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Electronic Supplementary Information

Facile Synthesis of NaYF₄:Yb, Ln/NaYF₄:Yb Core/Shell Upconversion Nanoparticles via Successive Ion Layer Adsorption and Reaction Technique in One-Pot

Qinghui Zeng,*a Bin Xue,a,b Youlin Zhang,a Dan Wang,a,b Xiaomin Liu,a Langping Tu,a,b Haifeng Zhao,a Xianggui Konga and Hong Zhang*ac

a State Key Laboratory of Luminescence and Applications, Changchun Institute of Optics, Fine Mechanics and Physics, Chinese Academy of Sciences, 3888 Eastern South Lake Road, Changchun 130033, China

b Graduate School of Chinese Academy of Sciences, Beijing 100039, P. R. China

c Van’t Hoff Institute for Molecular Sciences, University of Amsterdam, Science Park 904, 1098 XH Amsterdam, The Netherlands
**Fig. S1** Room-temperature 477 nm (a) and 801 nm (b) UC luminescent decay spectra of NaYF₄:Yb, Tm core NPs and the NaYF₄:Yb, Tm/NaYF₄:Yb CS NPs prepared at different shell growth temperature. λₑₓ=980 nm.

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

<table>
<thead>
<tr>
<th></th>
<th>C</th>
<th>CS-220 °C</th>
<th>CS-240 °C</th>
<th>CS-260 °C</th>
<th>CS-280 °C</th>
<th>CS-300 °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>477 nm</td>
<td>330 μs</td>
<td>365 μs</td>
<td>436 μs</td>
<td>453 μs</td>
<td>785 μs</td>
<td>543 μs</td>
</tr>
<tr>
<td>801 nm</td>
<td>599 μs</td>
<td>628 μs</td>
<td>738 μs</td>
<td>756 μs</td>
<td>1328 μs</td>
<td>848 μs</td>
</tr>
</tbody>
</table>

Comparing with the luminescent lifetime of core UCNPs, the lifetimes of the CS UCNPs were increased obviously. We know that: \( \tau^{-1} = k_r + k_{nr} \), where \( \tau \) is the observed luminescent lifetime; \( k_r \) and \( k_{nr} \) 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 NaYF₄:Yb, Tm/NaYF₄: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 N₂ 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 NaYF₄ 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 NaYF₄ 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 NaYF₄ core was homogeneously passivated by the shell through our SILAR technique.