

## **Electronic Supplementary Information**

# **Template synthesis of nanoparticle arrays of CdS in transparent layered double hydroxide films**

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## 1. Experimental Section

### 1.1 Preparation of Na<sub>2</sub>[Cd(EDTA)]

A mixture of CdO (0.02 mol) and ethylenediaminetetraacetic acid, C<sub>10</sub>H<sub>16</sub>O<sub>8</sub>N<sub>2</sub> (EDTA) (0.02 mol) was stirred in water (100 mL) 8 h at 100 °C. The pH of the reaction mixture was adjusted to 6.5 with 1.0 M NaOH aqueous solution. After concentration and slow evaporation, a white solid was subsequently isolated by filtration and dried under vacuum.

### 1.2 Synthesis of LDH-Cd(EDTA) by an ion-exchange method

Suspensions of Mg<sub>2</sub>Al-NO<sub>3</sub> LDH were prepared by the separate nucleation and aging steps (SNAS) method developed by our group.<sup>10</sup> The wet Mg<sub>2</sub>Al-NO<sub>3</sub> LDH (0.01 mol) was suspended in a solution of Na<sub>2</sub>[Cd(EDTA)]·xH<sub>2</sub>O (0.02 mol) in 100 mL of decarbonated water. The mixture was subjected to ultrasonic treatment for one hour, whilst the pH was controlled at 7.0 with a 1.0 M NaOH solution. The suspension was transferred into an 80 mL Teflon lined stainless autoclave and treated hydrothermally at 150 °C for 24 h. The resulting product, LDH-Cd(EDTA), was separated by centrifugation, and extensively washed with decarbonated water.

### 1.3 LDH-CdS thin films

A 10 × 30 mm quartz substrate was immersed in concentrated H<sub>2</sub>SO<sub>4</sub> for 24 h, cleaned by sonication for 30 min in a succession of deionized water, ethanol and acetone, and then dried at 60 °C. The treated quartz substrate was placed at the bottom of a glass vessel, in which a 2 wt.% LDH-Cd(EDTA) suspension was contained. Solvent evaporation in air at 60 °C for 6 h afforded a transparent thin film of LDH-Cd(EDTA) on the quartz substrate.

The film sample was placed in an evacuated and sealed glass reaction chamber. An excess of high purity H<sub>2</sub>S gas was injected into the chamber at room temperature. Upon introduction of the

H<sub>2</sub>S gas, the color of the LDH-Cd(EDTA) film in the chamber gradually turned from colorless to light yellow. The gas–solid reaction time was varied from 5, 10, 15, 20, to 30 min in order to tune the size of the CdS nanoparticles in the interlayer galleries of the LDH.

## 2. Characterization

The X-ray diffraction (XRD) patterns of the products were recorded on a Shimadzu XRD-6000 diffractometer using a high power Cu K<sub>α</sub> source, with a scan step of 0.02° and a scan range between 3° and 70° 2θ. Scanning electron microscopy (SEM) images were obtained using a Hitachi S-4700 field emission SEM at 20 kV, with the surface of the samples coated with a thin platinum layer to avoid a charging effect. Transmission electron microscopy (TEM) images were recorded on a JEOL JEM-2010 high-resolution TEM at an accelerating voltage of 200 kV. Film samples were scraped off the quartz substrate and then ultrasonically dispersed in ethanol, and the suspension was deposited on a microgrid coated with a holey carbon film. UV-visible spectra of the films were recorded at room temperature in air on a Hitachi U-3010 spectrophotometer. Elemental analyses for metals and sulfur were performed with a Shimadzu ICPS-7500 ICP instrument on solutions prepared by dissolving the samples in dilute HNO<sub>3</sub>. The X-ray photoelectron spectra (XPS) were recorded with a Physical Electronics PHI Quantum 2000 system equipped with a monochromatized Al K<sub>α</sub> X-ray source. An operating power of 25 W was used with a spot diameter of 100 μm.

## 3. Results and Discussion

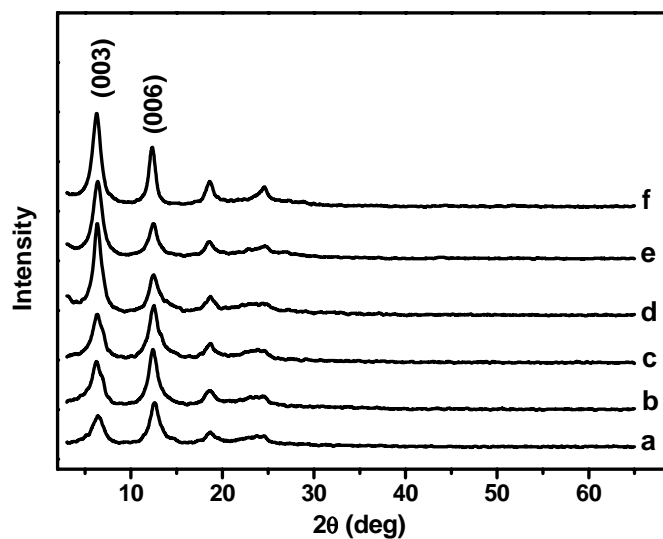


Fig. S1 XRD patterns of LDH-Cd(EDTA) film on quartz substrates before (a) and after reaction with H<sub>2</sub>S gas for 5 (b), 10 (c), 15 (d), 20 (e), and 30 min (f).

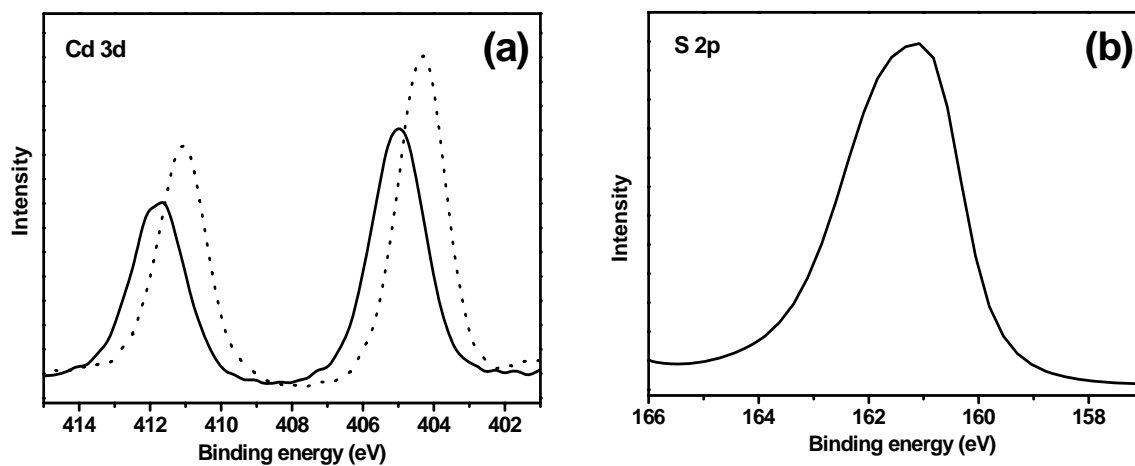


Fig. S2 XPS spectra of LDH-Cd(EDTA) films on quartz substrates before (dotted line) and after (solid lines) reaction with H<sub>2</sub>S gas for 30 min: (a) Cd 3d, (b) S 2p.