Building a near-infrared responsive upconverting nanoplatform against hypoxic tumors via NO-enhanced photodynamic therapy

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Supporting Figure Captions

\textbf{Figure S1.} X-ray diffraction patterns of NaYF\textsubscript{4}:Yb core, NaYF\textsubscript{4}:Yb@NaYbF\textsubscript{4}:Tm/Gd core-shell and NaYF\textsubscript{4}:Yb@NaYbF\textsubscript{4}:Tm/Gd@NaYF\textsubscript{4} core-shell-shell nanoparticles. The standard pattern of β-NaYF\textsubscript{4} (JCPDS 16-0334) is used as a reference.

\textbf{Figure S2.} TEM image and size distribution (inset) of the NaYF\textsubscript{4}@NaYbF\textsubscript{4}:Tm@NaYF\textsubscript{4} core–shell–shell nanoparticles with varying inner shell thickness. Scale bars for all images are 100 nm.

\textbf{Figure S3.} Comparison of upconversion emission intensity of the core-shell NaYF\textsubscript{4}:Yb/Tm@NaYF\textsubscript{4} with core-shell-shell NaYF\textsubscript{4}@NaYbF\textsubscript{4}:Tm@NaYF\textsubscript{4} nanoparticles.

\textbf{Figure S4.} Luminescence decay curves of a series of NaYF\textsubscript{4}:x\%Yb@NaYbF\textsubscript{4}:1\%Tm,30\%Gd@NaYF\textsubscript{4} (x=0, 15, 30, 45) UCNPs under 980 nm excitation.

\textbf{Figure S5.} Zeta potential of UCNPs@mSiO\textsubscript{2} and UCNPs@mSiO\textsubscript{2}-NH\textsubscript{2} in ethanol solution.

\textbf{Figure S6.} UV-Vis absorption spectra of standard curcumin (a) and RBS (b) solution with different concentrations. The calibration curve (concentration vs. absorbance) of solution at 425 nm for (c) curcumin and 350 nm for (d) RBS.
**Figure S7.** UV-Vis absorption spectra of supernatants of UCNPs@mSiO$_2$-CUR-RBS collected by centrifugation after they were soaked in deionized water, PBS buffer and cell culture medium (DMEM) for 24 hours.

**Figure S8.** Representative photographs of segregated tumors after 14 days of PDT treatment.

**Figure S9.** Standard curve of Griess assay for NO detection.
Figure S1

[Graph showing X-ray diffraction patterns for Core, Core-shell, and Core-shell-shell with JCPDS No. 16-0334]
Figure S2

NaYF₄ → Diverse shell thickness → @NaYbF₄:1%Tm → @NaYF₄
Figure S4

\[ \text{Tm}^{3+}: {^1D_2 \rightarrow ^3H_6} \]

\[
\begin{align*}
0\% \text{Yb} & \quad \tau = 221.45 \mu s \\
15\% \text{Yb} & \quad \tau = 299.95 \mu s \\
30\% \text{Yb} & \quad \tau = 414.72 \mu s \\
45\% \text{Yb} & \quad \tau = 285.10 \mu s
\end{align*}
\]
Figure S5

![Graph showing Zeta Potential (mV) for different samples: UCNP@mSiO2 and UCNP@mSiO2-NH2.](image-url)
**Figure S6**

(a) Absorbance vs. Wavelength (nm) for different concentrations of Curcumin.

(b) Absorbance vs. Wavelength (nm) for different concentrations of RBS.

(c) Line graph showing the relationship between Concentration (µg/mL) and Absorbance with the equation $Y = -0.00928 + 0.14125x$ and $R^2$ (COD) = 0.999.

(d) Line graph showing the relationship between Concentration (µg/mL) and Absorbance with the equation $Y = -0.0038 + 0.02858x$ and $R^2$ (COD) = 0.99772.
Figure S9

Equation: $Y = 0.00378 + 0.00662 \times x$

$R^2(\text{COD}) = 0.99965$