Supplementary Information

Development of dual-functional heterostructured g-C_3N_4/CdS nanocomposites for visible-light photocatalytic dye degradation and electrochemical hydrogen production

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Materials and Methods

Synthesis of g-C_3N_4 NPs

The g-C_3N_4 photocatalyst was synthesized using a straightforward thermal treatment technique, with urea serving as the precursor, based on a method described elsewhere.[1] In typical synthesis procedure, 10 grams of urea were measured and placed in a crucible. The crucible, covered with a lid, was positioned under ambient air pressure and placed in a dryer set at 80 °C for 2 hours. Subsequently, the precursor was transferred to a muffle furnace and heated at 550 °C for 3 hours to ensure the completion of reaction. The resulting white powders were cleansed with 0.1 M nitric acid and distilled water to eliminate residual alkaline substances, such as ammonia, from the surface. Finally, the material was dried at 80 °C for 24 hours. The resultant solid was collected and finely ground to obtain the final sample.

Synthesis of CdS NPs

CdS nanoparticles were synthesized via a hydrothermal method using cadmium acetate and sodium sulfide as precursors. In this typical process, a 4 mL solution of 0.1 M cadmium acetate was mixed with 50 mL of deionized water. Next, 30 μL of analytical grade thioglycolic acid and 4 mL of 0.1 M sodium sulfide were added to the mixture. The resulting suspension was stirred for 1 h and then transferred into a Teflon-lined stainless-steel autoclave for hydrothermal treatment at 180 °C for 14 h. After naturally cooling to room temperature, the product was collected by centrifugation and washed thoroughly with deionized water and ethanol to remove impurities. Finally, the yellowish powder was dried in an oven at 105 °C for 10 hours.

Scavenger Experiments

To verify the degradation mechanism of g-C_3N_4/CdS nanocomposites (CNCS), scavenger experiments were carried out to identify the main reactive species responsible for dye degradation. Different chemical scavengers were applied to quench the active species, including ethylenediaminetetraacetic acid (EDTA) for holes (h^+), p-benzoquinone (p-BQ) for superoxide radicals ($O_2^{\bullet-}$), $AgNO_3$ for conduction band electrons (e^-), and iso-propanol for hydroxyl radicals ($OH^\bullet$). In a typical procedure, 50 mg of the 40 wt% CNCS photocatalyst was dispersed in 100 mL of a 10 ppm methylene blue (MB) solution at pH 7. After achieving adsorption–desorption equilibrium under dark conditions, a specific scavenger (50 mg of EDTA, $AgNO_3$, or p-BQ, or 5 mL of iso-propanol) was added to the reaction mixture. The suspension was then exposed to visible light irradiation to initiate the photocatalytic reaction. During the experiment, 5 mL aliquots were withdrawn at predetermined time intervals, centrifuged to remove the catalyst particles, and the remaining dye concentration was determined using UV–Vis spectrophotometry. To further examine the combined effect of all reactive species, a separate experiment was conducted by adding a mixture of scavengers consisting of 50 mg EDTA, 50 mg $AgNO_3$, 50 mg p-benzoquinone, and 5 mL iso-propanol under the same reaction conditions. The change in degradation efficiency in the presence of each scavenger was used to identify the dominant reactive species involved in the photocatalytic degradation process.

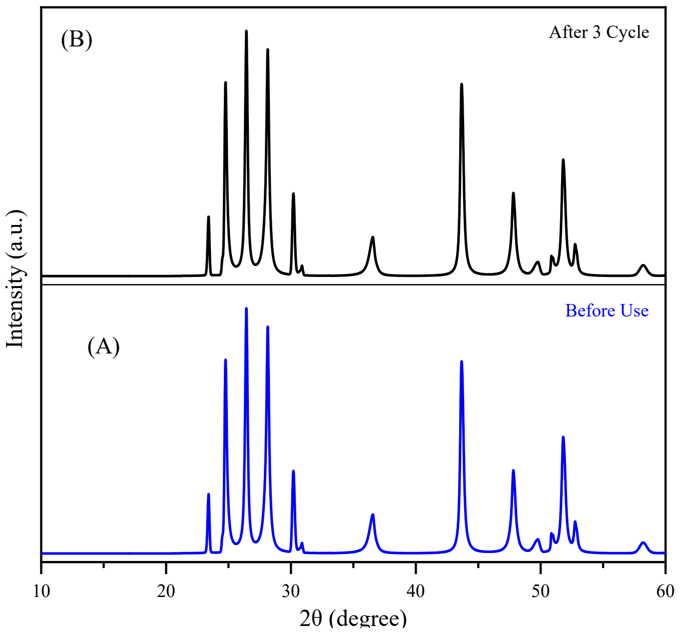

Fig. S1 XRD pattern of 40 wt% g-CNCS NC (A) before and (B) after cycling photocatalytic experiment