

Electronic Supporting Information (ESI)

for

**Coordination-induced and tunable layered rare-earth
hydroxides-complex intercalated nanohybrids
phosphorescent photosensitizer and therapy**

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1. Figures and tables

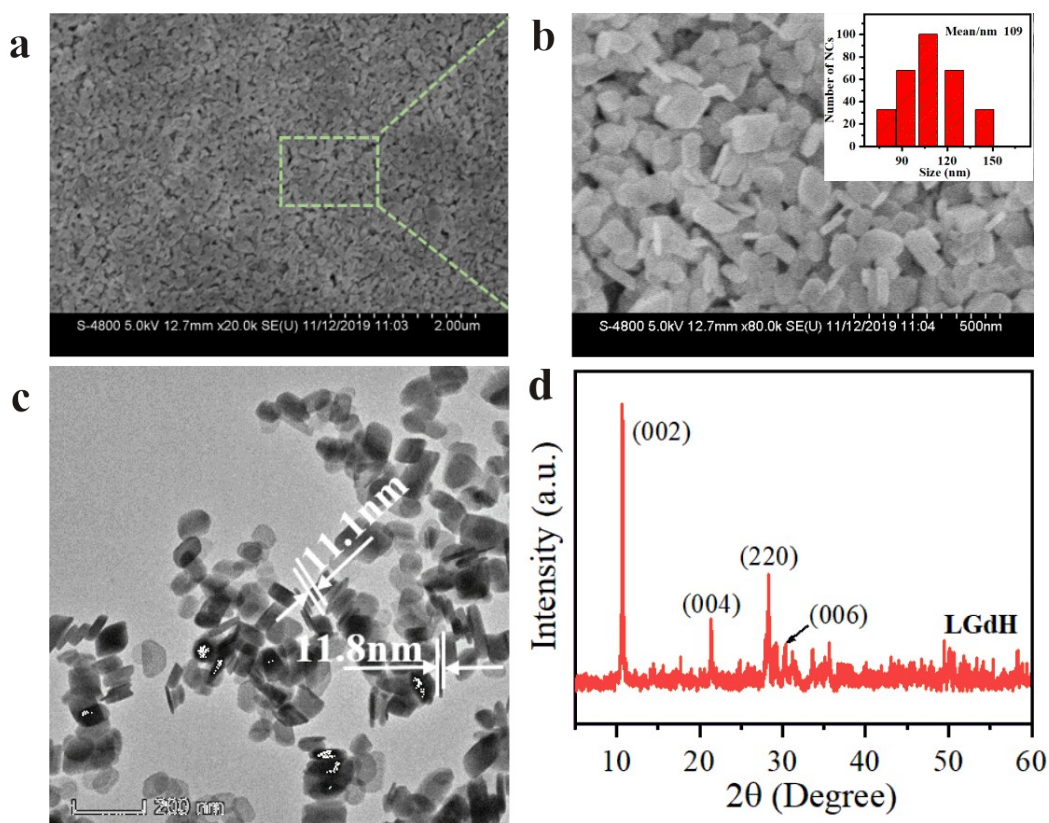


Fig. S1 (a) and (b) SEM images, (c) TEM images, (d) PXRD patterns ($2\theta = 5-60^\circ$) of LGdH.

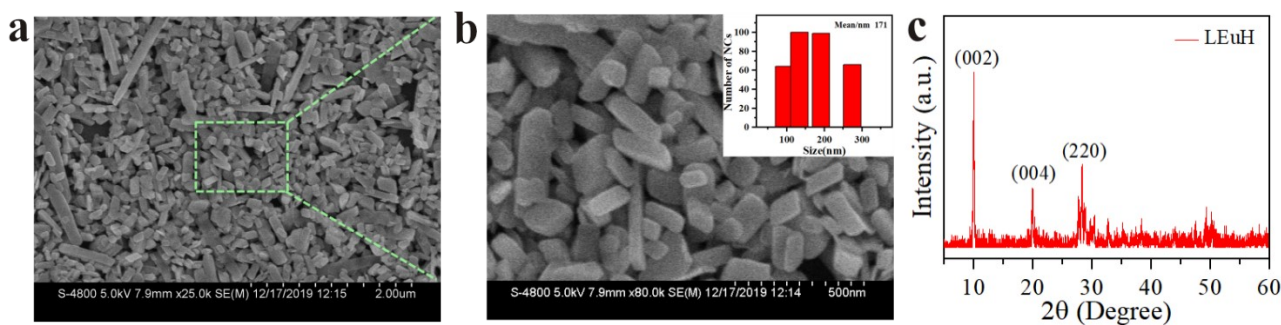


Fig. S2 (a) and (b) SEM images, (c) PXRD patterns ($2\theta = 5-60^\circ$) of LLeuH.

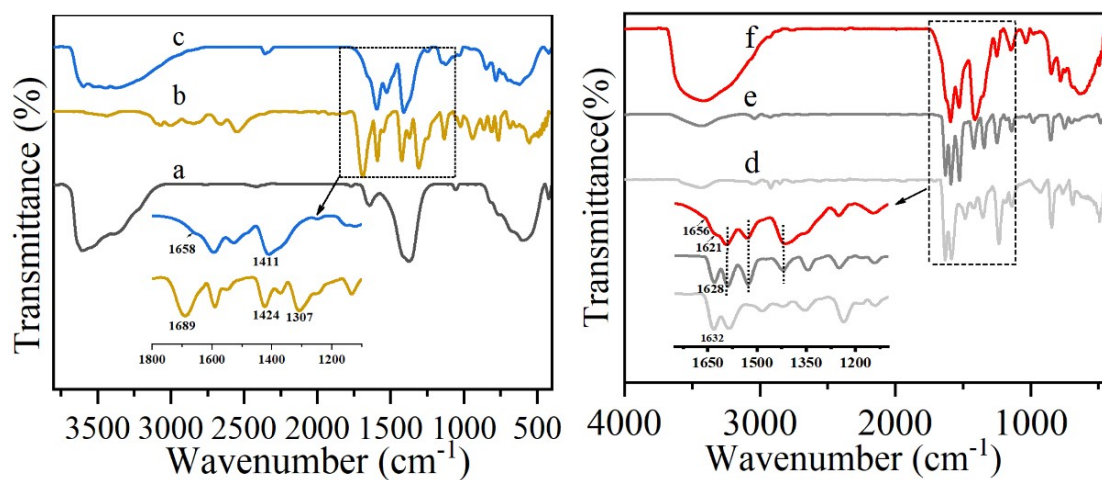


Fig. S3 FTIR spectra of (a) LGdH, (b) 5,5'-BDA, (c) LGdH-5,5'-BDA, (d) HPhN (L), (e) GdL_3 and (f) LGdH-5,5'-BDA- GdL_3

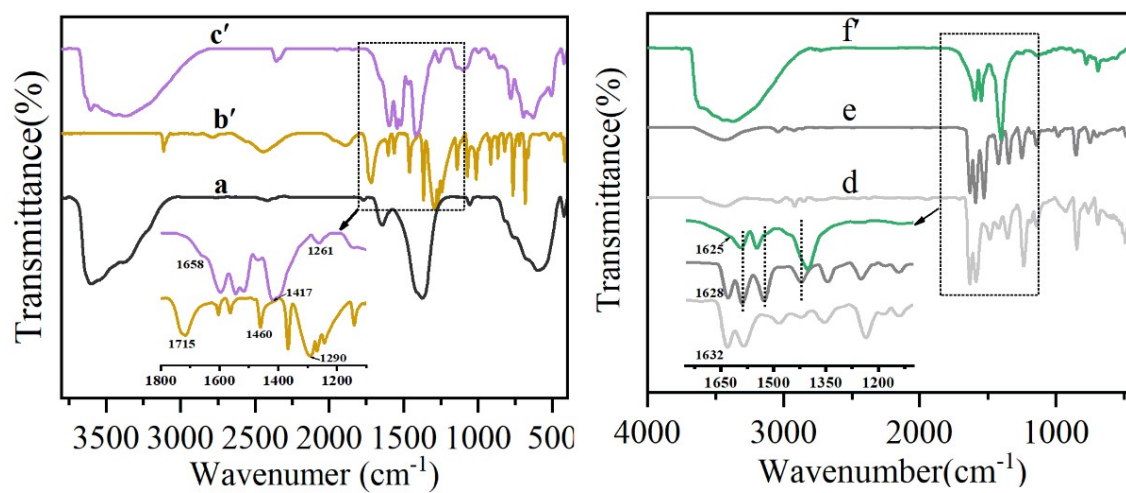


Fig. S4 FTIR spectra of (a) LGdH, (b') 4,4'-BDA, (c') LGdH-4,4'-BDA, (d) HPhN (L), (e) GdL_3 and (f') LGdH-4,4'-BDA- GdL_3

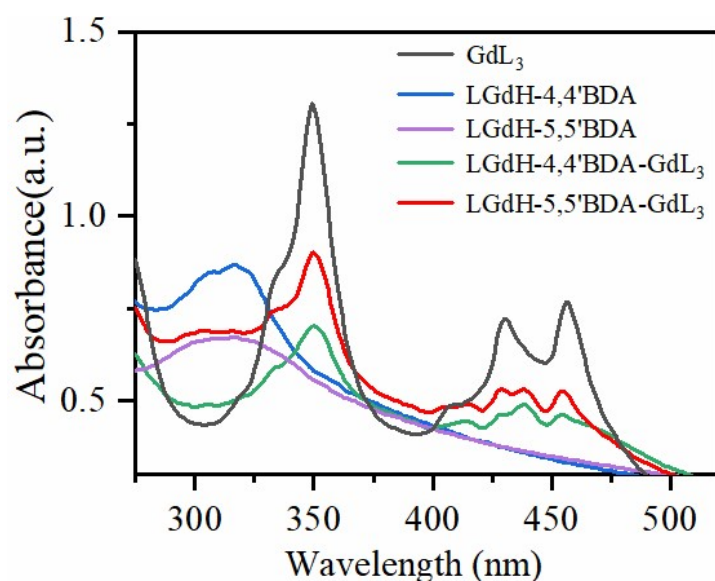


Fig. S5 UV-Vis absorption spectra of GdL₃ (1.0×10^{-5} mol/L), LGdH-4,4'BDA (0.5 mg/mL), LGdH-5,5'BDA (0.5 mg/mL), LGdH-4,4'BDA-GdL₃ (0.5 mg/mL) and LGdH-5,5'BDA-GdL₃ (0.5 mg/mL) samples in ethanol.

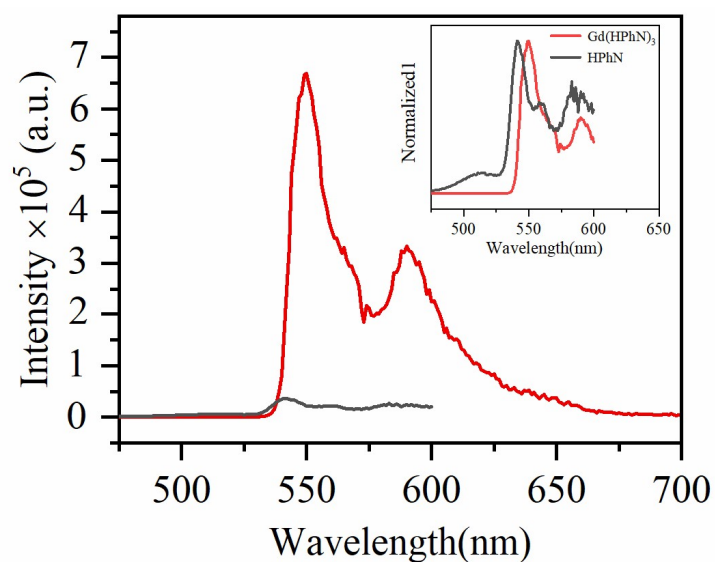


Fig. S6 The low temperature (77K) phosphorescence spectra of HPhN and Gd(HPhN)₃^{S1}

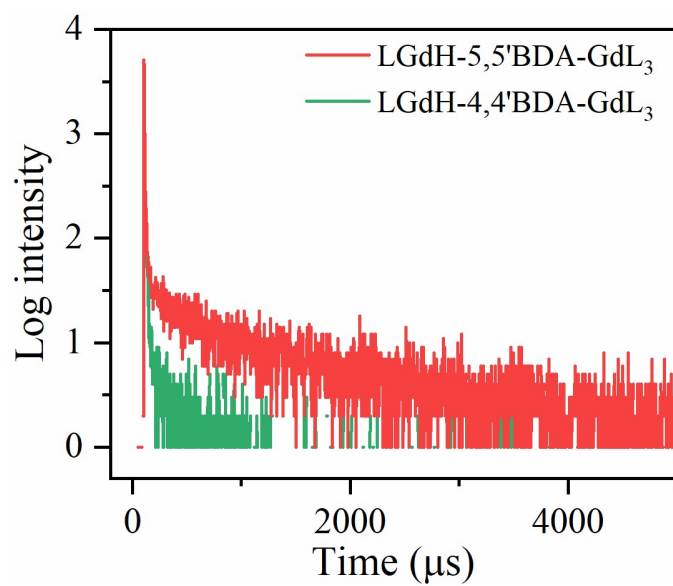


Fig. S7 The luminescence decays of LGdH-5,5'BDA-GdL₃ and LGdH-4,4'BDA-GdL₃ solid samples.

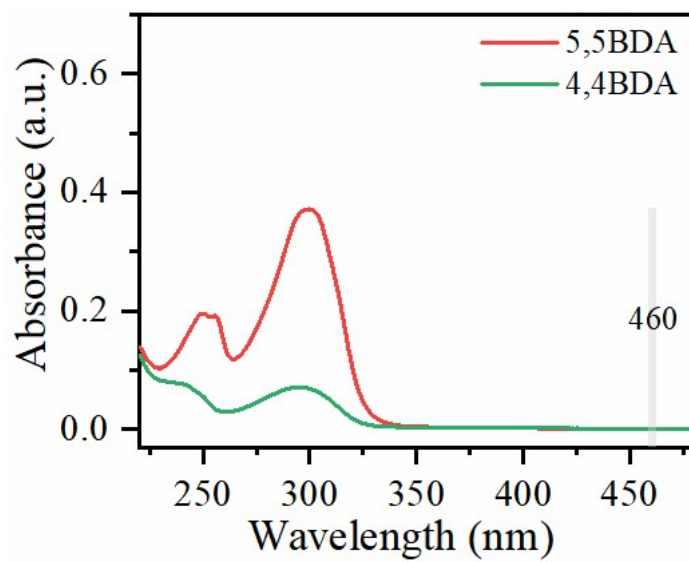


Fig. S8 UV-Vis absorption spectra of BDA

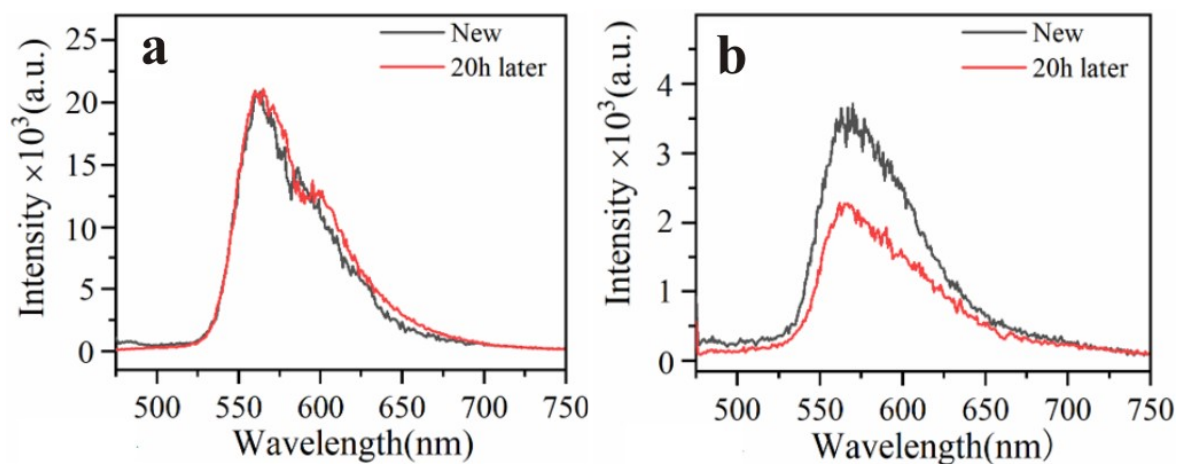


Fig. S9 The phosphorescence spectra of various hybrids in water, (a) LGdH-5,5'BDA-GdL₃; (b) LGdH-4,4'BDA-GdL

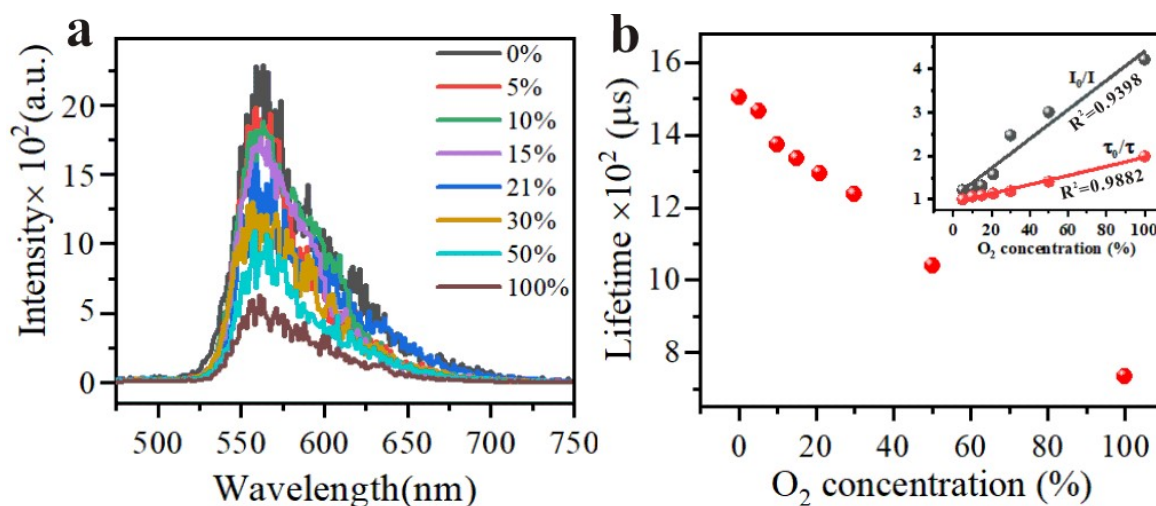


Fig. S10 The phosphorescence performance of LGdH-5,5'BDA-GdL₃ suspension (0.5g/mL, λ_{ex} = 460 nm) at room temperature, (a) luminescence intensity, (b) decay lifetime, inner: the linear relationship between I₀/I and τ₀/τ with O₂ concentration^{S2}.

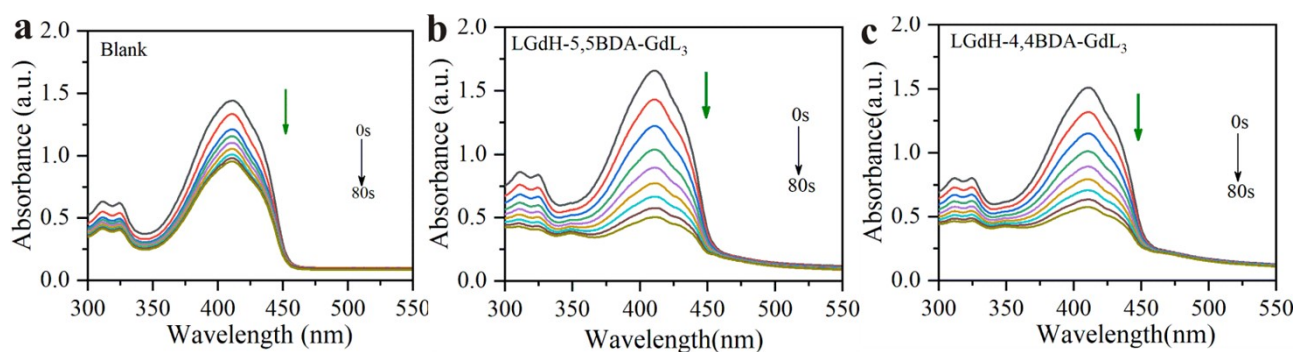


Fig. S11 UV-Vis absorption spectra of DPBF with different irradiation time (460nm LED lamp, irradiation time: 10 s every time), (a) Blank; (b) LGdH-5,5'BDA-GdL₃; (c) LGdH-4,4'BDA-GdL₃;

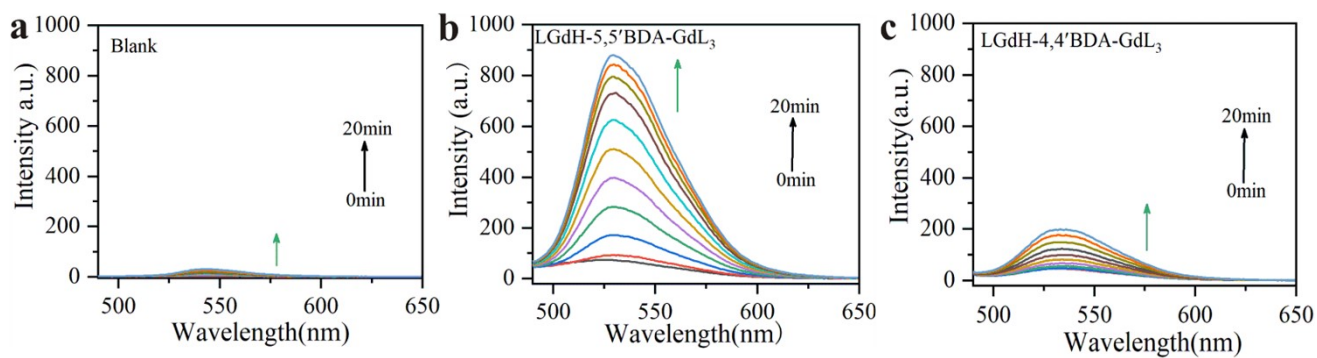


Fig. S12 The fluorescence spectra of DCFH at different irradiation time (460nm LED lamp, irradiation time: 2min every time), (a) Blank; (b) LGdH-5,5'BDA-GdL₃; (c) LGdH-4,4'BDA-GdL₃;

Table S1 The factor and level design of orthogonal test of LGdH

	Concentration of Gd ³⁺ ion (c) / mol·L ⁻¹	Stirring time (t ₁) /min	Hydrothermal temperature (T) / °C	Hydrothermal time (t ₂) / h
1	0.01	5	90	24
2	0.01	15	120	36
3	0.01	25	150	48
4	0.03	5	120	48
5	0.03	15	150	24
6	0.03	25	90	36
7	0.05	5	150	36
8	0.05	15	90	48
9	0.05	25	120	24

Table S2 The level of (002) diffraction peak and calculated basal spacing

	LGdH	LGdH-5,5'BDA	LGdH-4,4'BDA	LGdH-5,5'BDA-GdL ₃	LGdH-4,4'BDA-GdL ₃
2θ /Degree	10.82	5.48	5.96	5.60	5.94
Basal spacing/nm	0.83	1.64	1.50	1.58	1.50

Table S3 N₂ adsorption–desorption isotherm studies of solid hybrids

	LGdH-5,5'BDA	LGdH-4,4'BDA	LGdH-5,5'BDA-GdL ₃	LGdH-4,4'BDA-GdL ₃
BET Surface Area (m ² ·g ⁻¹)	26.2	19.0	10.1	23.0

2. Notes and references

(S1) Z. L. Zhao, J. X. Ru, P. P. Zhou, Y. S. Wang, C. F. Shan, X. X. Yang, J. Cao, W. S. Liu, H. C. Guo and Y. Tang, *Dalton Trans.*, 2019, **48**, 16952-16960.

(S2) M. Fernandes, Z. Bermudez, V. S. Ferreira, R. A. Carlos, L. D. Charas, A. Morgado, J. Silva and M. M. Smith, *Chem. Mater.*, 2007, **19**, 3892-3901.