

Electronic Supplementary Information (ESI)

A unique delaminated MoS₄/OS-LEuH composite exhibiting turn-on luminescence sensing for detection of water in formamide

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Experimental details.

Preparation of NO₃-LEuH precursor by homogeneous method. The NO₃-LEuH precursor was prepared *via* a homogeneous precipitation method under a hydrothermal condition. Firstly, 0.446 g Eu(NO₃)₃·6H₂O (1 mmol), 1.105 g NaNO₃ (13 mmol), and 0.140 g hexamethylenetetramine (HMT, 1 mmol) were dissolved in deionized water (80 mL). Then, the aqueous solution at 90 °C for 12 h was heated in a Teflon-autoclave (100 mL). Finally, the obtained product was filtered, washed with deionized water and ethanol, and then vacuum-dried at 40 °C for 6 h.

Co-intercalation of MoS₄²⁻ and 1-octane sulfonate (OS⁻) into LEuH by an ion-

exchange method. The intercalation reactions of MoS_4^{2-} and/or OS^- into LEuH were achieved by ion-exchange method using the NO_3 -LDH as a precursor, and two composites of OS-LEuH and MoS_4/OS -LEuH were obtained eventually.

Synthesis of OS-LEuH composite. A suspension containing the 0.100 g NO_3 -LEuH and 0.649 g of OS (3 mmol) in deionized water of 80 mL was heated at 70 °C for 24 h in a Teflon-autoclave (100 mL). Then, the obtained white-color product was filtered, washed with deionized water and ethanol, and vacuum-dried (40 °C for 6 h), to obtain the OS-LEuH composite.

Synthesis of MoS_4/OS -LEuH composite. The mixture of NO_3 -LEuH (0.100 g), OS (0.610 g, 2.82 mmol), and $(\text{NH}_4)_2\text{MoS}_4$ (0.047 g, 0.18 mmol) (the molar ratio of $\text{OS}^-/\text{MoS}_4^{2-}$ was 94:6) was dispersed in deionized water of 80 mL, then the formed suspension was heated at 70 °C for 24 h in a Teflon-autoclave (100 mL). Similarly, the light-brown precipitate was filtered, washed with deionized water and ethanol, then vacuum-dried (40 °C for 6 h), thus the MoS_4/OS -LEuH composite was prepared.

Delamination of OS-LEuH and MoS_4/OS -LEuH.

Pure formamide (FM) system. 0.02 g of the composite powders were dispersed respectively into 10 mL FM, followed by mechanical shaking for 16 h, to obtain their colloidal suspensions.

FM-water system. For exploring the effect of water on luminescence behaviors, firstly, the colloidal suspensions were prepared as the method (1), and took out 1 mL of the aforementioned suspensions to mix with 3 mL deionized water.

For further study, a series of colloidal suspensions were prepared by dispersing 0.02 g of the composite into 4 mL, 6 mL, 8 mL, 10 mL, 12 mL, 14 mL, 16 mL, 18 mL and 19 mL FM with mechanical shaking for 16 h, respectively, and then mixed with 16 mL, 14 mL, 12 mL, 10 mL, 8 mL, 6 mL, 4 mL, 2 mL, and 1 mL of deionized water. The total volume of the FM-water mixture was fixed to be 20 mL.

DMF-water system. A series of colloidal suspensions were prepared by dispersing 0.02 g of the composite into 4 mL, 8 mL, 14 mL, 18 mL, 19 mL, and 20 mL N,N-dimethylformamide (DMF) with mechanical shaking for 16 h, respectively,

and then mixed with 16 mL, 12 mL, 6 mL, 2 mL, 1 mL, and 0 mL of deionized water. The total volume of the DFM-water mixture was fixed to be 20 mL.

Characterization. The powder X-ray diffraction (XRD) patterns of the NO₃-LEuH precursor and the as-prepared composites were recorded by using a Phillips X'pert Pro MPD diffractometer with Cu-K radiation at room temperature. For the large degrees, it was operated under a step size of 0.0167°, scan time of 15 seconds per step, and 2θ ranging from 4.5 to 70°. The generator setting was 40 kV and 40 mA. For the small degrees, the XRD patterns were collected with a step size of 0.008°, scan time of 30 seconds per step, and 2θ ranging from 0.6 to 6°. Fourier transformed infrared (FT-IR) spectra of the samples were recorded on a Nicolet-360 Fourier-Transform infrared spectrometer using the KBr method. Scanning electron microscope (SEM) observations were conducted via a Hitachi S-4800 microscope. The metal ion concentrations were measured by ICP-AES (Jarrel-ASH, ICAP-9000) after the solid products were dissolved in a ~0.1 M HNO₃ solution. CHN analysis was carried out by using an Elementar vario EL elemental analyzer. The photoluminescence spectra were measured at room temperature with a FS5 fluorescence spectroscopy. The UV-vis adsorption spectra were obtained at room temperature on a UV-2450 spectrophotometer.

Table S1. Chemical compositions for the NO₃-LEuH precursor and the MoS₄/OS-LEuH composites.

Samples	Chemical formula	wt%, found (calcd)				
		Eu	Mo	C	H	N
NO ₃ -LEuH	Eu(OH) _{2.41} (NO ₃) _{0.49} (CO ₃) _{0.05} ·0.8H ₂ O	62.10 (62.50)	-	0.28 (0.25)	1.61 (1.73)	2.71 (2.83)
OS-LEuH	Eu(OH) _{2.41} (C ₈ H ₁₇ O ₃ S) _{0.64} NO ₃) _{0.01} ·0.8H ₂ O	44.72 (45.88)	-	18.05 (18.52)	4.13 (4.49)	0.04 (0.04)
MoS ₄ /OS-LEuH	Eu(OH) _{2.41} (OS) _{0.48} (MoS ₄) _{0.05} (NO ₃) _{0.02} ·1.8H ₂ O	44.25 (45.84)	1.54 (1.60)	13.74 (13.90)	3.91 (4.27)	0.08 (0.08)

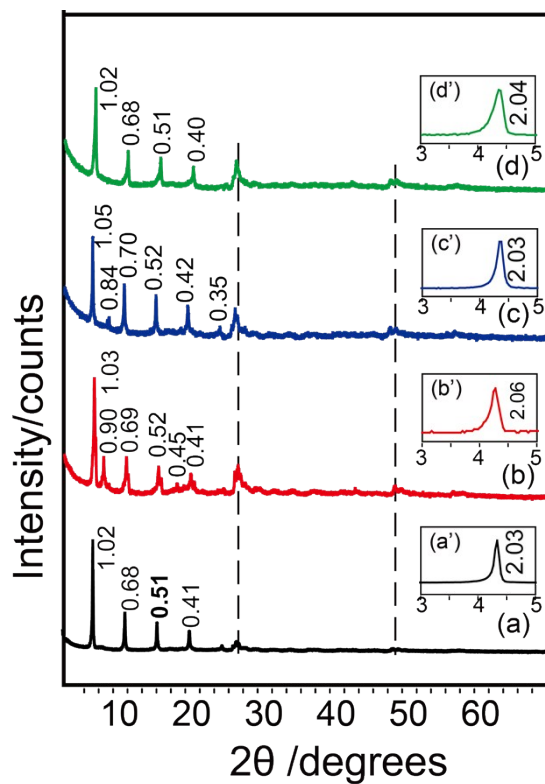


Fig. S1 XRD patterns of composites with OS⁻ /MoS₄²⁻ molar ratios of (a) 0.98:0.02, (b) 0.94:0.06, (c) 0.90:0.10, and (d) 0.80:0.20. The *d*-values in XRD patterns were given in nanometers.

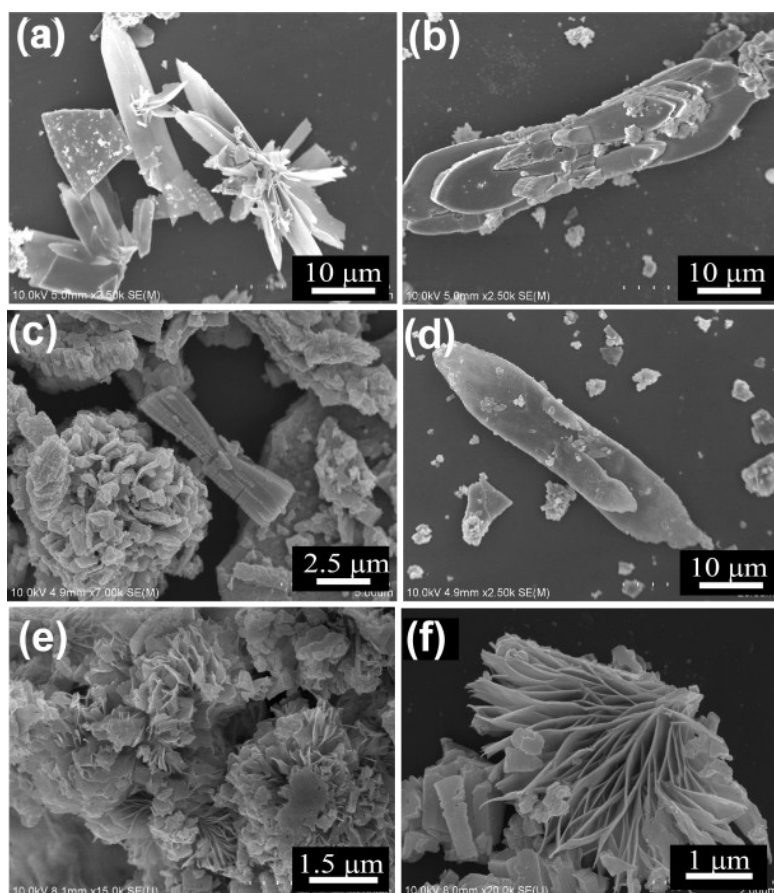


Fig. S2 SEM images of $\text{NO}_3\text{-LEuH}$ precursor (a, b), and the composites of OS-LEuH (c, d), and $\text{MoS}_4/\text{OS-LEuH}$ (e, f), respectively.

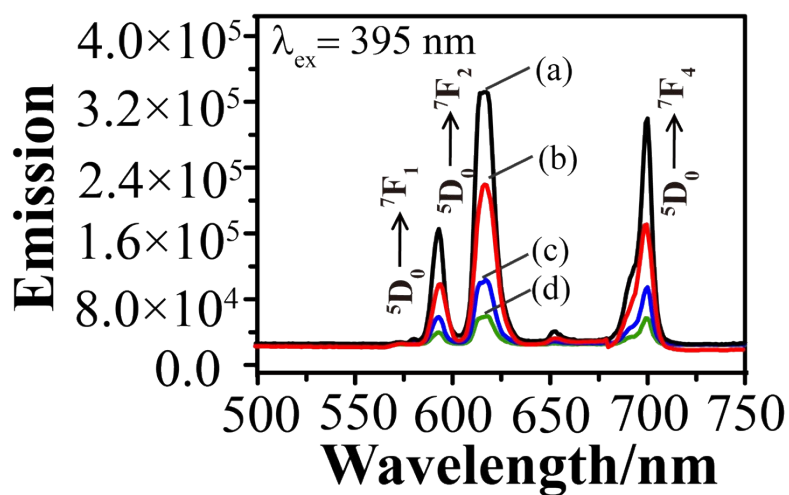


Fig. S3 Photoluminescence emission spectra of colloidal suspensions of composites with $\text{OS}^-/\text{MoS}_4^{2-}$ molar ratios of (a) 0.98:0.02, (b) 0.94:0.06, (c) 0.90:0.10, and (d) 0.80:0.20 ($\lambda_{\text{ex}} = 395 \text{ nm}$) in pure formamide.

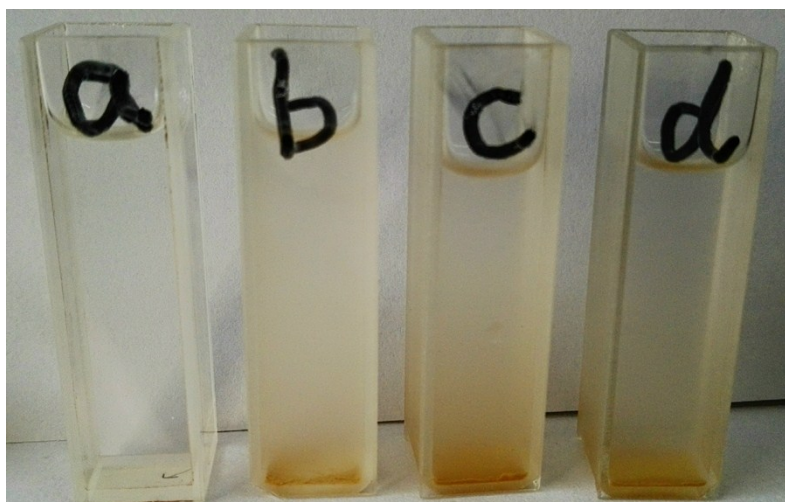


Fig. S4 Photographs of the formed suspensions after dispersing 0.02 g MoS₄/OS-LEuH powder into 20 mL of (a) DMF, (b) ethanol, (c) acetonitrile, and (d) acetone, respectively, for 16 h.

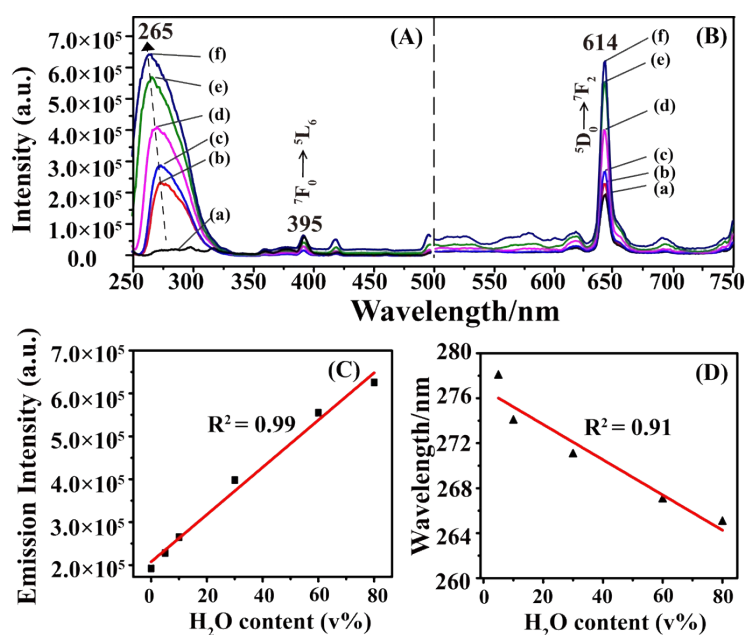


Fig. S5 Photoluminescence (A) excitation and emission (B) spectra of colloidal suspensions of MoS₄/OS-LEuH in DMF+H₂O (x is volume percentages of water): (a) $x = 0$ ($\lambda_{\text{ex}} = 395$ nm), (b) $x = 5\%$ ($\lambda_{\text{ex}} = 278$ nm), (c) 10% ($\lambda_{\text{ex}} = 274$ nm), (d) 30% ($\lambda_{\text{ex}} = 271$ nm), (e) 60% ($\lambda_{\text{ex}} = 267$ nm), (f) 80% ($\lambda_{\text{ex}} = 265$ nm). Plots show the functions of emission intensity (C) and maximum excitation wavelength (D) following the water content (v %).

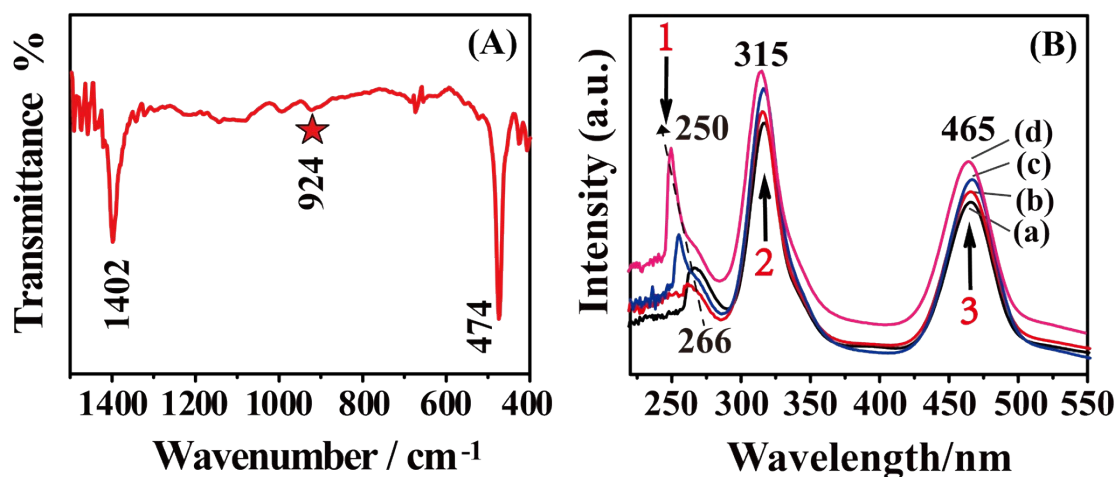


Fig. S6 (A) Liquid IR spectra of MoS_4^{2-} in FM/ H_2O (5 mg $(\text{NH}_4)_2\text{MoS}_4$ was dissolved in 25 mL FM and 75 mL H_2O). (B) UV-Vis absorption spectra of MoS_4^{2-} : (a) in pure FM ($\lambda_{1,a} = 266$ nm), and in FM/ H_2O system with H_2O v% of (b) 20% ($\lambda_{1,b} = 261$ nm, 5 mg $(\text{NH}_4)_2\text{MoS}_4$ was dissolved in 80mL FM and 20 mL H_2O), (c) 60% ($\lambda_{1,c} = 254$ nm, 5 mg $(\text{NH}_4)_2\text{MoS}_4$ in 40mL FM and 60 mL H_2O), and (d) 80% ($\lambda_{1,d} = 250$ nm, 5 mg $(\text{NH}_4)_2\text{MoS}_4$ in 20mL FM and 80 mL H_2O).