

Electronic Supplementary Information (ESI) available:

Niobium Pentoxide Hollow Nanospheres with Enhanced Visible Light Photocatalytic Activity

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Synthesis of niobium oxalates:

All chemicals were analytical-grade reagents, which bought from Sinopharam Chemical Reagent Beijing Co., Ltd and used without further purification. The starting materials niobium oxalates were prepared from Nb₂O₅. 3.99 g of Nb₂O₅ powder was dissolved in hydrofluoric acid (HF, 40%). Then the pH of the solution was tuned to 9.0 by adding ammonia aqueous solution. A white precipitate of niobic acid (Nb₂O₅·*n*H₂O) was observed. The precipitate was filtered and washed with distilled water and ethanol to remove the F⁻ ions completely. Then all of the obtained amorphous Nb₂O₅·*n*H₂O was mixed with 15.1 g of analytical reagent grade oxalic acid (>99.5%). The mixture was added into 100 mL distilled water and was dissolved and stirred at 90 °C for 1h. The solution was cooled at room temperature for 24 h and crystal niobium oxalates were observed via the precipitating crystallization. Finally, the sample was filtered to remove water and niobium oxalates were obtained.

Fourier transform infrared (FTIR; Model NEXUS670, Nicolet, USA) was carried out using the KBr method with spectrophotometry. The thermal behaviors of the precursor powders were studied by thermogravimetry-differential scanning calorimetry (TG-DSC, Model Labsys evo, Setaram, France) in air, at a heating rate of 10°C/min.

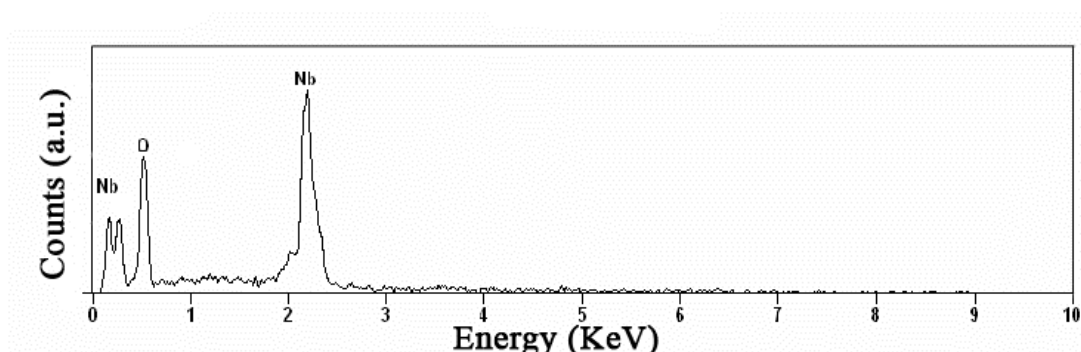


Fig. S1 Energy-dispersive X-ray (EDX) analysis of the as-prepared Nb₂O₅ hollow nanospheres

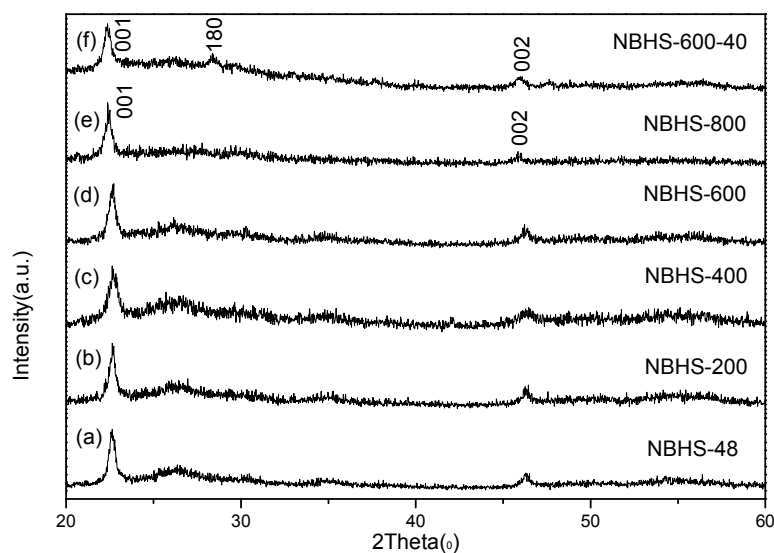


Fig. S2 XRD patterns of the Nb₂O₅ hollow nanospheres directly prepared by hydrothermal method for 48 h, (a) before heating, and calcined at (b) 200 °C, (c) 400 °C (d) 600 °C, (e) 800 °C for 2h, and (f) 600 for 40h, respectively.

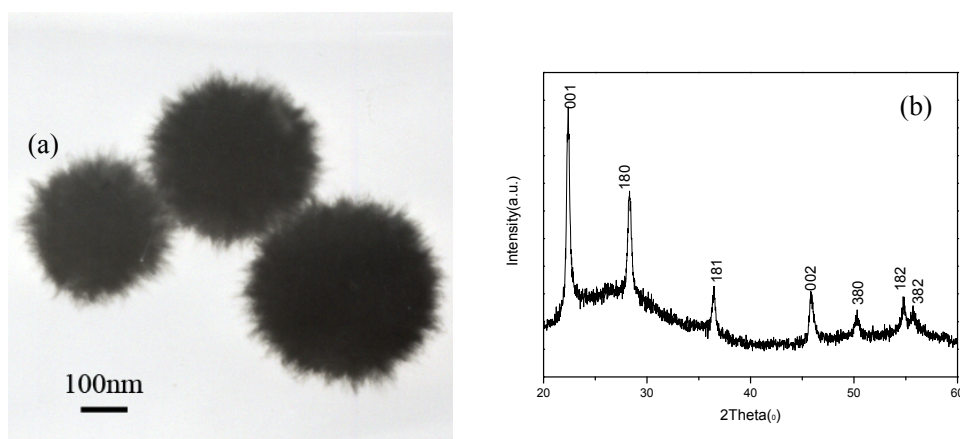


Fig. S3 (a) the TEM of the Nb_2O_5 solid spheres without $\text{NaH}_2\text{PO}_4 \cdot 2\text{H}_2\text{O}$ treated at 220 °C for 8h and (b) the XRD of that treated for 48h.

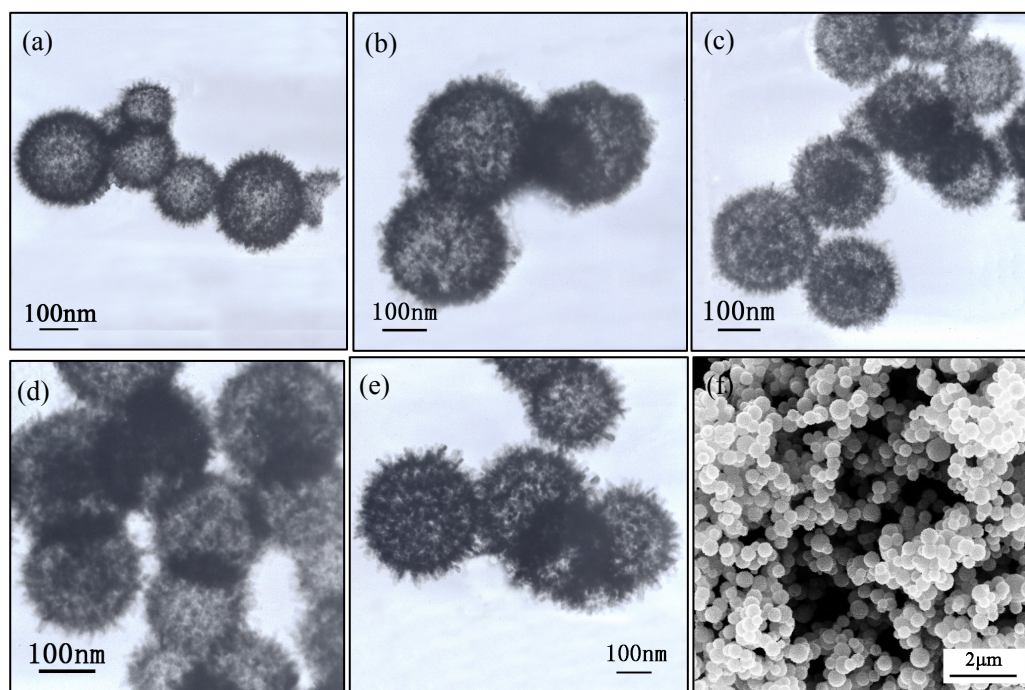


Fig. S4 TEM image of (a) NBHS-200, (b) NBHS-400, (c) NBHS-600, (d) NBHS-800, and (e) NBHS-600-40 hollow nanospheres and (f) SEM image of NBHS-600-40 hollow nanospheres.

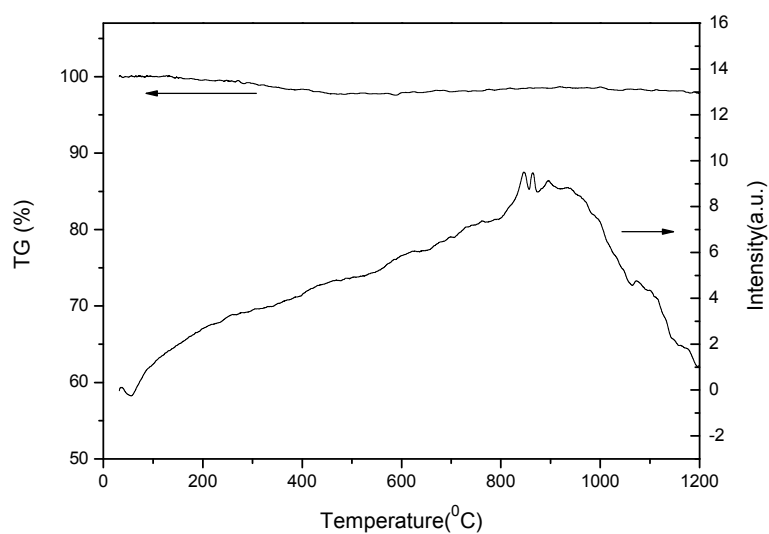


Fig. S5 shows a TG-DSC profile of NBHS-48.

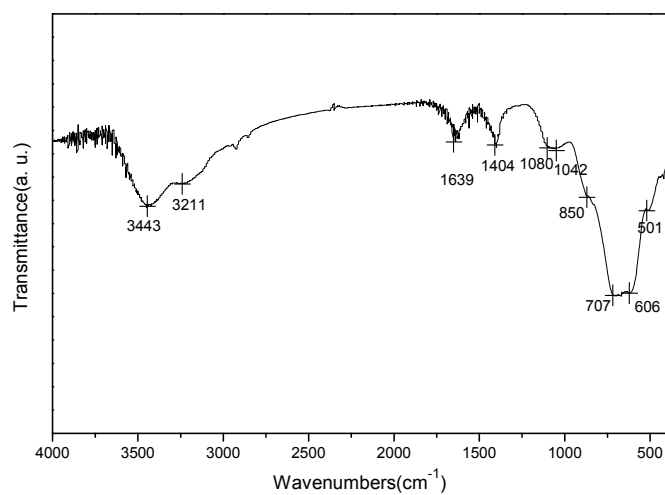


Fig. S6 FTIR spectrum of NBHS-48

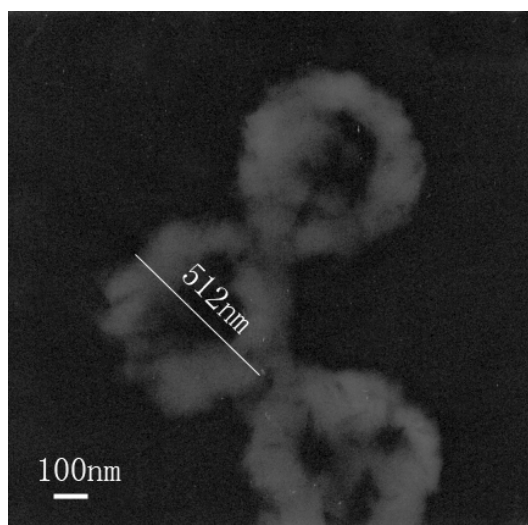


Fig. S7 TEM image of KNbO₃