

## Electronic Supplementary Information

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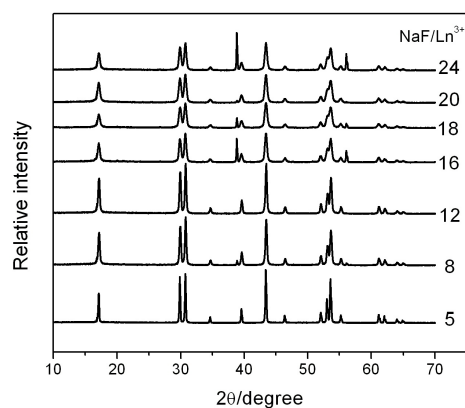
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**Title of the primary paper:** Monodisperse, Size-tunable and Highly Efficient  $\beta$ -NaYF<sub>4</sub>:Yb,Er(Tm) Up-Conversion Luminescent Nanospheres: Controllable Synthesis and Their Surface Modifications

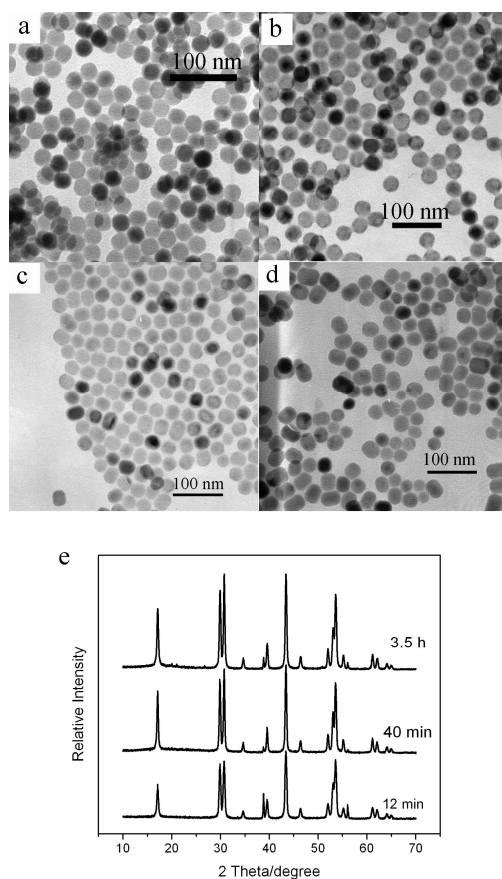
In this supplement, XRD patterns of NaYF<sub>4</sub>:Yb,Er(Tm) nanocrystals obtained under different NaF-to-Ln<sup>3+</sup> ratios (Fig. S1); effect of reaction time on the synthesis of NaYF<sub>4</sub>:Yb,Er(Tm) NPs (Fig. S2); XRD results of NaYF<sub>4</sub>:Yb,Er(Tm) nanocrystals synthesized under different ratios of OA/ODE (Fig. S3); TEM and XRD results of  $\alpha$ -NaYF<sub>4</sub>:Yb,Er and ( $\alpha$ + $\beta$ )-NaYF<sub>4</sub>:Yb,Er(Tm) nanocrystals prepared according to literature methods (Fig. S4); influence of silica coating on the UC luminescence of the  $\beta$ -NaYF<sub>4</sub>:Yb,Er NPs (Fig. S5); and the dispersibility of the NPs in several solvents (Fig. S6) are presented.

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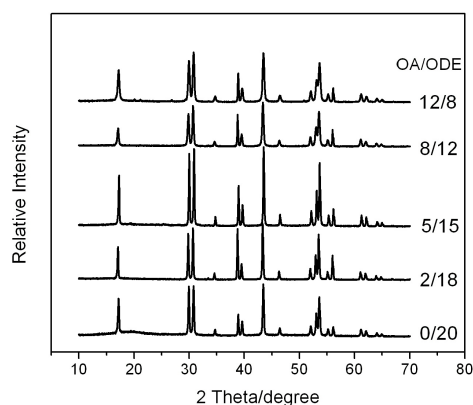
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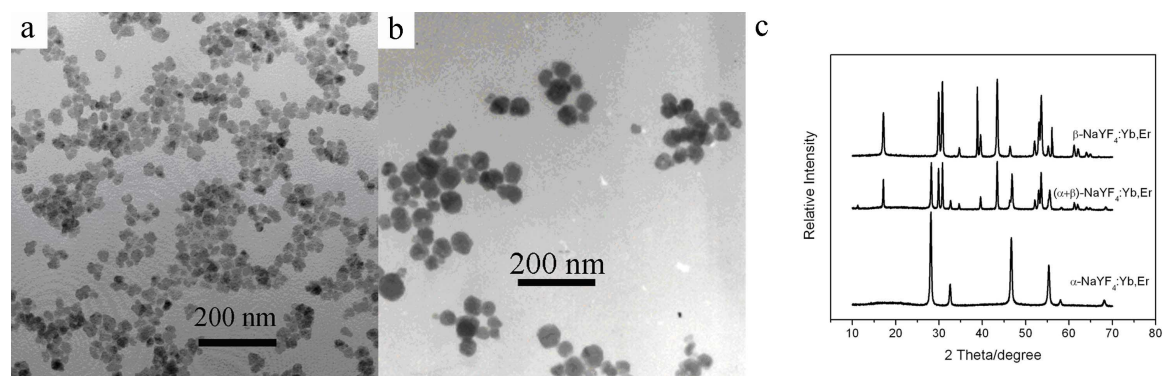
**Fig. S1:** XRD patterns of NaYF<sub>4</sub>:Yb,Er(Tm) nanocrystals obtained under different NaF-to-Ln<sup>3+</sup> ratios. Other synthetic conditions: 10 mL OA/10 mL ODE; 320 °C for 1.5 h.



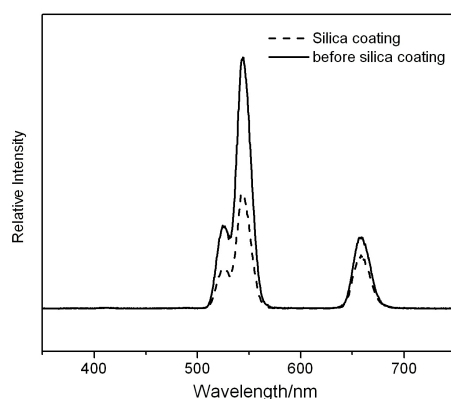
**Fig. S2:** Effect of reaction time on the synthesis of NaYF<sub>4</sub>:Yb,Er(Tm) NPs. (a-d) TEM images of the products obtained at 320 °C for 12 min, 40 min, 3.5 h, 5 h, respectively. (e) corresponding XRD patterns of NaYF<sub>4</sub>:Yb,Er(Tm) NPs obtained for different time. Other synthetic conditions: 10 mL OA/10 mL ODE; NaF-to-Ln<sup>3+</sup> ratio of 12.



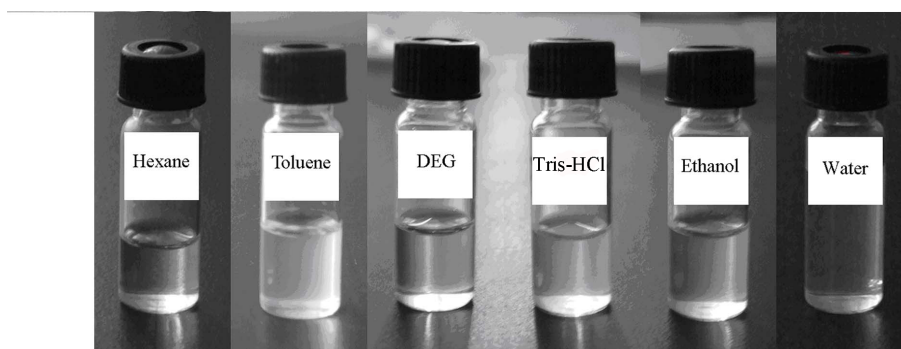
**Fig. S3:** XRD patterns of NaYF<sub>4</sub>:Yb,Er(Tm) nanocrystals obtained under different OA/ODE ratios. Other synthetic conditions: NaF-to-Ln<sup>3+</sup> ratio of 12; 320 °C for 1.5 h.



**Fig. S4:** (a) TEM image of  $\alpha$ -NaYF<sub>4</sub>:Yb,Er nanocrystals synthesized according to the literature method [1]. (b) TEM image of ( $\alpha$ + $\beta$ )-NaYF<sub>4</sub>:Yb,Er nanocrystals obtained by literature method [2]. (c) corresponding XRD patterns.



**Fig. S5:** Room-temperature UC luminescence spectra of the  $\beta$ -NaYF<sub>4</sub>:Yb,Er NPs before (solid line) and after silica coating (dash line).



**Fig. S6:** Photographs of  $\sim 2 \text{ mg mL}^{-1}$  stable colloidal solutions of the  $\beta$ -NaYF<sub>4</sub>:Yb,Er NPs dispersed in different solvents (in nonpolar solvents before surface modifications and in polar solvents after the PAA-exchange).

**References:**

- [1] Boyer, J. C.; Vetrone, F; Cuccia, L. A.; Capobianco, J. A. *J. Am. Chem. Soc.* **2006**, *128*, 7444-7445.
- [2] Yi, G. S.; Lu, H. C.; Zhao, S. Y.; Ge, Y.; Yang, W. J.; Chen, D. P.; Guo, L. H. *Nano Lett.* **2004**, *4*, 2191.