Electronic Supplementary Material (ESI) for Sustainable Energy & Fuels.

Supplementary Information

Synthesis of P-doped CdS nanorods for efficient blue LED light induced photocatalytic hydrogen evolution

Sankar Das and Young-Ho Ahn*

Department of Civil Engineering, Yeungnam University, Gyeongsan, 38541, South Korea

*Email: yhahn@ynu.ac.kr, Tel.: +82-53-810-3511, Fax: +82-53-810-4622.

Total number of Pages: 11

Total number of Figures: 9

Total number of Tables: 0

Materials

Cadmium chloride hemipentahydrate and sodium hypophosphite hydrate were obtained from Sigma Aldrich. Thiourea was purchased from Alfa Aesar. Ethylenediamine anhydrous, ethanol, methanol and lactic acid were obtained from Daejung chemicals, South Korea. All the chemicals were used as received without further purification.

Characterization

The surface morphology of the synthesized materials was analyzed by a scanning electron microscopy (FEI QUANTA FEG 250) and transmission electron microscopy (TEM: FEI TECNAI G2 F20-ST) using an accelerating voltage of 200 kV after drop casting a drop of solution on a silicon wafer and a carbon coated copper grid, respectively. High resolution transmission electron microscopy (HR-TEM) and Energy dispersive X-ray spectroscopy (EDS) analyses were performed in the above mentioned TEM using an accelerating voltage of 200 kV. Powder X-ray diffraction (XRD) patterns were carried out on a RIGAKU MiniFlex II powder diffractometer using Cu Kα radiation in the 2θ range of 20°−60° with 35 kV beam voltage and 15 mA beam current. Fourier transform infrared (FTIR) spectroscopy was carried out using Alpha Bruker spectrometer. Specific surface area was determined by the Brunauer–Emmett–Teller (BET) method using nitrogen adsorption/desorption isotherms at 77 K with 3flex Micromeritics analyzer.The UV-visible diffuse reflectance absorption spectrum of the CdS nanorods was studied by the spectrophotometer (Shimadzu UV-2600) coupled with a diffuse reflectance auxiliary with a diffuse reflectance accessory. The photoluminescence (PL) emission of catalysts was carried out using a FLUORA MAX 4P spectrofluorometer at an excitation

wavelength of 400 nm. X-ray photoelectron spectroscopy (XPS) was carried out using a monochromatized Al Kα (1486.6 eV) as X-ray source (PHI 5000 VersaProbe-II, Physical Electronics Inc., USA). The residual pressure of the analysis chamber was maintained to~10^{-8} mbar throughout the analysis. Inductively coupled plasma optical emission spectrometry (ICP-OES) analysis was performed using the PerkinElmer ICP-OES instrument (PerkinElmer, Inc., Shelton, CT, USA). For ICP-OES analysis, CdS-P nanorods was dissolved in aqua regia and the supernatant was used to estimate the phosphorous concentration in the nanorods. The Transient photocurrent responses experiments were carried on a CHI660E electrochemical workstation (CHI Instruments) in a three-electrode cell. A platinum wire and Ag/AgCl and FTO electrodes deposited with samples were used as the counter, reference and working electrodes, respectively. Electrochemical impedance spectroscopy (EIS) was measured in the frequency range 0.1 Hz to 100 kHz with an AC amplitude of 0.005 V. The produced H_2 was measured by gas chromatograph (GC, SHIMADZU) with a thermal conductivity detector.

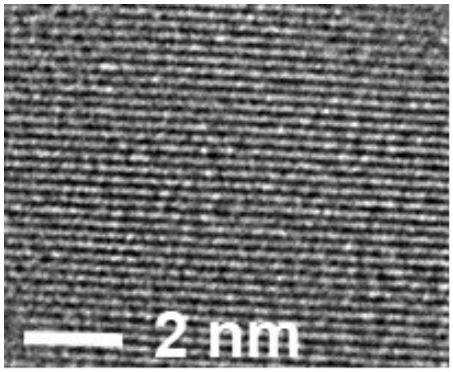

Fig. S1 HR-TEM images of (E) CdS NRs, indicating the crystal structure of hexagonal CdS nanorods.

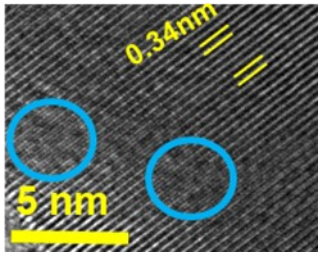

Fig. S2 HR-TEM images of CdS-$P_{0.8}$ NRs.

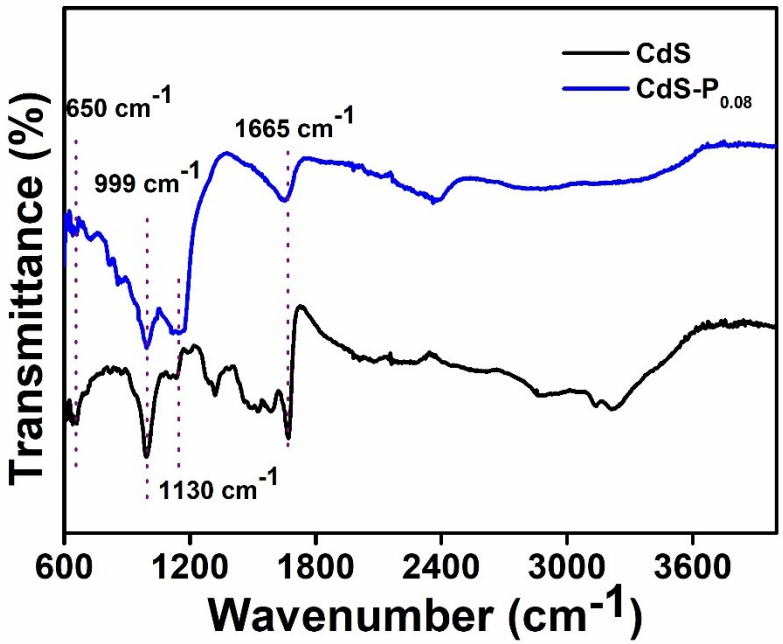

Fig. S3 FTIR spectra of pristine CdS and CdS-$P_{0.8}$ NRs samples.

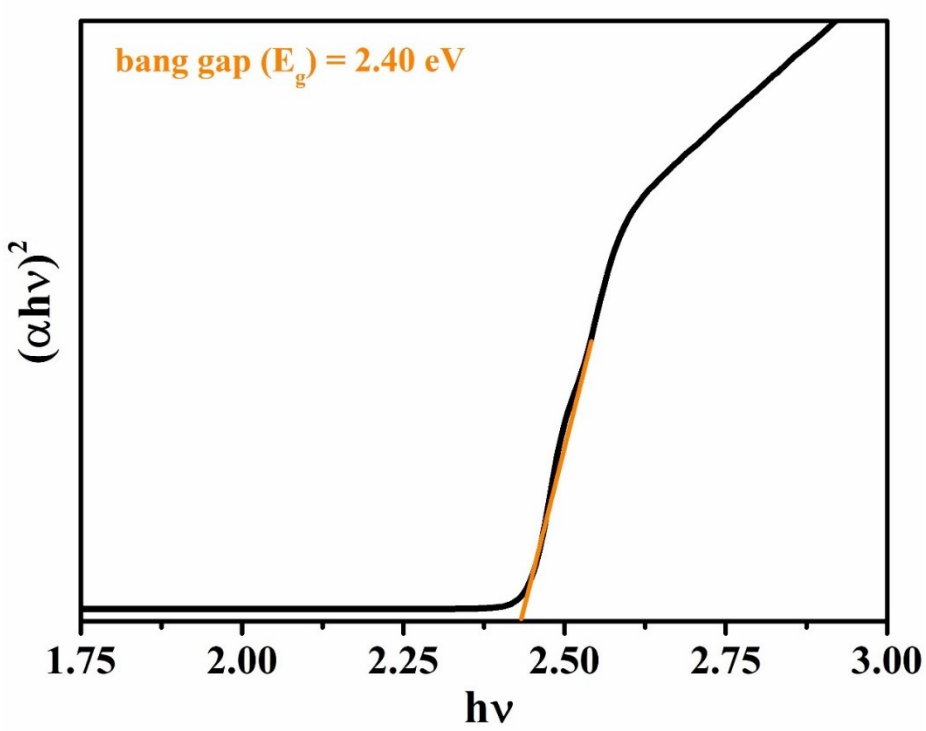

Fig. S4 Band gap energy plot from UV–Vis diffuse reflectance spectrum of the CdS NRs.

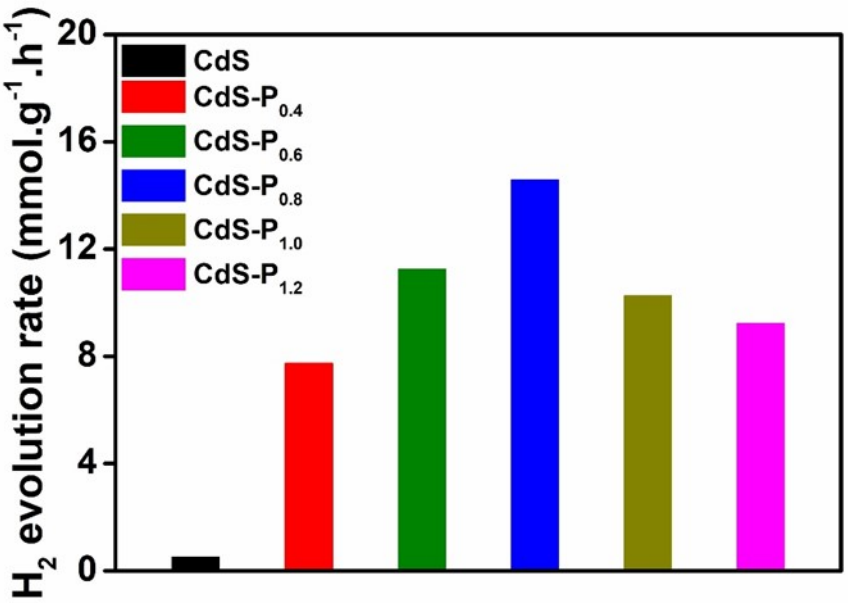

Fig. S5 Hydrogen evolution rates of a series of CdS-P and CdS NRs under blue LED light irradiation using 15 vol% lactic acid as a sacrificial agent.

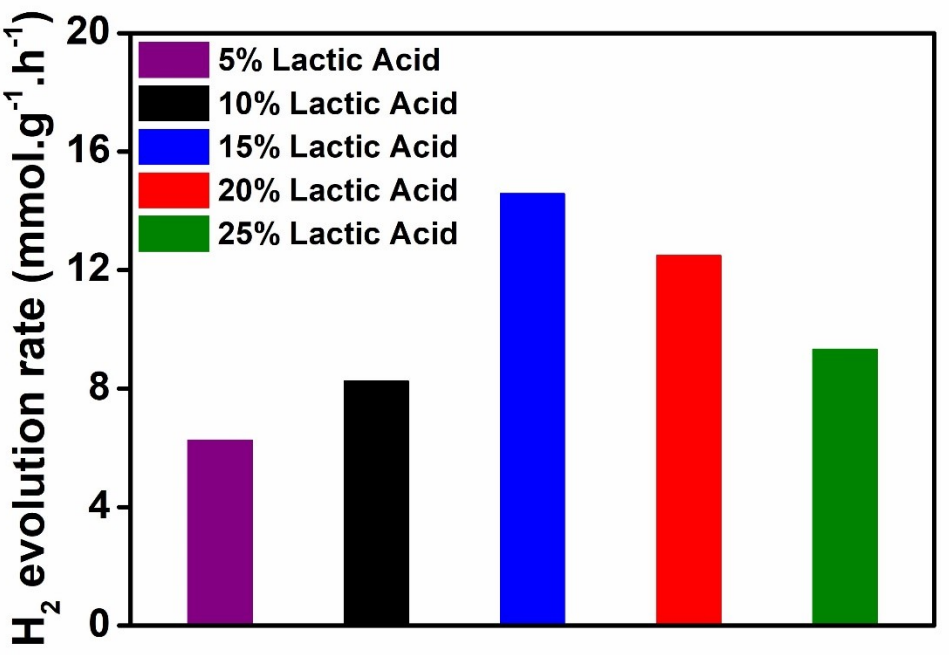

Fig.S6 Effect of lactic acid concentration on H_2 evolution performance of CdS-$P_{0.8}$ NRs.

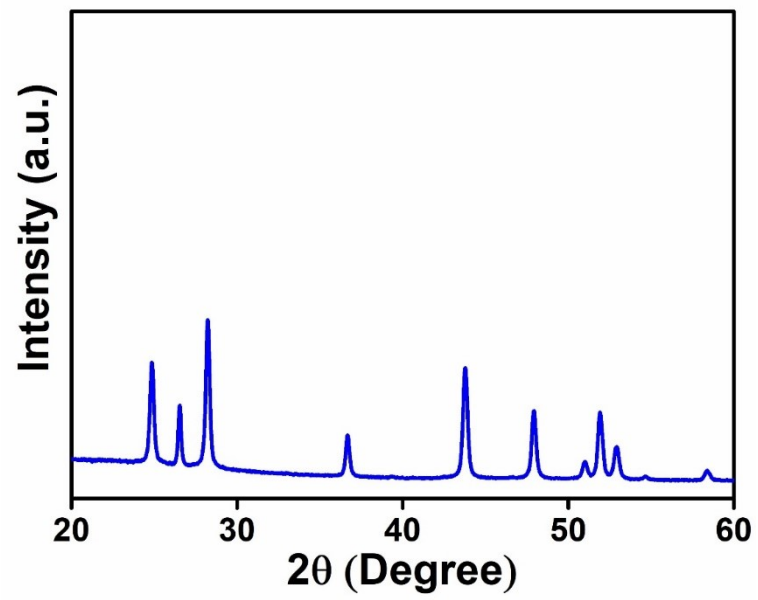

Fig. S7 XRD patterns of CdS-$P_{0.8}$ NRs after photocatalytic hydrogen production under blue LED light irradiation using lactic acid as a sacrificial agent, indicating that this results were well-matched of the CdS-$P_{0.8}$ NRs before and after photocatalytic hydrogen production reactions.

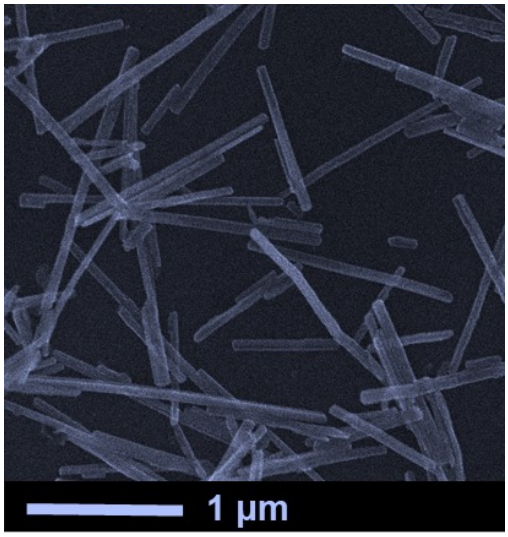

Fig. S8 SEM images of CdS-$P_{0.8}$ NRs after photocatalytic hydrogen production under blue LED light irradiation using lactic acid as a sacrificial agent.

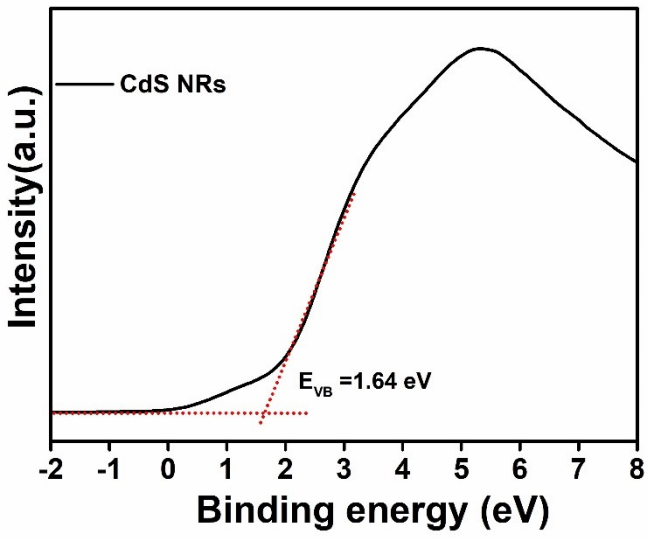

Fig. S9 Valence band XPS spectra of CdS-$P_{0.8}$ NRs.