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## Electronic Supplementary Information

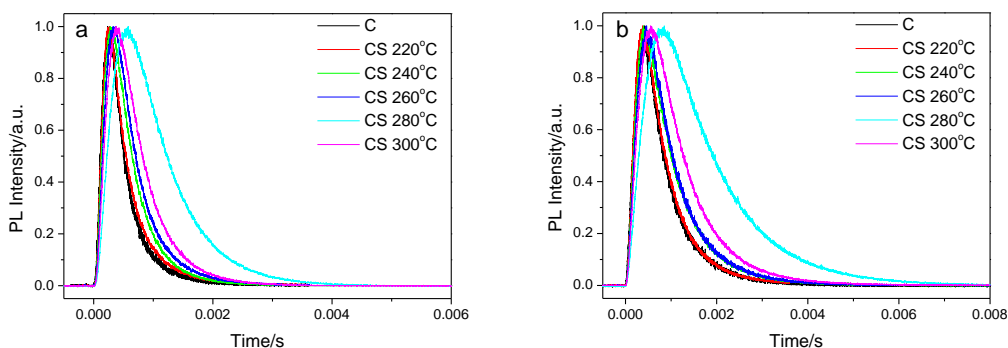
# Facile Synthesis of NaYF<sub>4</sub>:Yb, Ln/NaYF<sub>4</sub>:Yb Core/Shell Upconversion Nanoparticles via Successive Ion Layer Adsorption and Reaction Technique in One-Pot

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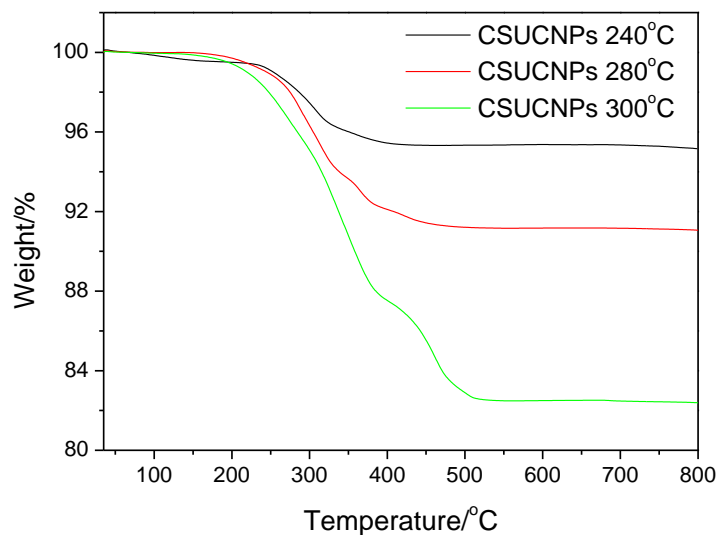


**Fig. S1** Room-temperature 477 nm (a) and 801 nm (b) UC luminescent decay spectra of NaYF<sub>4</sub>:Yb, Tm core NPs and the NaYF<sub>4</sub>:Yb, Tm/NaYF<sub>4</sub>:Yb CS NPs prepared at different shell growth temperature.  $\lambda_{\text{ex}}=980$  nm.

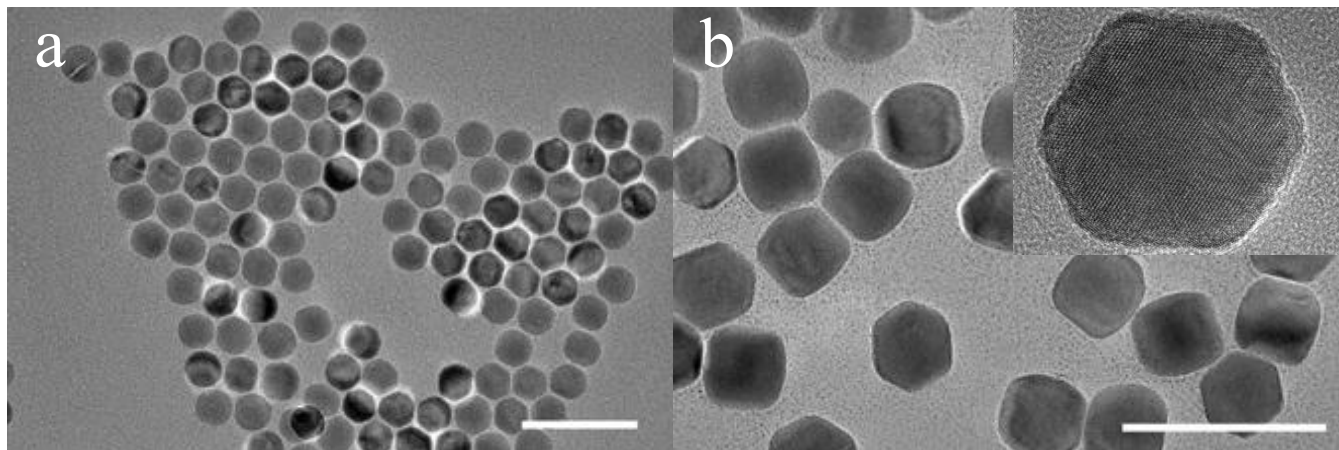
**Table S1** Upconversion luminescent lifetime of the samples shown in Fig. S1.

	C	CS-220 °C	CS-240 °C	CS-260 °C	CS-280 °C	CS-300 °C
477 nm	330 $\mu\text{s}$	365 $\mu\text{s}$	436 $\mu\text{s}$	453 $\mu\text{s}$	785 $\mu\text{s}$	543 $\mu\text{s}$
801 nm	599 $\mu\text{s}$	628 $\mu\text{s}$	738 $\mu\text{s}$	756 $\mu\text{s}$	1328 $\mu\text{s}$	848 $\mu\text{s}$

Comparing with the luminescent lifetime of core UCNPs, the lifetimes of the CS UCNPs were increased obviously. We know that:  $\tau^{-1} = k_r + k_{\text{nr}}$ , where  $\tau$  is the observed luminescent lifetime;  $k_r$  and  $k_{\text{nr}}$  are the excited state radiative and non-radiative decay rates respectively. The epitaxial growth of shell would increase the distance between the luminescence centers and surface defects of the UCNPs, separating the core from vibrational solvents or surface-bound ligands and thus leading to the decrease of the non-radiative decay rate. If  $k_r$  was kept constant,  $\tau$  would be increased correspondingly. Since the CS UCNPs grown at 280 °C showed the maximum luminescent lifetime, the non-radiative decay rate was obviously the minimum value, which indicated the optimal shell passivated effect could be achieved at this shell growth temperature.



**Fig. S2** TGA curves of NaYF<sub>4</sub>:Yb, Tm/NaYF<sub>4</sub>:Yb CS NPs prepared at 240 °C (black line), 280 °C (red line), and 300 °C (green line). The measurements were performed under the protection of N<sub>2</sub> flowing. It is observed obviously that the surface ligand adsorbed onto the UCNPs is thermal energy dependent.



**Fig. S3** Transmission electron microscopy (TEM) images of NaYF<sub>4</sub> core (a) and 14 ML CS (b) UCNPs prepared in one-pot. Inset is the HRTEM image of the 14 ML CS UCNPs. The scale bar is 100 nm. The TEM measurement was performed on a Tecnai G2 F20 S-TWIN D573 electron microscope operated at 300 kV. From the TEM results, both the NaYF<sub>4</sub> core and the 14 ML CS UCNPs are hexagonal prism structure. No interface structure could be observed from the TEM and HRTEM results. These results indicated that the NaYF<sub>4</sub> core was homogeneously passivated by the shell through our SILAR technique.