#### **†** Electronic Supplementary Information

## **1.** Preparation of (Y, Gd)(OH)CO<sub>3</sub> and (Y, Gd)(OH)CO<sub>3</sub>:Ln<sup>3+</sup> (Ln = Eu, Yb, Er, and Ho) Nanoparticles

- <sup>5</sup> Typically for Y(OH)CO<sub>3</sub>, Y<sub>2</sub>O<sub>3</sub> (0.2216 g) was dissolved in dilute HNO<sub>3</sub> solution (1:1 v/v) by heating to 80 °C with agitation to form clear Y(NO<sub>3</sub>)<sub>3</sub> aqueous solution. Superfluous HNO<sub>3</sub> was evaporated by continuous heating until the pH value of the Y(NO<sub>3</sub>)<sub>3</sub> solution reaches about 4. The urea (3.0
- <sup>10</sup> g) was dissolved in deionized water (30 mL), then added the above  $Y(NO_3)_3$  solution (30 mL) drop by drop. The mixture was stirred continuously at room temperature for half an hour, and then heated to 90 °C for 2 h in the water bath. The resulting suspension was separated by centrifugation and <sup>15</sup> collected after washing with deionized water for three times.
- The  $Ln^{3+}$  (Ln = Eu, Yb, Er, and Ho) doped Y(OH)CO<sub>3</sub> were prepared by the same procedures except that a stoichiometric amount of Ln(NO<sub>3</sub>)<sub>3</sub> was added to Y(NO<sub>3</sub>)<sub>3</sub>; the preparations of matrixes and Ln<sup>3+</sup>-doped (Y<sub>0.5</sub>, Gd<sub>0.5</sub>)(OH)CO<sub>3</sub> and <sup>20</sup> Gd(OH)CO<sub>3</sub> also adopted the same route.

# 2. Preparation of Monodisperse Ellipsoid-like Hollow $YVO_4$ and $YVO_4$ :Ln<sup>3+</sup> (Ln = Eu, Yb, Er, and Ho)

For a typical YVO<sub>4</sub> synthesis, the as-obtained Y(OH)CO<sub>3</sub> <sup>25</sup> precursor was dispersed into 10 ml of deionized water by ultrasonic. Then NH<sub>4</sub>VO<sub>3</sub> (0.2319 g) was dissolved in deionized water (10 ml) with stirring and heating, and dripped into the above suspension followed by further stirring. After the mixture was agitated at 70 °C for half an hour, the final

- <sup>30</sup> pH value was measured to be about 7.5. Then it was transferred into a Teflon bottle held in a stainless-steel autoclave and heated at 200 °C for 12 h. After cooling down naturally to room temperature, the product was separated from the reaction media by centrifugation, washed several times
- <sup>35</sup> with deionized water and ethanol, and finally dried overnight at 50 °C. The power yield ratio was calculated to be as high as 90%. The  $Ln^{3+}$  (Ln = Eu, Yb, Er, and Ho) doped YVO<sub>4</sub> hollow ellipsoids were prepared by the same procedure except for using Y(OH)CO<sub>3</sub>:Ln<sup>3+</sup> as precursors.

## <sup>40</sup> 3. Preparation of Mesoprorous (Y, Gd)VO<sub>4</sub>/(Y, Gd)VO<sub>4</sub>:Ln<sup>3+</sup>@nSiO<sub>2</sub>@mSiO<sub>2</sub> (Ln = Eu, Yb, Er, and Ho) with Amino-Modification

Specifically for YVO<sub>4</sub>@nSiO<sub>2</sub>@mSiO<sub>2</sub>, the as-prepared YVO<sub>4</sub> sample (0.1 g) was treated with ethanol by <sup>45</sup> ultrasonication for half an hour, which was then separated by centrifugation and dispersed in a mixture of ethanol (40 mL), deionized water (10 mL), and concentrated ammonia aqueous solution (28 wt%, 0.5 mL). TEOS (0.03 g) was added dropwise to the above suspension. After stirring for 5 h, the <sup>50</sup> products were separated by centrifugation and washed with ethanol and water, and then redispersed in a mixed solution containing CTAB (0.15 g), deionized water (40 mL), concentrated ammonia aqueous solution (28 wt%, 0.5 mL), and ethanol (30 mL). When the resulting solution was stirred

- <sup>55</sup> for half an hour, TEOS (0.15 g) was added dropwise with stirring. After another 5 h, the products were collected and separated by centrifugation, washed with ethanol and water several times, then dried overnight at 50 °C. The structuretemplate CTAB was removed by refluxing in ethanol solution <sup>60</sup> of ammonium nitrate (NH<sub>4</sub>NO<sub>3</sub>/C<sub>2</sub>H<sub>5</sub>OH, 10 mg mL<sup>-1</sup>) for 8 h
- at 80 °C. The preparations of other samples followed the same procedures.
- Typically for the amino-modification of YVO4@nSiO2@mSiO2, the as-prepared sample of 65 YVO<sub>4</sub>@nSiO<sub>2</sub>@mSiO<sub>2</sub> (0.15 g) was dispersed into methylbenzene (50 mL) containing triethylamine (0.1 mL) and 3-aminopropyltriethoxysilane (0.5 mL). The above suspension was refluxed at 105 °C for 12 h, then separated by centrifugation, washed with methylbenzene and ethanol 70 several times. The final products were dried overnight at 50 °C, and denoted as YVO<sub>4</sub>@nSiO<sub>2</sub>@mSiO<sub>2</sub>-NH<sub>2</sub>. The aminomodification for other core-shell structural samples followed the same procedures.

### 4. In Vitro Cytotoxicity and Morphological 75 Observation

### 4.1 Cell Culture

Hela and 293T cells  $(5 \times 10^5 \text{ cells mL}^{-1})$  were maintained in Dulbecco's minimum Essential medium (DMEM Hyclone) supplemented with 10% fetal bovine serum (FBS, Hyclone) <sup>80</sup> and incubated in 5% CO<sub>2</sub> at 37 °C humidified atmosphere.

#### 4.2 MTT Assay of Ellipsoid-like Hollow YVO<sub>4</sub>

This was determined by (3-(4,5-dimethylthiazol-2-yl)-2,5diphenyl-tetra-zolium bromide (MTT, Amresco 0793) assays to determine the cytotoxicity of various concentrations of the 85 YVO<sub>4</sub> sample on Hela and 293T cells under normal physiological conditions (pH = 7.2-7.4). YVO<sub>4</sub> were dissolved in DMEM. Hela and 293T cells  $(1 \times 10^4 \text{ cells well}^{-1})$  were plated in a flat-bottom 96-well plate (Costar) in culture medium (100 µL) and incubated in 5% CO2 at 37 °C. After 90 overnight incubation, the cells were treated with compound concentrations of 25, 50, 75, 100, 125, 150, 175, 200, and 500  $\mu g$  mL<sup>-1</sup>, respectively. After incubating the cells and YVO<sub>4</sub> compound for 24 h, the cells were washed with phosphate buffered saline (PBS) for three times. The MTT reagent (5 mg  $_{95}$  mL<sup>-1</sup>) was then added to each well (10  $\mu$ L well<sup>-1</sup>, 0.5 mg mL<sup>-1</sup>) and incubated for 4 h. Subsequently the medium was removed and the water insoluble formazan crystals formed, dissolved by the addition of DMSO (100 µL well<sup>-1</sup>). The absorbance of purple formazan was measured using a Perkin Elmer 100 VICTOR<sup>3</sup> 1420 Multilabel Plate Reader at 490 nm. The relative cell viability (mean $\% \pm$  SD, n = 3) was expressed as Abs compound/Abs control, where Abs control was obtained in the absence of the compounds.

#### 4.3 Crystal Violet Staining

<sup>105</sup> HeLa cell line was cultured in DMEM (Hylcone), containing 10% FBS (Hyclone), in 6-well plate (Costar) and treated with 0, 25, 50, and 125 μg mL<sup>-1</sup> YVO<sub>4</sub> for 24 h. After the culture medium was pumped out by a vacuum pump, cells were washed twice with PBS and the PBS was discarded afterwards. Appropriate amount of 99% methanol was added 5 to fixate the cells for 10 minutes, then the methanol was discarded. Appropriate amount of crystal violet solution was

- then added, keeping it for 10 min at room temperature. Recycle the crystal violet solution and wash the cells with PBS for 5 times to decolor the background. Then pump the
- <sup>10</sup> PBS by a vacuum pump. The cells morphological features were examined by Nikon Ti-U Fluorescent Inverted Microscope and the photos were taken with a digital camera (COOPLIX P5100, Nikon).

## 5. Drug Loading and Release in Vitro

- <sup>15</sup> Typically, IBU was loaded to the hollow  $YVO_4$  sample as follows: the  $YVO_4$  sample (50 mg) was dispersed in hexane solution (10 mL) with an IBU concentration of 5 mg mL<sup>-1</sup>. The mixture was allowed to stand at room temperature for 24 h, and the loading amount was determined according to the
- $_{20}$  changed concentration of solution before and after stirred by spectrophotometric method (Beijing Puxi TU-1810-UV spectrophotometer) at 263 nm. The loaded materials were denoted IBU-loaded YVO<sub>4</sub> and rinsed three times with hexane and then dried under vacuum at 30  $^{\circ}\text{C}$ .
- <sup>25</sup> In vitro release profiles of IBU were evaluated by the dialysis method. First, a dialysis bag (cut-off molecular weight 3500 Da) was filled with a IBU-loaded YVO<sub>4</sub> buffer solution (5 mL, 10.0 mg mL<sup>-1</sup>) and soaked of a buffer solution(0.05 M, 45 mL) of pH 7.4 at 37  $\pm$  1 °C in a water bath with gentle
- <sup>30</sup> shaking. The released IBU outside of the dialysis bag was sampled at a predetermined time and measured by UV-Vis absorption at 263 nm.

The other samples were given the same treatment to evaluate the effect of drug storage and release.

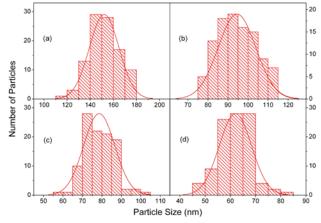
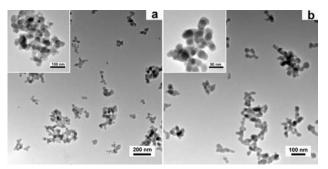


Fig. S1 Particle size distribution for  $YVO_4$  obtained by hydrothermal process for  $Y(OH)CO_3$  and  $NH_4VO_3$  solutions of (a) 30 + 30 mL, (b) 40 + 40 mL, (c) 50 + 50 mL, and (d) 60 + 60 mL.

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40 Fig. S2 TEM images of Y(OH)CO<sub>3</sub> obtained by hydrothermal process for Y(NO<sub>3</sub>)<sub>3</sub> and urea solutions of (a) 30 + 30 mL, and (b) 50 + 50 mL.

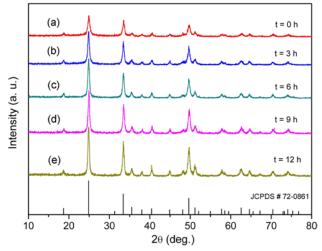


Fig. S3 XRD patterns of  $YVO_4$  for various hydrothermal times ((a) t = 0 h, (b) t = 3 h, (c) t = 6, (d) t = 9, and (e) t = 12 h).

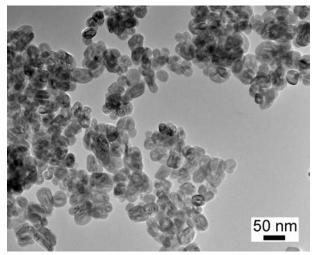


Fig. S4 TEM image of GdVO<sub>4</sub> prepared by hydrothermal process for  $Y(NO_3)_3$  and urea solutions of 50 + 50 mL.

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