Two-step Ion-Exchange Synthetic Strategy towards Monodisperse NaYF₄:Ln³⁺

Nanostructures with Multicolor Luminescence Properties

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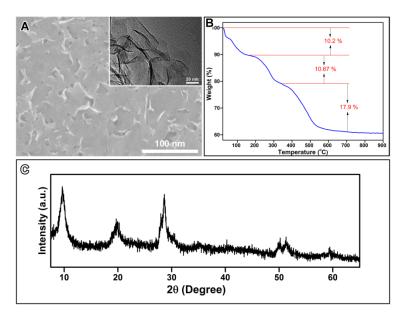


Fig. S1 SEM image (A), TEM image (inset panel), TG curve, and XRD pattern (C) of the as-prepared precursors. The XRD pattern can be indexed to $Y_2(OH)_5(NO_3) \cdot nH_2O$ (n is calculated to be about 1.8 by the TG analysis). The XRD pattern is consistent with the previous reports.^{1, 2}

The TG curve displays three distinct weight losses, which is consistent with the results of other LRHs. The initial weight loss of 10.2 % before 180 °C corresponds to the removal of the cointercalated water molecules form the interlayer gallery. Such weight loss is calculated for 1.8 mmol of water molecular per chemical formula unit. The subsequent weight loss of 10.67 % (calculated weight loss is 10.6 %) is owing to the partial dehydroxylation of the hydroxide layers. The final weight loss of 17.9 % comes from the complete decomposition of the hydroxide layers to

 Y_2O_3 (calculated weight loss is 17.6 %).

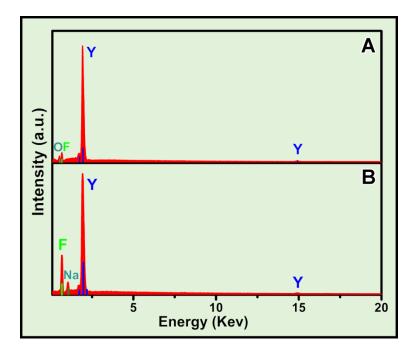


Figure S2. EDX spectra of $Y(OH)_{1.57}F_{1.43}$ precursor (A) and β -NaYF₄ product (B).

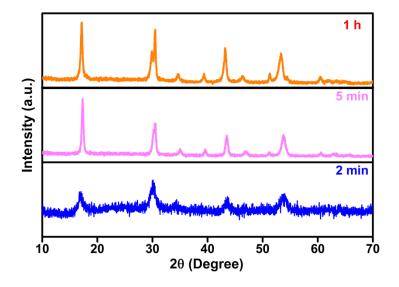


Figure S3. XRD patterns of the $Y(OH)_{1.57}F_{1.43}$ precursor prepared at different reaction intervals: 2 min, 5 min, and

1h. The time period began upon the adding of NH₄F.

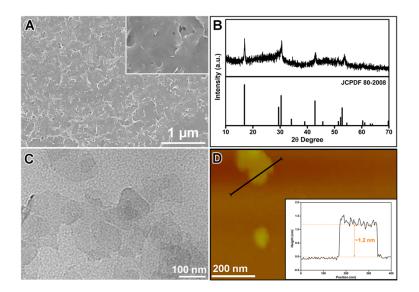


Figure S4. SEM image (A), XRD pattern (B), and TEM image (C) of the nanosheets prepared at 20 °C for 10 min.

The tapping-model AFM image with corresponding height profile (D) of the nanosheet collected with reaction time

for 2 min at 80 °C.

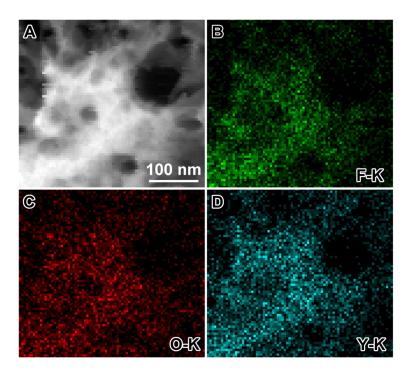


Figure S5 High-angle annular dark-field scanning transmission electron microscopy (A), and elemental mapping

images for F (B), O (C), and Y (D) of the nanosheets collected with reaction time for 2 min at 80 °C.

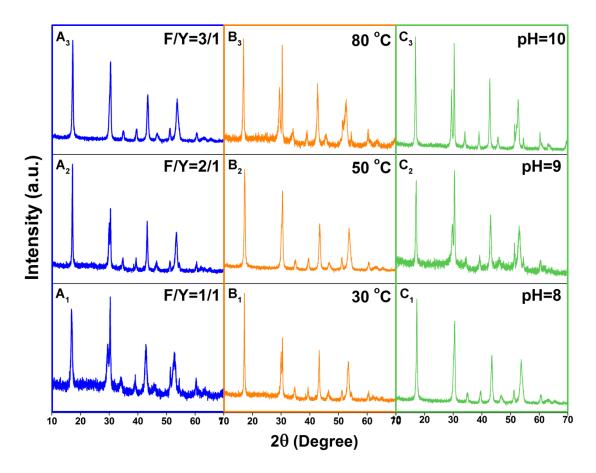


Figure S6 XRD patterns of the products prepared with different experimental variables: F/Y molar ratio (Column A), reaction temperature (Column B), and pH value (Column C).

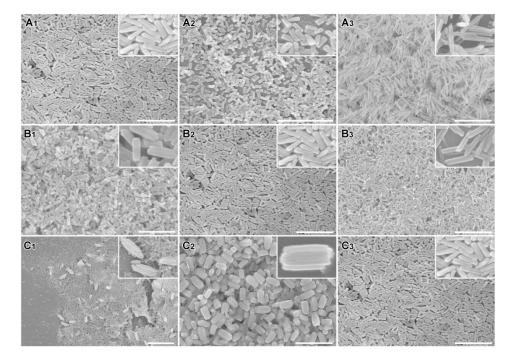


Figure S7 SEM image of the morphological evolution of the final products versus experimental

variables. The scale bars is 1 µm. **Row A**: pH=8.0, T=80 °C, and **F/Y molar ratio** (A₁) 0.5:1, (A₂) 1:1, and (A₃) 3:1. **Row B**: F/Y=0.5:1, T=80 °C, and **pH value** (B₁) 7.0, (B₂) 8.0, and (B₃) 10.0. **Row C**: F/Y=0.5:1, pH=8.0, and **reaction temperature T** (C₁) 20 °C, (C₂) 50 °C, and (C₃) 80 °C.

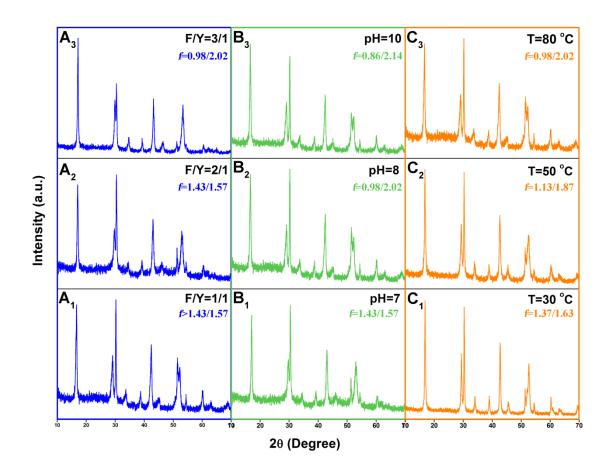
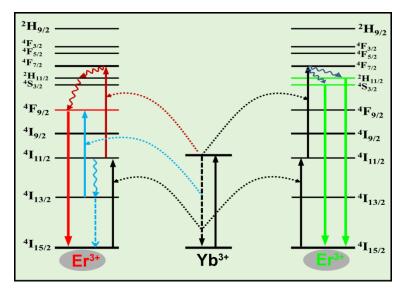


Figure S8 XRD patterns of the $Y(OH)_xF_{3-x}$ precursors prepared with different experimental variables: F/Y molar ratio (Column A), pH value (Column B), and reaction temperature (Column C). *f* is the F/OH molar ratio in $Y(OH)_xF_{3-x}$.

In a typical synthesis, dilute $NH_3 \cdot H_2O$ was introduced to the 20 mL aqueous solution containing 1 mmoL Y(NO₃)₃ until pH=8.0 under magnetic stirring. After stirring for 5 min, the LYH precipitation was collected by centrifugation to remove residual ammonia and nitric ions. The precipitation was then dispersed into 25 mL aqueous solution containing 0.5 mmol NH_4F . After magnetic stirred at 80 °C for 2 h, the products were collected and washed with deionized water and



absolute ethanol, and finally dried at 60 °C for 12 h.

Figure S9 Schematic illustration for the two-photon mechanism UC process of NaYF₄Yb³⁺,Er³⁺

sample (left for red emission at 654 nm, right for green emissions at 521 and 540 nm).