## **Supporting information**



Fig.S1 XRD patterns of the precursor prepared at various pH values pH = 7, 9, 10, 13 (a) NH<sub>3</sub>•H<sub>2</sub>O as the pH regulator; (b) using NaOH as the pH regulator.



Fig.S2 XRD patterns of the ultimate product prepared at various pH values pH = 7, 9, 10, 13 (a)  $NH_3 \bullet H_2O$  as the pH regulator; (b) using NaOH as the pH regulator.

(a)				
Formula		Gd <sub>e</sub> O <sub>s</sub> F		
Crystal system		Orthorhombic		<b>c</b>
Space group		<i>Pcmb</i> (57)		
Cell parameters		a=5.5251 b=33.4860 c=5.5829		α <b>=</b> β=ν <b>=90°</b>
Reliability factors		Sig=1.383		R <sub>wp</sub> =6.83%
		R <sub>exp</sub> =5.53%		R <sub>p</sub> =4.29%
(b)				
Atom	Wyck	x	У	z
Gd(1)	4c	0.7967	0	0.25
Gd(2)	8e	0.2906	0.0837	0.2439
Gd(3)	8e	0.7182	0.1665	0.2348
Gd(4)	4c	0.2864	0.25	0.2899
0(1)	8e	0.49	0.0417	0.025
0(2)	8e	0.464	0.1253	0.99
0(3)	8e	0.448	0.2097	0.006
F(1)	8e	0.448	0.2097	0.006
F(2)	8e	0.031	0.0360	0.426
F3)	8e	0.928	0.111	0.11
F(4)	8e	0.09	0.182	0.368
F(5)	4d	0.903	0.251	0.149

Table.S1 (a) Refinement structure parameters and reliability factors and (b) atom site parameters.



Fig.S3 SEM images of precursor while using NH<sub>4</sub>F as fluorine source and diluted NH<sub>3</sub>•H<sub>2</sub>O as pH regulator (a1) pH=7, (a2) pH=9, (a3) pH=10, (a4) pH=13; while using NH<sub>4</sub>F as fluorine source and diluted NaOH as pH regulator (b1) pH=7, (b2) pH=9, (b3) pH=10, (b4) pH=13; while using NaF as fluorine source and diluted NH<sub>3</sub>•H<sub>2</sub>O as pH regulator (c1) pH=7, (c2) pH=9, (c3) pH=10, (c4) pH=13.



Fig.S4 PL spectra under UV (a) emission of Gd(OH)<sub>2.14</sub> $F_{0.86}$ : x% Eu<sup>3+</sup> (1 ≤x ≤25); (b) excitation of Gd(OH)<sub>2.14</sub> $F_{0.86}$  and (c) emission of Gd(OH)<sub>2.14</sub> $F_{0.86}$ : x% Tb<sup>3+</sup> (1 ≤x ≤8).



Fig.S5 CL spectra from (a)  $Gd(OH)_{2.14}F_{0.86}$ :  $Eu^{3+}$  (20%) and (b)  $Gd(OH)_{2.14}F_{0.86}$ :  $Tb^{3+}$  (5%) measured at 10 kV and various filament currents.