respectively (Fig. 5c). Compared to CdS, the increased binding energy and slight shift (0.1–0.2 eV) of S 2p in 2.5wt% MoS$_2$/CdS was due to the addition of MoS$_2$ (Wu et al. 2017). The slight shift of Cd 3d (i.e., Cd 3d$_{5/2}$ from 405.2 to 405.4 eV, Cd 3d$_{3/2}$ from 411.9 to 412.1 eV, Fig. S4 of SI) was due to the change of Cd 3d orbit caused by the chemical bond (Yuan et al. 2018).

For 2.5wt% MoS$_2$/CdS, the change in the binding energy of Cd 3d and S 2p may be likely to accelerate the separation of excited charges of 2.5 wt% MoS$_2$/CdS under the light irradiation, improving the photocatalytic activity. U 4f peak at 381.62 and 392.50 eV were assigned to U 4f$_{7/2}$ and U 4f$_{5/2}$ of U(VI), respectively (Fig. 5d). U 4f$_{7/2}$ can be decomposed into two peaks of 380.1 and 381.6 eV, which were attributed to U(VI) and U(IV), respectively (Zhang et al. 2020). The similarity of the U 4f$_{5/2}$ peak was also observed, indicating photoreduction of adsorbed U(VI) into U(IV) by photogenerated electrons in a short time (Wang et al. 2019).

**Mechanism analysis**

Based on the above experimental results and band structure theory, the mechanism of catalytic reduction of U(VI) with 2.5 wt% MoS$_2$/CdS catalyst under all-light is rough as follows (Chang et al. 2015):
The electrons ($e^-$) in the valence band (VB) are excited under light irradiation when the light energy is greater than or equal to the band gap of MoS$_2$/CdS ($h\nu \geq E_g$) (Gorshkov et al. 2006). Then photogenerated electrons were jumped into the conduction band (CB), an equal number of holes ($h^+$) are generated in VB (Eqn. 1, Fig. 6). The holes of VB and electrons of CB have certain oxidation and reduction ability, respectively (Yin et al. 2016). $h^+$ can react with water and organics to form OH radicals (Eqn. 2) and final degradation products (i.e., CO$_2$ and H$_2$O, Eqn. 3), respectively. The O$_2^-$ radicals were generated by reaction of $e^-$ and dissolved O$_2$ (Eqn. 4). Therefore, the adsorbed U(VI) by the active site of catalyst can be reduced to U(IV) by O$_2^-$ (Eqn. 5). The generated O$_2$ was quickly captured by $e^-$ again (Eqn. 4).

It is inferred that the formation of the p-n junction between MoS$_2$ and CdS was not only promoted the separation of photoexcited electrons and holes but also accelerated the efficient transformation of photo-generated electrons from CdS to MoS$_2$ under the irradiation of visible light, which greatly enhanced the photocatalytic activity of MoS$_2$/CdS composite for U(VI) photoreduction. Meanwhile, the holes were rapidly captured by methanol (as a hole trapping agent), which was furtherly favorable for reducing the recombination rate of electron-hole pair. For MoS$_2$/CdS composite, MoS$_2$ actually acted as the catalytic center and photocenter for absorbing solar light to generate charge carriers.

**Conclusions**

In summary, the 2.5 wt% MoS$_2$/CdS composite exhibited a high photocatalytic reduction of U(VI) (97.5% within 15 min) and fast reaction equilibrium (complete removal within 30 min) due to the narrow band gap, which can effectively inhibit the recombination of photogenerated electron-hole pairs. The high photocatalytic reduction of U(VI) (~97 %) after 5 recycles revealed the chemical stability of the MoS$_2$/CdS composite. XPS analysis indicated that adsorbed U(VI) was reduced to U(IV) by photogenerated electrons.

\[
\text{MoS}_2/\text{CdS} + h\nu \rightarrow e^- + h^+ \quad (1)
\]
\[
\text{H}_2\text{O} + h^+ \rightarrow \text{OH} + \text{H}^+ \quad (2)
\]
\[
\text{CH}_3\text{OH} + h^+ + \cdot\text{OH} \rightarrow \text{CO}_2 + \text{H}_2\text{O} \quad (3)
\]
\[
\text{O}_2 + e^- \rightarrow \cdot\text{O}_2^- \quad (4)
\]
\[
\text{U(VI)} + \cdot\text{O}_2^- \rightarrow \text{U(IV)} + 2\text{O}_2 \quad (5)
\]
These findings showed that the highly photocatalytic activity of MoS$_2$/CdS composite can be obtained by integrating structure and the suitable band position, which is crucial for the application of CdS-based composite toward the high-efficient removal of U(VI) in actual environmental cleanup.

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Data availability All data generated or analyzed during this study are included in this published article.

Author contribution Jingting Xiao: conceptualization, methodology, investigation, and writing and revising original draft. Xinshui Huang: methodology and editing. Huihui Wang: methodology, analysis, and editing. Yubing Sun: conceptualization, writing–review, revising, editing, and supervision.

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Declarations

Ethics approval Not applicable.

Consent to participate Not applicable.

Consent for publication Not applicable.

Competing interests The authors declare no competing interests.

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