

## SUPPORTING INFORMATION

### Synthesis of monoclinic Ho, Tm:KLu(WO<sub>4</sub>)<sub>2</sub> microrods with high photothermal conversion efficiency via a thermal decomposition-assisted method

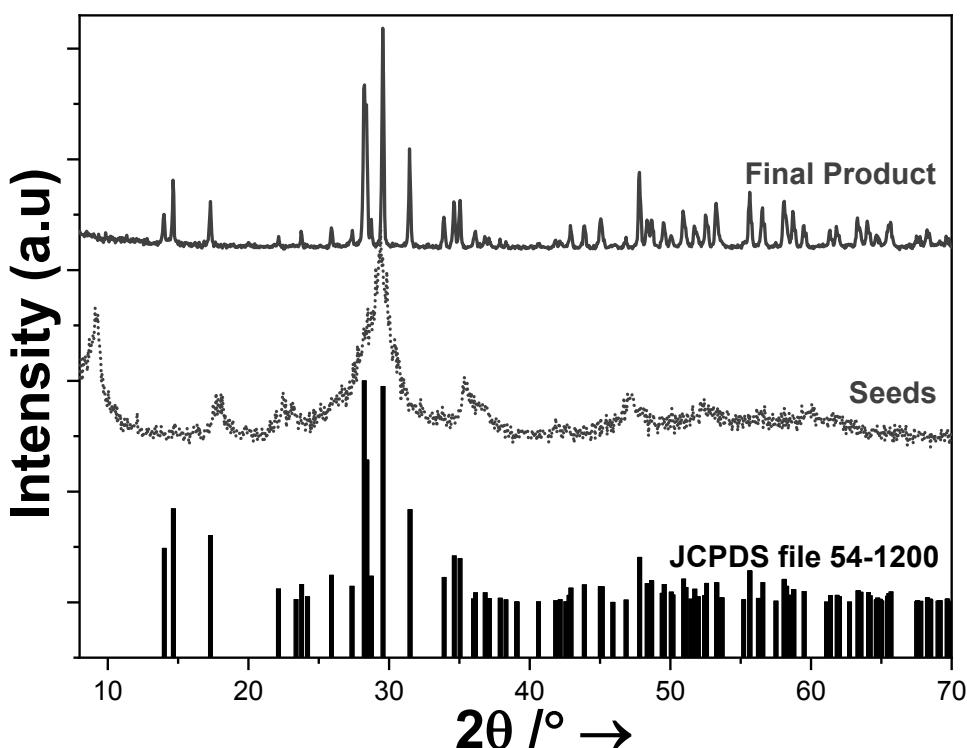
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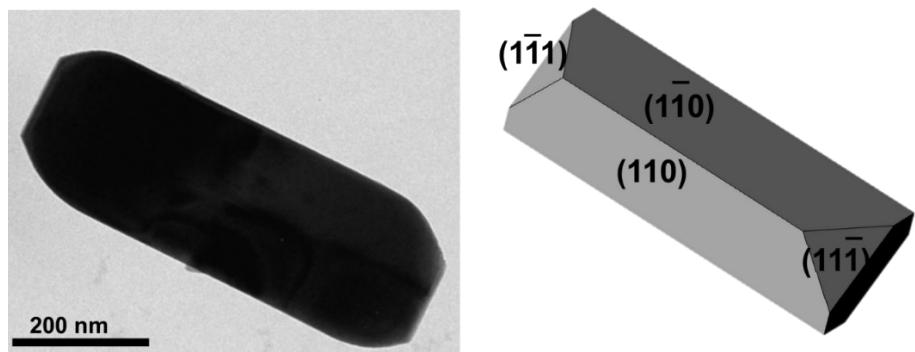
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**Table S1.** ICP-OES analysis of the dopant concentration of Ho<sup>3+</sup>,Tm<sup>3+</sup> in KLu(WO<sub>4</sub>)<sub>2</sub> rods synthesized by the thermal decomposition-assisted methodology.

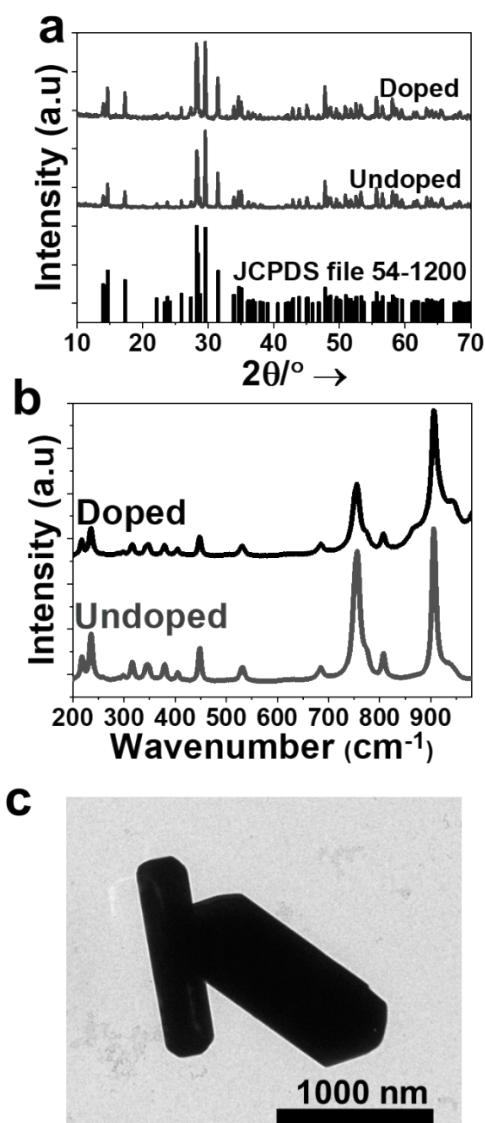
Material $KL_{1-x-y}Ho_xTm_y(WO_4)_2$	Ho <sup>3+</sup> (w/w %) x (%)	Tm <sup>3+</sup> (w/w %) y (%)	Lu <sup>3+</sup> (w/w %) 1-x-y (%)
x=0.03, y=0.05	2.2	3.57	94.22
x=0.01, y=0.1	0.98	9.8	89.22



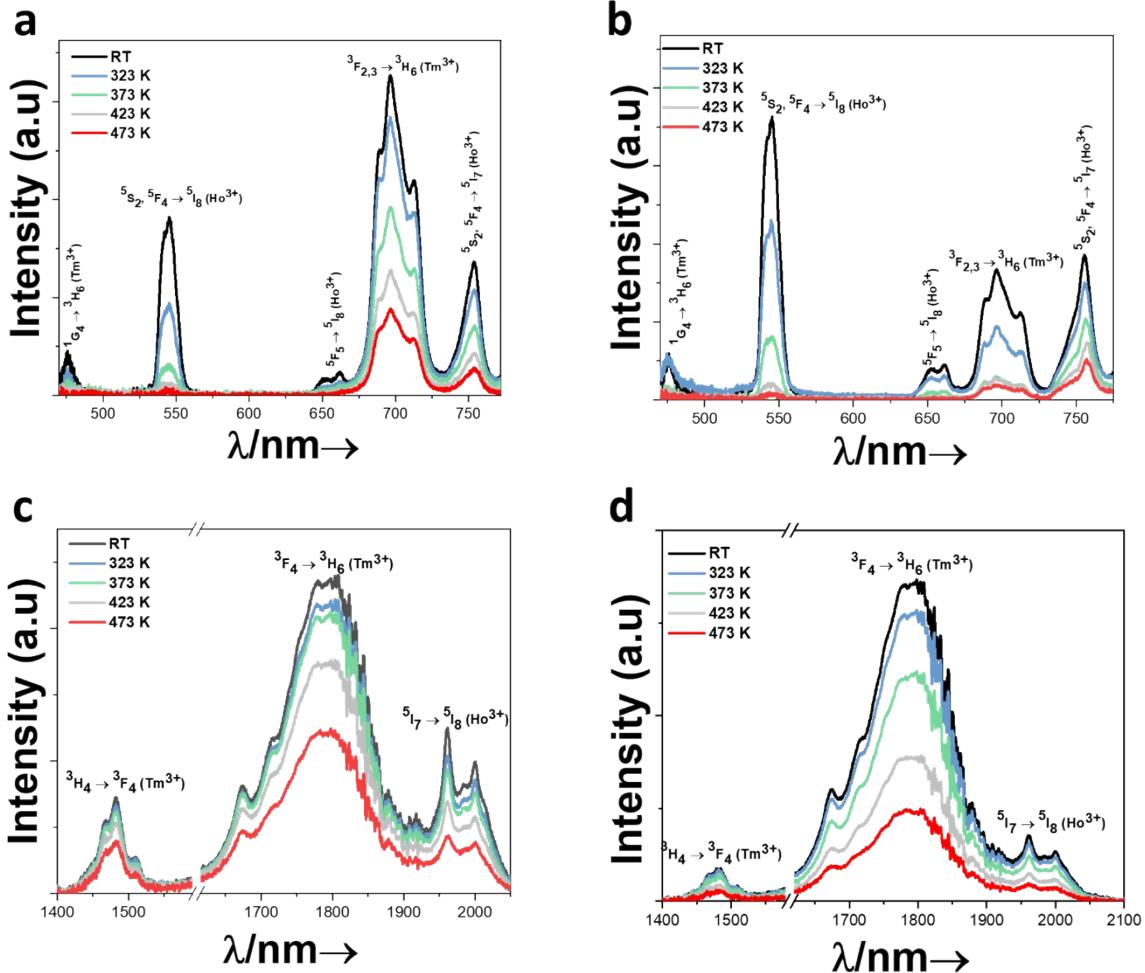
**Fig. S1** XRPD patterns of the seeds and the final products (calcined at 1023 K for 2 h as an example) obtained in the synthesis of undoped KL<sub>W</sub> particles via the thermal decomposition-assisted method. The KL<sub>W</sub> reference pattern (JCPDS file 54-1200) was included for comparison.



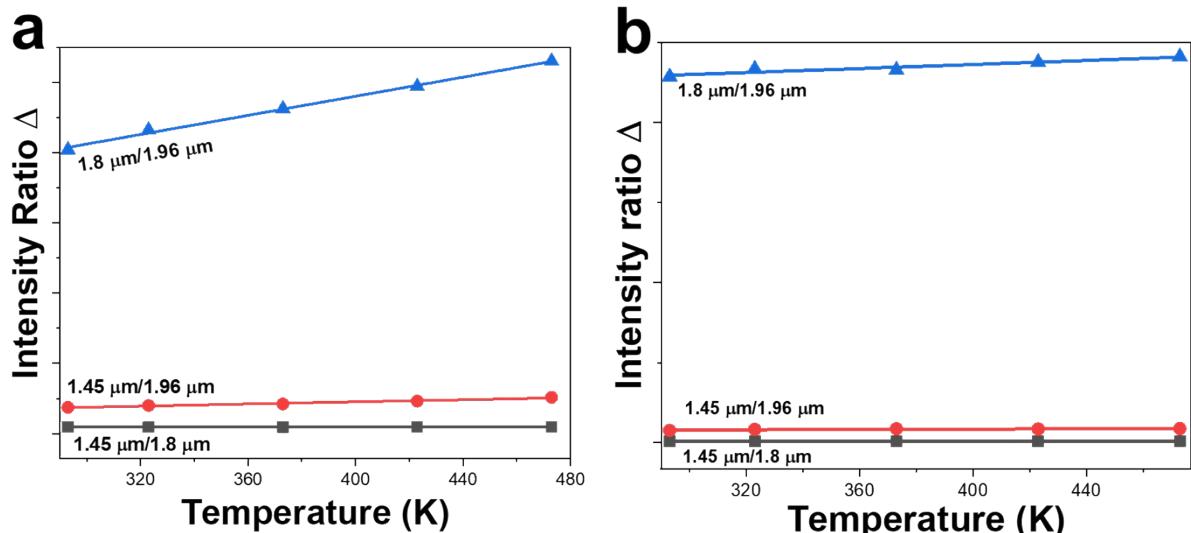
**Fig. S2** Crystalline habit of the undoped KLuW rods, transmission electron microscopic image and morphology predicted by the SHAPE software.



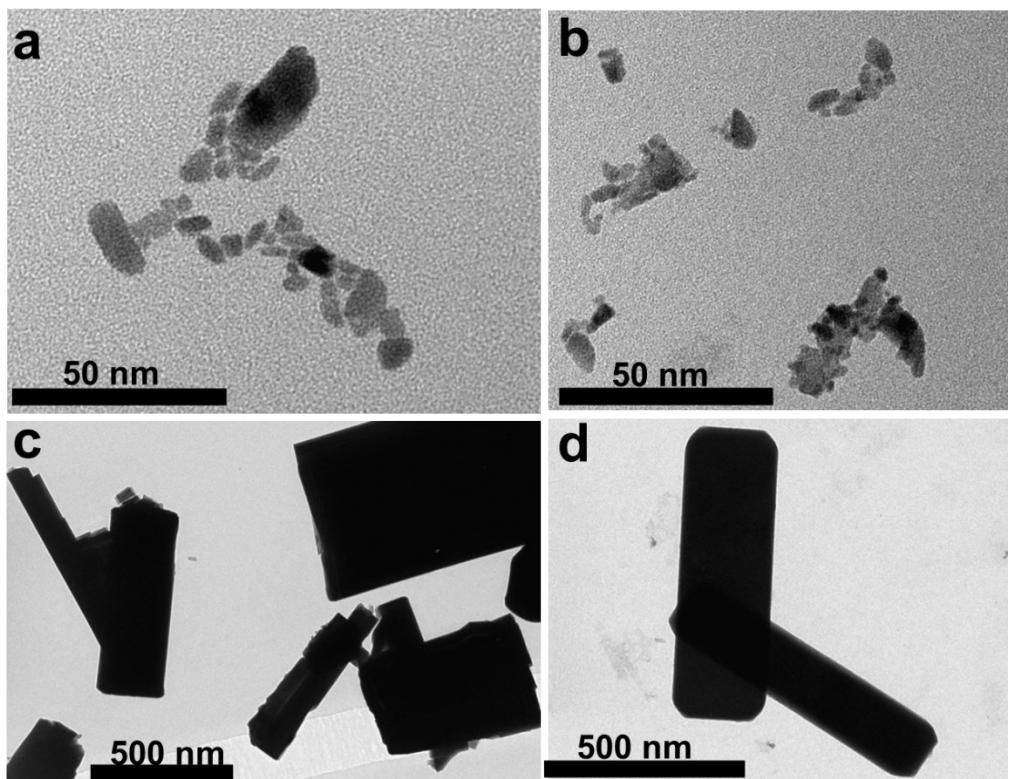
**Fig. S3** (a) XRPD pattern of undoped KLuW and doped Ho, Tm:KLuW microrods. (b) Raman spectroscopy of undoped KLuW and doped Ho, Tm:KLuW microrods. (c) TEM image of doped Ho, Tm:KLuW microrods.



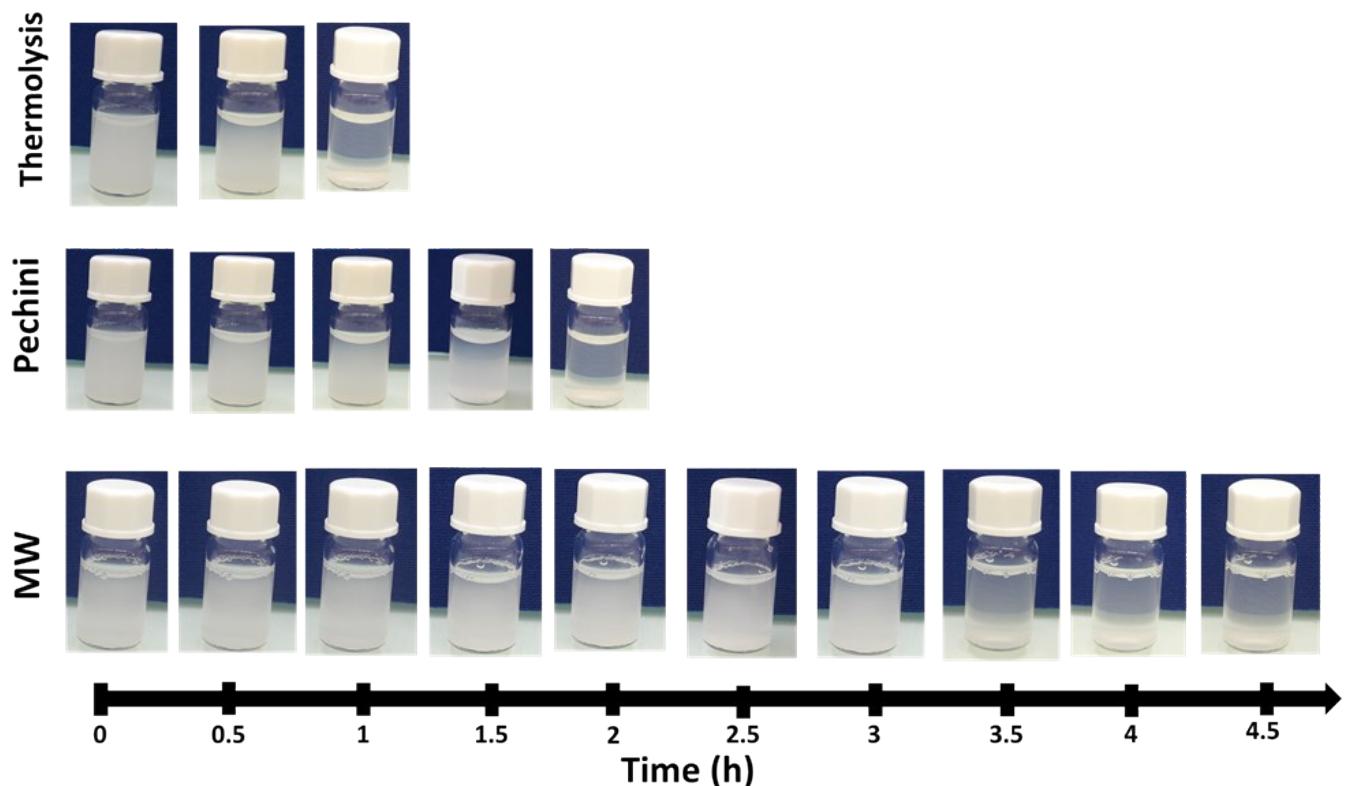
**Fig. S4** Evolution with temperature of the intensity of the emission bands in the visible of Ho, Tm doped KLuW microrods containing: (a) 3 mol% Ho<sup>3+</sup>, 5 mol% Tm<sup>3+</sup>, and (b) 1 mol% Ho<sup>3+</sup>, 10 mol% Tm<sup>3+</sup>. Evolution with temperature of the intensity of the emission bands in the NIR of Ho, Tm:KLuW rods containing: (c) 3 mol% Ho<sup>3+</sup>, 5 mol% Tm<sup>3+</sup> and (d) 1 mol% Ho<sup>3+</sup>, 10 mol% Tm<sup>3+</sup>.



**Fig. S5** Temperature dependence of the three possible intensity ratios in the NIR of Ho, Tm doped KLuW microrods containing: (a) 3 mol% Ho<sup>3+</sup>, 5 mol% Tm<sup>3+</sup> and (b) 1 mol% Ho<sup>3+</sup>, 10 mol% Tm<sup>3+</sup>.



**Fig. S6** TEM images of Ho, Tm doped KLuW particles synthesized via four different synthetic methodologies: (a) MW=microwave-assisted solvothermal method, (b) CA=conventional autoclave solvothermal method, (c) P=modified sol-gel Pechini method, and (d) TD=thermal decomposition-assisted method.



**Fig. S7** Sedimentation test of Ho, Tm doped KLu(WO<sub>4</sub>)<sub>2</sub> nanocrystals synthesized from the modified sol-gel Pechini, solvothermal (MW as an example) and thermolysis methodologies.

**Table S2** Fitting parameters and thermometric performance of Ho, Tm doped KLuW microrods synthesized via the thermal decomposition-assisted method, operating in different spectral regimes and on the temperature range 293 K-473 K.

**Based on Equation 1**

Doping	Regime	$\Delta_0$	B	$\alpha$	R <sup>2</sup>	$S_{abs}$ (K <sup>-1</sup> )	$S_{rel}$ (% K <sup>-1</sup> )	$\delta T$ (K)
3 mol% Ho, 5 mol% Tm	VIS	3.95	0.0066	0.011	0.98	0.013	0.25	1.97
1 mol% Ho, 10 mol% Tm	VIS	0.55	10312	0.032	0.94	0.028	1.9	0.26

**Based on Equation 5**

Doping	Regime	B	C	$\Delta E_1 - \Delta E_2$ (cm <sup>-1</sup> )	R <sup>2</sup>	$S_{abs}$ (K <sup>-1</sup> )	$S_{rel}$ (% K <sup>-1</sup> )	$\delta T$ (K)
3 mol% Ho, 5 mol% Tm	NIR	8.07	203.9	141.7	0.99	0.0096	0.24	2.1
1 mol% Ho, 10 mol% Tm	NIR	10.38	36.6	25.4	0.90	0.0039	0.04	11.7

**Table S3** Fitting parameters, thermometric performance, and photothermal conversion efficiency of Ho, Tm doped KLuW particles synthesized via four different synthetic methodologies (P = modified sol-gel Pechini method; MW = microwave-assisted solvothermal method; CA = conventional autoclave solvothermal method; and TD = thermal decomposition-assisted method).

Doping	Synthesis	Size (nm)	B	C	$\Delta E_1 - \Delta E_2$ (cm <sup>-1</sup> )	R <sup>2</sup>	$S_{abs}$ (K <sup>-1</sup> )	$S_{rel}$ (% K <sup>-1</sup> )	$\delta T$ (K)	$\eta$ (%)	Ref.
1 mol% Ho, 10 mol% Tm	P	2000	155.6 ± 15.1	773.2 ± 30.7	537	0.99	0.097	0.90	0.55	40	1
1 mol% Ho, 10 mol% Tm	MW	12	8.5 ± 0.3	159.7 ± 9.9	111	0.98	0.0091	0.18	2.6	45	2
1 mol% Ho, 10 mol% Tm	CA	16	5.1 ± 0.7	172.7 ± 44.8	120	0.99	0.0056	0.20	2.4	43	2
<b>1 mol% Ho, 10 mol% Tm</b>	<b>TD</b>	<b>1480</b>	<b>12.1 ± 0.66</b>	<b>172.7 ± 17</b>	<b>60</b>	<b>0.92</b>	<b>0.009</b>	<b>0.1</b>	<b>5</b>	<b>41</b>	<b>This Work</b>
3 mol% Ho, 5 mol% Tm	P	2000	13.2 ± 1.2	318.2 ± 29.3	221	0.95	0.016	0.37	1.34	30	1
3 mol% Ho, 5 mol% Tm	MW	12	3.7 ± 0.1	198.9 ± 10.8	138	0.98	0.0044	0.23	2.1	33	2
3 mol% Ho, 5 mol% Tm	CA	16	4 ± 0.1	284.9 ± 10.1	198	0.99	0.0051	0.33	1.5	36	2
<b>3 mol% Ho, 5 mol% Tm</b>	<b>TD</b>	<b>1480</b>	<b>12.5 ± 0.2</b>	<b>354.6 ± 5.1</b>	<b>247</b>	<b>0.99</b>	<b>0.015</b>	<b>0.41</b>	<b>1.2</b>	<b>66</b>	<b>This Work</b>

**References**

1. A. Nexha, J. J. Carvajal, M. C. Pujol, F. Díaz and M. Aguiló, *J. Mater. Chem. C*, 2020, **8**, 180-191.
2. A. Nexha, M. C. Pujol, J. J. Carvajal, F. Díaz and M. Aguiló, *Submitted at Nanomaterials*