

Reversible photochromism and tunable luminescence switching in lanthanide-polyoxotungstate-agarose composite films

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ABSTRACT: Stimuli-responsive materials integrating photochromism and luminescence switching are widely utilized in anticounterfeiting, data encryption, optical storage, green printing, and sensing. Here, a series of photoresponsive composite films with tunable multicolor luminescence were fabricated by doping lanthanide-polyoxotungstates into an agarose matrix via a combined sol-gel and casting method. Characterization via X-ray photoelectron spectroscopy, Fourier transform infrared spectroscopy, scanning electron microscopy, transmission electron microscopy, and elemental mapping confirmed the successful fabrication and uniform doping of the films. Spectroscopic analyses revealed that the dopant content effectively tuned the optical transmittance and emission intensity of the films. Crucially, the films exhibited reversible photochromism and synchronous luminescence switching upon alternating ultraviolet irradiation and oxygen exposure. This switching behavior was repeatable for a minimum of 10 cycles with minimal fatigue, underscoring their potential as multifunctional, stimuli-responsive smart optical materials.

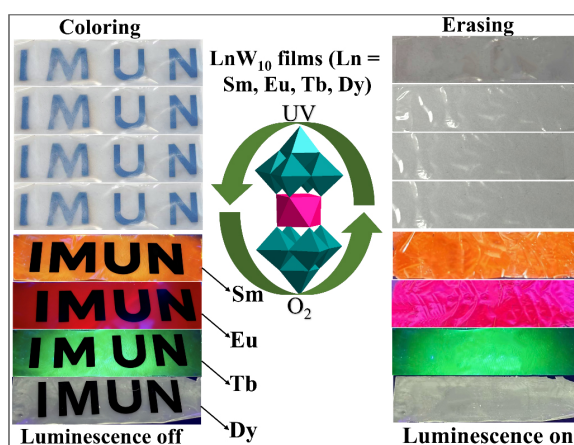
KEYWORDS: polyoxometalate, stimuli-responsive, photochromism, luminescent switch, smart materials

1 Introduction

Stimuli-responsive smart materials integrating photochromism and luminescence switching have attracted significant attention for their vast potential in applications, such as anticounterfeiting [1], data encryption [2], optical information storage [3], green printing [4], bioimaging [5], and sensing [6]. The conventional strategy for constructing these dual-signal readout materials involves integrating luminescent moieties into photochromic molecular skeletons [7, 8]. Considerable progress has been achieved in the field of luminescent photoswitches, mainly driven by the isomerization and ring-opening/closing reactions of a family of organic photochromic units, including diarylethenes [9], hydrazones [10], spirooxazines, and spiropyrans [11]. These systems are characterized by high fluorescence brightness, tunable

emission colors, rapid stimulus-responsive kinetics, and high on/off contrast [12]. Nevertheless, key obstacles to their practical application persist, including poor photostability and thermal stability, low fatigue resistance, and a pronounced environmental dependence of their emission behaviors [13]. These limitations necessitate the urgent need for advanced dual-signal readout materials integrating photochromism and luminescence switching and characterized by cost-effective, facile preparation; high photostability; and excellent switching performance.

Polyoxometalates (POMs), i.e., metal-oxygen anionic clusters assembled from early transition metals (V, Nb, Ta, Mo, and W) coordinated with oxygen atoms, exhibit a range of desirable properties, including diverse compositions and structures, well-defined crystal structures, oxygen-rich surfaces, high photostability/thermal stability, and reversible redox properties [14–16]. Furthermore, they exhibit reversible electrochromism and photochromism under light or electric field stimulation without suffering structural alteration [17, 18]. The colored species (heteropoly blue or brown) exhibit broad absorption in the visible region, making them promising energy acceptors in luminescence-switching materials [19, 20]. Lanthanide-containing POMs (Ln-POMs), synthesized by coordinating lanthanide ions with POM



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frameworks, combine the reversible photochromism of POMs with the exceptional luminescence of the ions (e.g., high color purity, large Stokes shifts, long lifetimes, and environmental insensitivity). These make them promising candidates for bifunctional photochromic and luminescence-switching materials [21–24].

To date, only a few studies have explored synchronous photochromism and luminescence switching in Ln-POM composite thin films. For instance, Yao et al. [25] first constructed an agarose-europium-substituted silicotungstomolybdate ($\text{EuSiW}_{10}\text{Mo}$) composite film via a sol-gel technique, leveraging intermolecular hydrogen bonding between the agarose matrix and the $\text{EuSiW}_{10}\text{Mo}$ clusters. The film exhibited reversible photochromism and luminescence switching upon alternating ultraviolet (UV) and oxygen (UV/O_2) stimulation, with the luminescence-quenching mechanism attributed to intramolecular luminescence resonance energy transfer. Similarly, Gao et al. [26] reported the reversible photochromism and luminescence-switching behavior of an agarose-europium decatungstate (EuW_{10}) composite film upon high-energy UV photoreduction and humid-air oxidation. The UV-reduced film exhibited a relative humidity (RH)-dependent luminescence recovery, demonstrating its potential for applications in UV and moisture dual-responsive materials. Li et al. [27] fabricated a photoresponsive EuW_{10} -gelatin film via solution casting. The reversible photochromism of the film coupled with luminescence switching upon UV/O_2 exposure positioned it as a promising candidate for rewritable inkless security printing media. Multiple encryption strategies have been developed to strengthen information security. Overall, current studies on synchronous photochromism and luminescence switching in rare earth POM films have mainly explored the red emission of Eu systems, with analogous studies on Sm (orange), Tb (green), and Dy (white) remaining unexplored. Thus, exploring photochromic luminescence-switching materials based on Ln-POM with diverse emission colors is essential for cross-field applications.

Here, a series of distinct lanthanide-polyoxotungstate (LnW_{10})-agarose ($\text{Ln} = \text{Sm}^{3+}$, Eu^{3+} , Tb^{3+} , Dy^{3+}) composite films with diverse luminescent properties were fabricated via a combined sol-gel and casting method, leveraging hydrogen bonding interactions between the stimuli-responsive LnW_{10} clusters and the agarose matrix. All four films exhibited reversible photochromism coupled with luminescence switching upon alternating UV/O_2 stimulation, demonstrating several advantages that make them promising candidates for developing smart materials. First, the preparation process is facile and cost-effective, with readily tunable thickness and dimensions. Second, films incorporating different lanthanide ions displayed distinct luminescent colors: orange for Sm^{3+} , red for Eu^{3+} , green for Tb^{3+} , and white for Dy^{3+} , rendering them suitable for cross-field applications. Third, the reversible redox properties of LnW_{10} imparted the films with excellent photochromism and luminescence-switching reversibilities upon UV/O_2 stimulation. Fourth, the luminescence on/off contrasts (E) of the four film types were systematically compared, reaching a peak value of 97.3%. These results suggest that Ln-POM-based films, owing to their notable advantages, are highly promising candidates for stimuli-responsive smart materials.

2 Experimental

2.1 Materials and measurements

As all reagents were of analytical grade, they were used as received.

Sodium tungstate dihydrate ($\text{Na}_2\text{WO}_4 \cdot 2\text{H}_2\text{O}$), europium(III) nitrate hexahydrate ($\text{Eu}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$), glacial acetic acid, and agarose were purchased from Sinopharm Chemical Reagent Co., Ltd. Fourier transform infrared (FT-IR) spectra were recorded on a Shimadzu IRAffinity-1S spectrometer in the $4000\text{--}400\text{ cm}^{-1}$ range. Powder X-ray diffraction (PXRD) patterns were observed on a Rigaku SmartLab diffractometer with $\text{Cu K}\alpha$ radiation ($\lambda = 1.5418\text{ \AA}$). Scanning electron microscopy (SEM) was performed with a Hitachi S4800 instrument at a 5 kV accelerating voltage. High-resolution transmission electron microscopy (HRTEM) imaging was performed using a Hitachi H-7650 microscope at a 200 kV accelerating voltage. X-ray photoelectron spectroscopy (XPS) measurements were performed using a Shimadzu Kratos AXIS SUPRA+ spectrometer; the binding energy scale was calibrated using the C 1s peak (C–C was set to 284.8 eV). UV-visible (UV-Vis) absorption spectra were recorded using a Shimadzu UV-2600 spectrophotometer, and luminescence spectra were recorded using a Hitachi F-4600 fluorescence spectrometer equipped with an Xe lamp as the excitation source. The excitation wavelengths were set at 255, 278, 315, and 318 nm for the decatungstosamarate(III) (SmW_{10})-agarose, EuW_{10} -agarose, decatungstoterbate(III) (TbW_{10})-agarose, and decatungstodysprosate(III) (DyW_{10})-agarose films, respectively. Time-resolved luminescence decay analysis was performed on an Edinburgh FLS1000 photoluminescence spectrometer. The composite films were irradiated with UV light from a Xe lamp (CEL-HXUV300, Beijing Zhongjiao Jinyuan Technology Co., Ltd.) to induce synchronous photochromism and luminescence quenching. The output power in the UV region ($< 390\text{ nm}$) was approximately 2.6 W, with the emission spectrum of the light source shown in Fig. S1 in the Electronic Supplementary Material (ESM). The bleaching and luminescence-recovery experiments were conducted in a closed container filled with O_2 and maintained at 98% RH. RH levels of 33%, 68%, 86%, and 98% were established using saturated aqueous solutions of magnesium chloride, copper(II) chloride, potassium chloride, and distilled water, respectively.

2.2 Synthesis of lanthanide-polyoxotungstate

Sodium decatungstolanthanate(III) ($\text{Na}_9\text{LnW}_{10}\text{O}_{36}$; $\text{Ln} = \text{Sm}$, Eu , Tb , or Dy) was synthesized following the procedure in Ref. [28]. For sodium decatungstoeuropate(III) ($\text{Na}_9\text{EuW}_{10}\text{O}_{36}$): $\text{Na}_2\text{WO}_4 \cdot 2\text{H}_2\text{O}$ (8.30 g) was dissolved in deionized water (20 mL), and the pH was adjusted to 7.0–7.5 using glacial acetic acid. Thereafter, $\text{Eu}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ (0.92 g) in water (2 mL) was added dropwise to this solution over approximately 10 min. The resulting mixture was stirred and heated at $85\text{ }^\circ\text{C}$. After cooling to room temperature ($\sim 25\text{ }^\circ\text{C}$), the solution was filtered, and subsequent slow evaporation of the filtrate yielded white rod-shaped crystals of $\text{Na}_9\text{EuW}_{10}\text{O}_{36}$. The Sm, Tb, and Dy analogs were prepared similarly using the corresponding rare earth nitrates. The yields (based on Na_2WO_4) were 56.4%, 72.6%, 43.9%, and 56.8% for SmW_{10} , EuW_{10} , TbW_{10} , and DyW_{10} , respectively. The structures of these LnW_{10} compounds were characterized using PXRD and FT-IR spectroscopies. The PXRD pattern (Fig. S2 in the ESM) displayed characteristic diffraction peaks of the Weakley-type polyoxotungstate within the 2θ range of $5^\circ\text{--}30^\circ$, consistent with the single-crystal simulation [29]. The FT-IR spectrum (Fig. S3 in the ESM) displayed key stretching vibrations at 934 cm^{-1} ($\text{W}=\text{O}$), 841 cm^{-1} ($\text{W}-\text{O}_b-\text{W}$), and 787 and 706 cm^{-1} ($\text{W}-\text{O}_c-\text{W}$) [30], confirming the successful synthesis of LnW_{10} .

2.3 Preparation of the lanthanide-polyoxotungstate-agarose films

Four distinct LnW_{10} -agarose ($\text{Ln} = \text{Sm}, \text{Eu}, \text{Tb}, \text{and Dy}$) composite films with different Ln compositions were prepared via a combined sol-gel and casting method [31]. A representative EuW_{10} -agarose film was fabricated by dissolving agarose (1 g) in boiling water (50 mL), followed by adding a predetermined amount of EuW_{10} solid and heating to form a homogeneous sol. This product (2.5 mL) was subsequently cast onto a glass slide (2.5 cm $\times$ 7.5 cm), cooled, dried in a vacuum oven at 30 °C for 1 day, and peeled to yield a colorless, transparent, flexible, and self-supporting film. Films containing SmW_{10} , TbW_{10} , and DyW_{10} were prepared similarly by substituting EuW_{10} with the corresponding rare earth polyoxotungstates. Under UV excitation of 365 nm, the films exhibited characteristic lanthanide-ion-dependent luminescence: orange, red, green, and white for Sm, Eu, Tb, and Dy, respectively. Figure 1 illustrates the proposed hydrogen bonding interactions and the preparation process.

3 Results and discussion

3.1 Compositional and microstructural characterization of the lanthanide-polyoxotungstate-agarose films

XPS was performed to analyze the oxidation states and chemical environments of the elements in the four LnW_{10} -agarose ($\text{Ln} = \text{Sm}, \text{Eu}, \text{Tb}, \text{or Dy}$) films (Figs. 2, and Figs. S4–S6 in the ESM for Eu, Sm, Tb, and Dy, respectively). The full spectrum of the EuW_{10} -agarose film displayed the characteristic peaks (Eu 3d, W 4f, Na 1s,

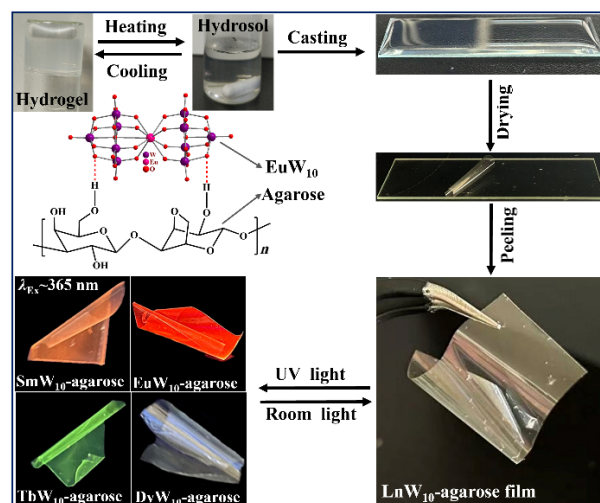


Figure 1 Preparation process and photographs of the LnW_{10} -agarose films.

C 1s, and O 1s) of EuW_{10} and agarose (Fig. 2(a)). The deconvolution of the C 1s region yielded three distinct carbon environments at binding energies of 284.8, 286.3, and 288.2 eV (Fig. 2(b)), corresponding to C–C, C–OH, and C–O–C, respectively. The W 4f spectrum exhibited peaks at 35.6 and 37.8 eV, which were assigned to the $\text{W}^{6+} 4f_{7/2}$ and $4f_{5/2}$ states, respectively, with no detectable W^{5+} (Fig. 2(c)). The Eu 3d spectrum displayed characteristic $\text{Eu}^{3+} 3d_{5/2}$ and $3d_{3/2}$ signals at 1135.1 and 1164.5 eV, respectively (Fig. 2(d)) [32].

FT-IR spectroscopy was used to probe structural changes and elucidate the interactions between the LnW_{10} species and the

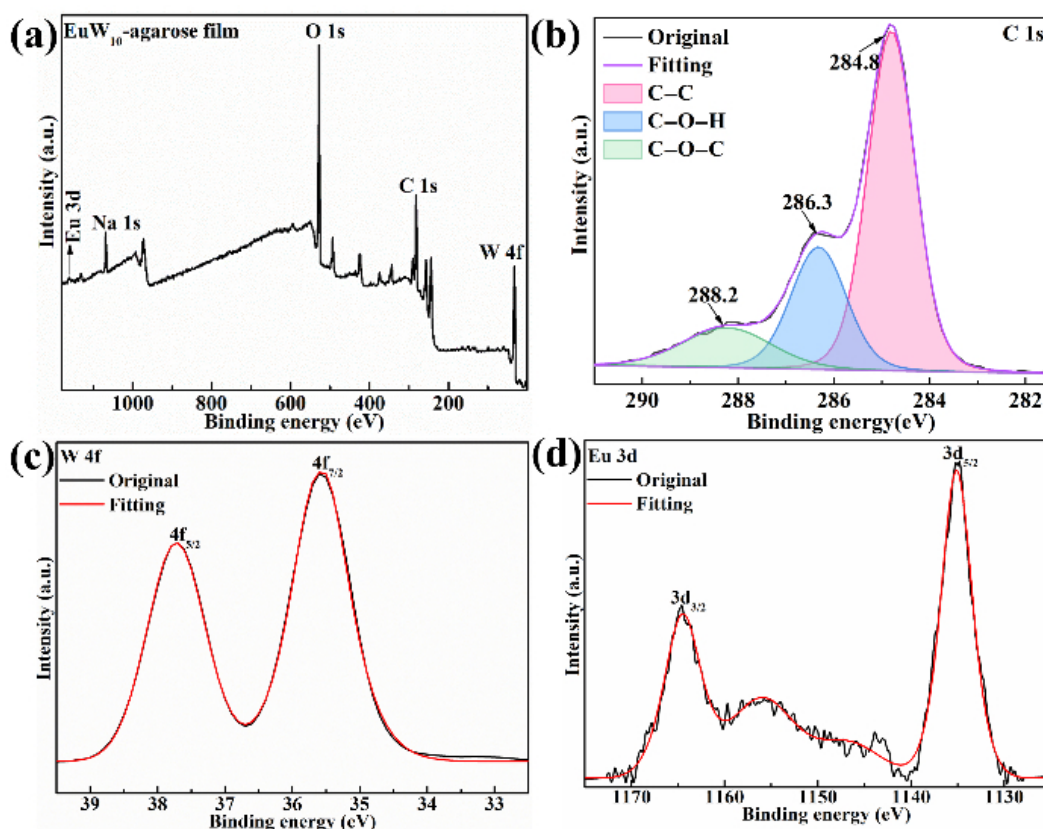


Figure 2 XPS spectra of the EuW_{10} -agarose film: (a) full spectrum, (b) C 1s, (c) W 4f, and (d) Eu 3d.

agarose matrix. Compared with the FT-IR spectra of the pristine LnW_{10} solids, those of the as-prepared LnW_{10} -agarose ($\text{Ln} = \text{Sm}, \text{Eu}, \text{Tb}, \text{or Dy}$) films (Fig. S7 in the ESM) confirmed successful doping and structural preservation of the LnW_{10} units. Characteristic peaks were retained, with measurable shifts in all cases (Table S1 in the ESM). These shifts demonstrate the hydrogen bonding interactions between the LnW_{10} clusters and the agarose matrix [33, 34].

The dispersion of the EuW_{10} nanoclusters in the EuW_{10} -agarose film was examined via HRTEM and selected-area electron diffraction (SAED). Black spots ranging from several to tens of nanometers in size were uniformly distributed throughout the film, indicating that EuW_{10} existed as monodisperse entities or aggregates within the agarose matrix (Fig. 3(a)) [35]. The SAED pattern exhibited distinct lattice fringes with an interplanar spacing of 3.13 Å, which indexes perfectly to the (140) lattice plane of EuW_{10} (Fig. 3(b)). The morphology and elemental composition of the DyW_{10} -agarose nanocomposite film were characterized by SEM and energy-dispersive X-ray spectroscopy (EDX) mapping. The cross-sectional image revealed a smooth surface and a uniform thickness of approximately 5.1 μm (Fig. 3(c)). Furthermore, the EDX elemental maps displayed the homogeneous distributions of C, O, Na, Dy, and W (Figs. 3(d)–3(h)), further confirming the uniform dispersion of DyW_{10} within the agarose matrix [36].

3.2 Photophysical properties of the lanthanide-polyoxotungstate-agarose films

UV–Vis spectroscopy characterized the optical absorption of the LnW_{10} -agarose ($\text{Ln} = \text{Sm}, \text{Eu}, \text{Tb}, \text{and Dy}$) films. Similar to the corresponding LnW_{10} solutions, the films exhibited absorption peaks at ~195 and 260 nm, corresponding to the ligand-to-metal charge transfer (LMCT) transitions of $\text{O}_d \rightarrow \text{W}$ and $\text{O}_{bc} \rightarrow \text{W}$,

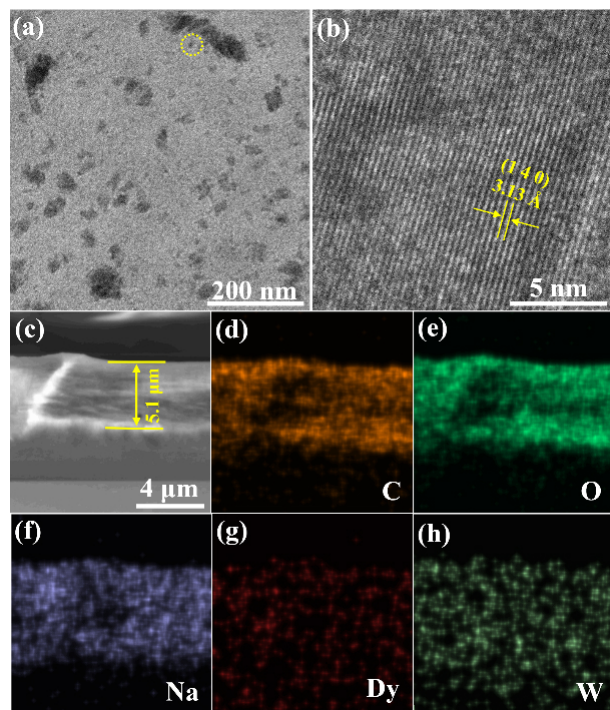


Figure 3 (a) HRTEM image and (b) SAED pattern of the EuW_{10} -agarose film; (c) cross-sectional SEM image of the DyW_{10} -agarose film and (d)–(h) elemental distribution maps of C, O, Na, Dy, and W.

respectively [24, 37]. This result confirms the retention of the optical activity of LnW_{10} . Notably, the $\text{O}_d \rightarrow \text{W}$ absorption peaks of the films were redshifted relative to those in solution (Fig. S8 in the ESM), likely because of the hydrogen bonding interaction between LnW_{10} and the agarose matrix. Transmittance decreased gradually with increasing LnW_{10} loading (Fig. S9 in the ESM). Even at a 70 wt% loading, the films maintained a transmittance of more than 60% in the visible region, demonstrating high optical transparency.

The luminescent properties of the LnW_{10} ($\text{Ln} = \text{Sm}, \text{Eu}, \text{Tb}, \text{or Dy}$) solids and composite films were also examined. All films exhibited characteristic emission peaks of each lanthanide ion (Fig. 4): Sm^{3+} at 564 nm ($^4\text{G}_{9/2} \rightarrow ^6\text{H}_{5/2}$), 602 nm ($^4\text{G}_{9/2} \rightarrow ^6\text{H}_{7/2}$), and 649 nm ($^4\text{G}_{9/2} \rightarrow ^6\text{H}_{9/2}$) (Fig. 4(a)) [23]; Eu^{3+} at 585 nm ($^5\text{D}_0 \rightarrow ^7\text{F}_0$), 598 nm ($^5\text{D}_0 \rightarrow ^7\text{F}_1$), 619 nm ($^5\text{D}_0 \rightarrow ^7\text{F}_2$), 658 nm ($^5\text{D}_0 \rightarrow ^7\text{F}_3$), and 709 nm ($^5\text{D}_0 \rightarrow ^7\text{F}_4$) (Fig. 4(b)) [31, 32]; Tb^{3+} at 492 nm ($^5\text{D}_4 \rightarrow ^7\text{F}_6$), 547 nm ($^5\text{D}_4 \rightarrow ^7\text{F}_5$), and 588 nm ($^5\text{D}_4 \rightarrow ^7\text{F}_4$) (Fig. 4(c)) [38]; and Dy^{3+} at 481 nm ($^4\text{F}_{9/2} \rightarrow ^6\text{H}_{15/2}$) and 577 nm ($^4\text{F}_{9/2} \rightarrow ^6\text{H}_{13/2}$) (Fig. 4(d)) [39]. The insets show the luminescence photographs of the LnW_{10} solids and films under a UV excitation of 365 nm: orange (Sm^{3+}), red (Eu^{3+}), green (Tb^{3+}), and white (Dy^{3+}). The films displayed the same emission peaks as the solids, indicating the preservation of their luminescent properties. However, relative intensities differed across the solid and film samples. For example, in the EuW_{10} system, the intensity ratio of the electric ($^5\text{D}_0 \rightarrow ^7\text{F}_2$) to magnetic ($^5\text{D}_0 \rightarrow ^7\text{F}_1$) dipole transitions increased from 0.297 (solid) to 0.747 (film) because of the sensitivity of the magnetic dipole transition to the symmetry of the Eu^{3+} coordination environment. The hydrogen bonding between the EuW_{10} clusters and the agarose matrix lowered the symmetry around Eu^{3+} , accounting for the observed variation in the emission-intensity ratio [27, 31]. Furthermore, the effect of doping content of LnW_{10} (10 wt%–70 wt%) on luminescence was also investigated. As shown in Fig. S10 in the ESM, the emission intensity increased progressively with the doping content, with no concentration quenching occurring even at 70 wt%. This was attributed to the excellent compatibility between the LnW_{10} clusters and the agarose matrix.

3.3 Photochromic and luminescence-switching properties

The photochromic behavior of the LnW_{10} -agarose ($\text{Ln} = \text{Sm}, \text{Eu}, \text{Tb}, \text{or Dy}$) films was analyzed via UV–Vis absorption spectroscopy. Upon UV irradiation, a broad absorption band emerged around 630 nm (400–900 nm), and the films changed from colorless to blue (Figs. 5(a)–5(d)). Both absorption and the blue color intensified with the irradiation time. Subsequent kinetic profiles (insets of Figs. 5(a)–5(d)), plotting the absorbance at 630 nm vs. time, displayed similar response dynamics and a saturation time of approximately 25 min across all four films. Furthermore, the logarithm of the absorbance at this wavelength increased linearly with the irradiation time (Fig. S11 in the ESM), indicating that the photochromic processes for all LnW_{10} -agarose films ($\text{Ln} = \text{Sm}, \text{Eu}, \text{Tb}, \text{or Dy}$) followed pseudo-first-order kinetics, yielding rate constants (k) of 0.029, 0.060, 0.039, and 0.033 min^{-1} for Sm, Eu, Tb, and Dy, respectively. Conversely, upon exposing the colored films to O_2 at approximately 25 °C, the absorption peak gradually diminished, accompanied by fading. Kinetic analysis of the fading process, performed by plotting the absorbance at 630 nm against the exposure time, revealed a fading response time of approximately 75 min for all films (Figs. S12(a)–S12(d) in the ESM). Similarly, the logarithm of the absorbance decreased linearly with the exposure time (Fig. S13 in the ESM), suggesting that the fading processes also

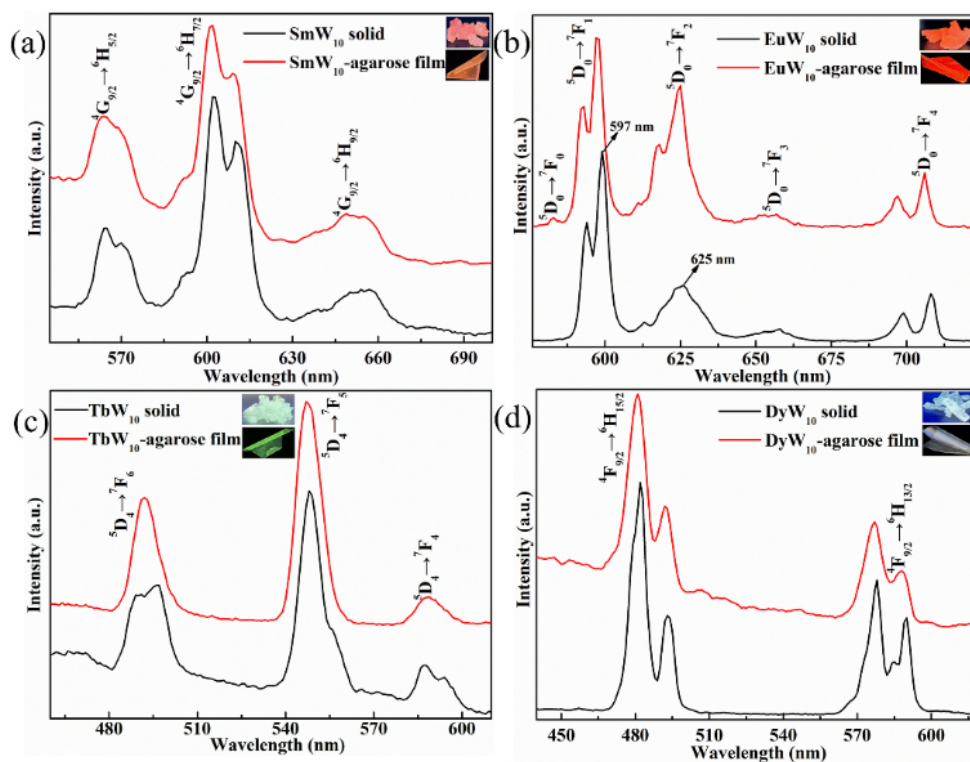


Figure 4 Photoluminescence spectra of LnW_{10} -agarose films: (a) SmW_{10} , (b) EuW_{10} , (c) TbW_{10} , and (d) DyW_{10} .

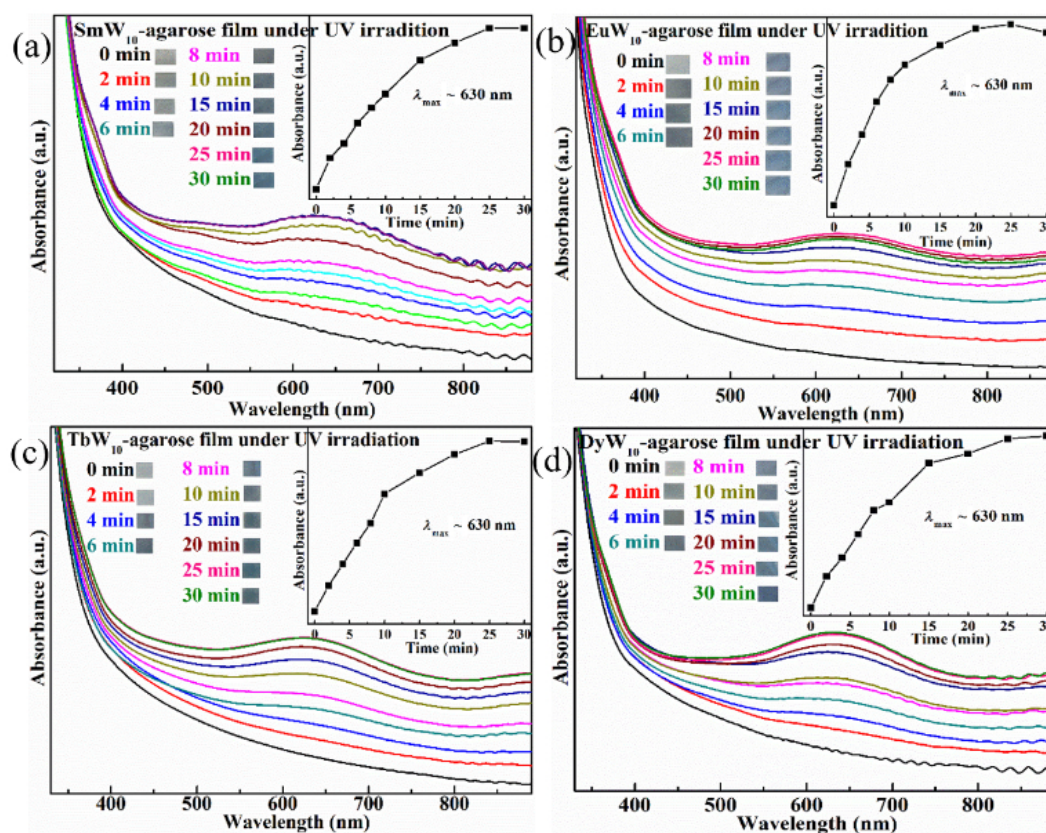


Figure 5 UV-Vis spectral evolution of LnW_{10} -agarose films under UV irradiation: (a) SmW_{10} , (b) EuW_{10} , (c) TbW_{10} , and (d) DyW_{10} . The insets show daylight photographs and plots of absorbance at 630 nm versus irradiation time.

followed pseudo-first-order kinetics, with rate constants (k) of approximately 0.011, 0.011, 0.012, and 0.016 min⁻¹ for Sm, Eu, Tb, and Dy, respectively.

Accompanying the color change, the luminescence of the LnW₁₀ films was significantly altered upon UV irradiation. The pristine films exhibited characteristic emission peaks: Sm³⁺ at 601 nm (⁴G_{9/2} → ⁶H_{7/2}), Eu³⁺ at 598 nm (⁵D₀ → ⁷F₁), Tb³⁺ at 547 nm (⁵D₄ → ⁷F₅), and Dy³⁺ at 481 nm (⁴F_{9/2} → ⁶H_{15/2}). Concurrent with film coloration, these emissions were heavily quenched, as visually evidenced by the fading of orange, red, green, and white luminescence profiles (Fig. 6). The quenching kinetics (insets of Fig. 6) demonstrated a progressive intensity decrease, reaching a minimum within 25 min, matching the photochromic response and indicating concurrent processes. E was calculated at the strongest emission peak using Eq. (1)

$$E = \frac{F_0 - F_t}{F_0} \times 100\% \quad (1)$$

where F_0 and F_t denote the luminescence intensities before and after UV irradiation for 25 min, respectively. The contrast values were 84.3% (Sm³⁺), 92.5% (Eu³⁺), 97.3% (Tb³⁺), and 92.4% (Dy³⁺), as shown in Fig. S14 in the ESM. Conversely, upon O₂ exposure, the quenched luminescence recovered gradually, as confirmed by the reemergence of the characteristic emissions (Fig. S15 in the ESM). Kinetic analysis (insets of Figs. S15(a)–S15(d) in the ESM) showed full recovery within 75 min, consistent with the fading time and indicating synchronous processes.

Thus, the effects of RH and temperature on the bleaching and luminescence-recovery kinetics of the EuW₁₀-agarose films upon O₂ exposure were investigated. At approximately 25 °C, the time

evolutions of the UV–Vis absorption (Fig. S16 in the ESM) and luminescence (Fig. S17 in the ESM) spectra were recorded at RH 33% (Figs. S16(a) and S17(a) in the ESM), 68% (Figs. S16(b) and S17(b) in the ESM), and 86% (Figs. S16(c) and S17(c) in the ESM). The absorbance at 630 nm (Fig. S16(d) in the ESM) and the emission intensity at 598 nm (Fig. S17(d) in the ESM) were examined as a function of the O₂ exposure time. The results indicate that the bleaching and luminescence-recovery rates increased with RH, with corresponding response times of 290, 175, and 120 min, respectively. Similarly, the temperature dependence of these processes was examined at a constant RH of 98%. The temporal evolutions of the UV–Vis absorption (Fig. S18 in the ESM) and luminescence (Fig. S19 in the ESM) spectra were recorded at room temperature (25 °C; Figs. S18(a) and S19(a) in the ESM), 40 °C (Figs. S18(b) and S19(b) in the ESM), and 60 °C (Figs. S18(c) and S19(c) in the ESM). The absorbance at 630 nm (Fig. S18(d) in the ESM) and the emission intensity at 598 nm (Fig. S19(d) in the ESM) were examined against the O₂ exposure time, revealing that both bleaching and luminescence recovery were accelerated at higher temperatures, with corresponding response times of 70, 60, and 40 min, respectively.

3.4 Mechanisms of the photochromic and luminescence-switching process

To elucidate the mechanisms of synchronous photochromism and luminescence switching, the EuW₁₀-agarose film was analyzed by combining the UV–Vis and luminescence spectral overlap, XPS, and luminescence lifetime (τ) measurements. Control experiments on pure agarose films (Fig. S20 in the ESM) did not reveal significant absorption (Fig. S20(a) in the ESM) or luminescence

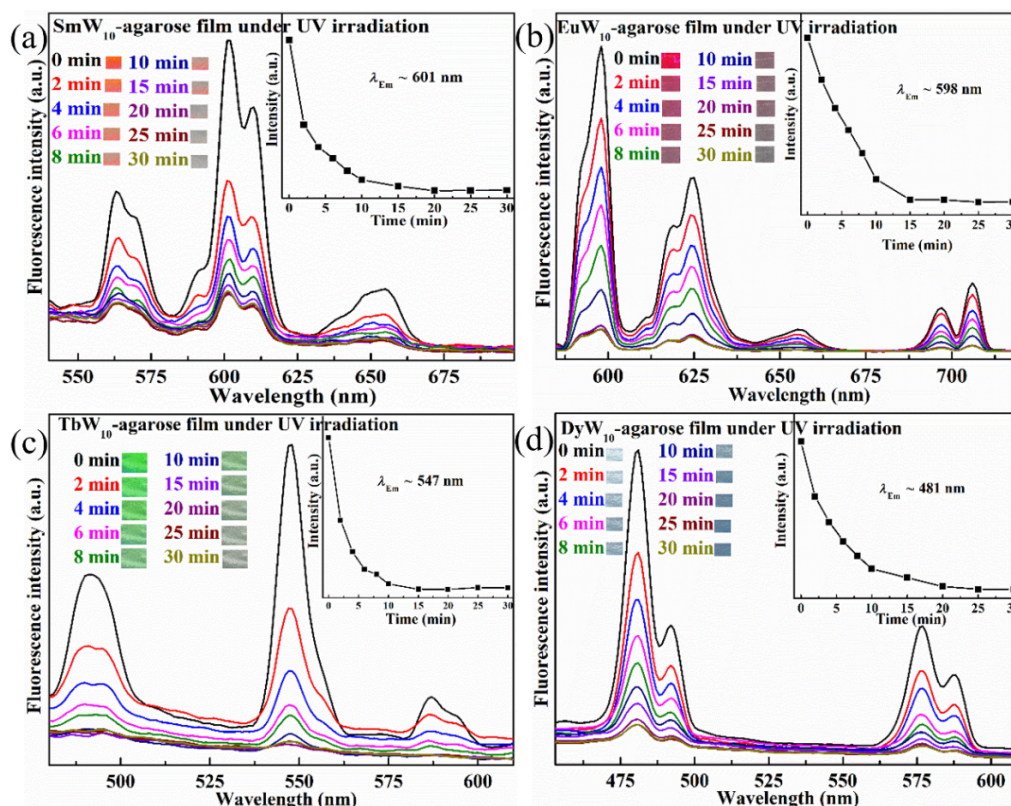


Figure 6 Luminescence spectral evolution of LnW₁₀-agarose films under UV irradiation: (a) SmW₁₀, (b) EuW₁₀, (c) TbW₁₀, and (d) DyW₁₀. Insets show photographs of the films under 365 nm UV excitation and plots of luminescence intensity versus irradiation time.

(Fig. S20(b) in the ESM) signals in the visible region before or after UV irradiation, confirming that EuW_{10} is the active component driving the observed photochromic and switching properties. Before UV irradiation, the EuW_{10} -agarose film did not exhibit absorption in the visible region (black line, Fig. 7(a)). However, upon UV irradiation, $\text{O} \rightarrow \text{W}$ LMCT was induced, triggering the reduction of W^{6+} to W^{5+} , followed by proton transfer from a hydroxyl group of agarose to an O atom at the photoreduced W^{5+} site. This yielded a broad absorption band around 630 nm (blue line, Fig. 7(a)), which was attributed to the $\text{W}^{5+} \rightarrow \text{W}^{6+}$ intervalence charge transfer (IVCT), which changed the color of the film to blue [25, 40]. Consequently, the Eu^{3+} emission spectrum (red line, Fig. 7(a)) overlapped with this IVCT absorption of photogenerated W^{5+} , with this favorable spectral overlap enabling intramolecular Förster resonance energy transfer (FRET) from excited Eu^{3+} (donor) to W^{5+} (acceptor), leading to luminescence quenching (Fig. 7(b)) [25, 27].

XPS confirmed the photoreduction of W^{6+} to W^{5+} . The W 4f spectrum of the initial film displayed two peaks at 37.8 and 35.6 eV, which were assigned to $\text{W}^{6+} 4f_{5/2}$ and $\text{W}^{6+} 4f_{7/2}$, respectively. After 25 min of UV irradiation, these peaks broadened, shifting by approximately 0.2 eV to lower binding energy. Subsequently, deconvolution revealed that each peak split into two components, resolving into $\text{W}^{6+}/\text{W}^{5+}$ pairs at 37.8/37.3 and 35.6/35.2 eV [41]. Quantitative analysis indicated that approximately 35.9% of the W species was W^{5+} after UV irradiation, confirming that photochromism originated from the UV-induced reduction of W^{6+} (Fig. 7(c)). The FRET hypothesis was further supported by the luminescence decay curves (Fig. 7(d)). The decay curves exhibited double-exponential behavior before and after UV irradiation for

25 min, indicating the coexistence of two distinct EuW_{10} states in the film: one that is hydrogen-bonded to agarose and another that is nonhydrogen-bonded. The average τ of the EuW_{10} -agarose film in different states was calculated using Eq. (2)

$$\tau = \tau_1 \times \alpha_1 \% + \tau_2 \times \alpha_2 \% \quad (2)$$

where τ_1 and τ_2 are the short and long decay components, respectively, and α_1 and α_2 are their corresponding fractional amplitudes, respectively (Table S2 in the ESM). Notably, τ decreased sharply from 2809.96 to 376.80 μs upon UV irradiation, with a concomitant decrease in the luminescence quantum yield from 19.68% to 0.11%. This significant decrease is attributed to energy transfer, which provides an additional nonradiative decay pathway for the excited Eu^{3+} , as described by Eq. (3)

$$\tau = \frac{1}{k_r + k_{nr} + k_{ET}} \quad (3)$$

where k_r , k_{nr} , and k_{ET} denote the rate constants for radiative decay, nonradiative decay, and energy transfer, respectively. The reduced excited-state τ confirmed that the intramolecular energy transfer from excited Eu^{3+} to W^{5+} was the dominant luminescence-quenching mechanism [42, 43].

Similarly, upon UV irradiation, the LnW_{10} -agarose films ($\text{Ln} = \text{Sm}^{3+}$, Tb^{3+} , or Dy^{3+}) exhibited phenomena that are analogous to those of the EuW_{10} system, including a spectral overlap between the photoreduced W^{5+} absorption and the Ln^{3+} luminescence (Fig. S21 in the ESM), a shift in the XPS W 4f signals to lower binding energy (Fig. S22 in the ESM), and a decrease in τ (Fig. S23 and Tables S3–S5 in the ESM). Together, these observations indicate that

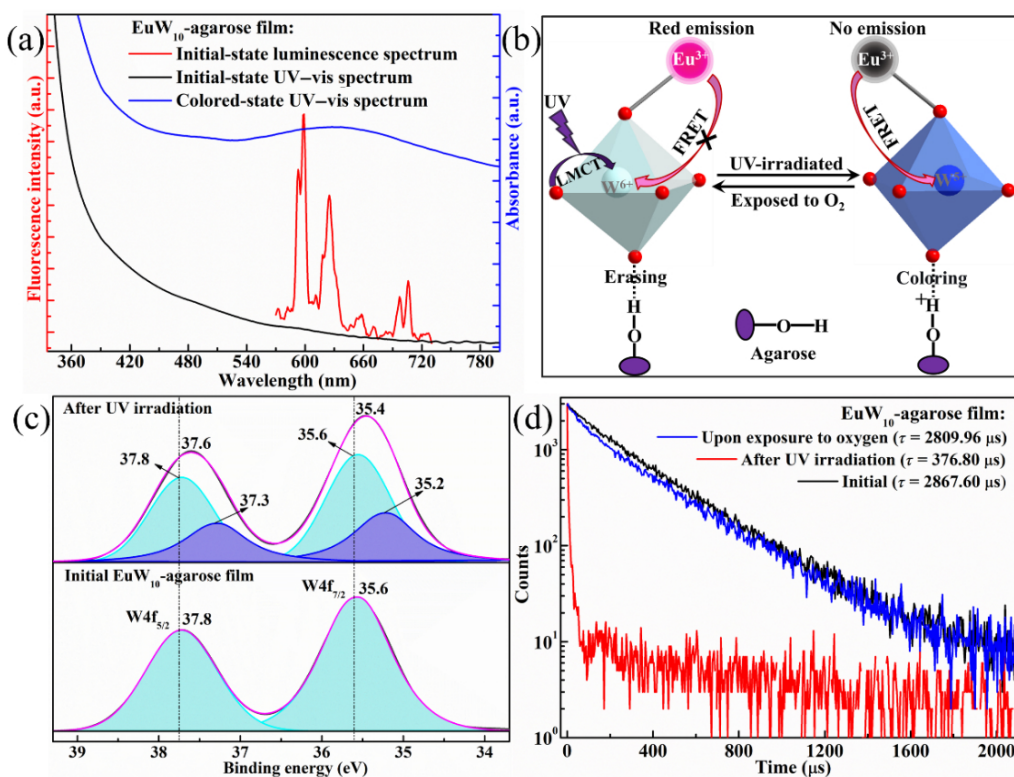


Figure 7 (a) Luminescence (red) and UV-Vis absorption spectra of the EuW_{10} -agarose film in pristine (black) and colored (blue) states. (b) Schematic mechanism of coupled photochromism and luminescence switching. (c) W 4f XPS spectra of the initial (lower) and UV-irradiated (upper) film. (d) Luminescence decay curves in the initial (black), UV-irradiated (red), and oxygen-bleached (blue) states.

intramolecular energy transfer from excited Ln^{3+} to W^{5+} also occurs in these systems.

3.5 Reversibility of photochromic and luminescence switching

The reversibility of photochromic and luminescence switching in LnW_{10} -agarose ($\text{Ln} = \text{Sm}, \text{Eu}, \text{Tb}, \text{or Dy}$) films was evaluated across 10 UV/ O_2 alternating stimulation cycles. After each cycle, the UV-Vis absorption (Fig. S24 in the ESM) and luminescence (Fig. S25 in the ESM) spectra of the colored and bleached states nearly overlapped with those of the initial cycle, demonstrating highly repeatable switching behavior. This excellent reversibility was attributed to the inherent reversible redox process of LnW_{10} .

Upon partial irradiation via a photomask bearing the hollow letters "IMUN" (abbreviation for Inner Mongolia University for Nationalities), the films exhibited a blue "IMUN" pattern in the exposed regions owing to LnW_{10} reduction (Fig. 8(a)). Concurrently, the irradiated regions exhibited quenched luminescence, whereas the masked regions retained their characteristic emissions: orange (Sm^{3+}), red (Eu^{3+}), green (Tb^{3+}), and white (Dy^{3+}), as shown in Fig. 8(b). The blue "IMUN" pattern was fully erasable (Fig. 8(c)), and the luminescence recovered (Fig. 8(d)) upon O_2 exposure via the reoxidation of LnW_{10} . These results demonstrate the suitability of the composite films for erasable inkless security printing, anticounterfeiting labels, and rewritable optical memory.

4 Conclusions

Four distinct LnW_{10} -agarose ($\text{Ln} = \text{Sm}, \text{Eu}, \text{Tb}, \text{or Dy}$) composite films with high transparency and distinct luminescent colors were prepared via a combined sol-gel and casting method using different Ln compositions. FT-IR, TEM, SEM, and EDX mapping analyses confirmed that the Ln-LnW_{10} films were uniformly doped into the agarose matrix through hydrogen bonding. As the LnW_{10} doping content increased, the transmittance of the films gradually decreased, whereas their luminescence intensity increased. Notably, the films exhibited photochromism and luminescence quenching under UV irradiation, with quenching efficiencies of 84.3% (Sm^{3+}), 92.5% (Eu^{3+}), 97.3% (Tb^{3+}), and 92.4% (Dy^{3+}). Upon O_2 exposure, both color and luminescence recovered to their initial states. The films maintained robust, reversible photochromism and luminescence switching across a minimum of 10 alternating UV/ O_2 exposure cycles. These rewritable properties highlight their potential for applications in erasable inkless security printing,

anticounterfeiting labels, and rewritable optical memory devices. Furthermore, this study opens pathways for diverse next-generation applications, including near-infrared-emitting Ln-POM systems based on Yb^{3+} , Nd^{3+} , and Er^{3+} , as well as multistimuli-responsive materials sensitive to light, electricity, and humidity.

Electronic Supplementary Material: Supplementary material (PXRD, FT-IR, XPS, UV-vis spectra, luminescence spectra, etc.) is available in the online version of this article at <https://doi.org/10.26599/POM.2026.9140145>.

Data availability

All data needed to support the conclusions in the paper are presented in the manuscript and the Electronic Supplementary Material. Additional data related to this paper may be requested from the corresponding author upon request.

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Declaration of competing interest

The authors declare no competing interests relevant to this article.

Author contribution statement

H.: Investigation, software, writing original draft. X. L.: Software, investigation, analyzing. A.: Investigation, data curation. Y. T. Y.: Investigation. X. H. W.: Supervision, data curation, investigation. P. H. L.: Data curation, review. Y. W.: Investigation, data curation. J. H. L.: Data curation, review. B. W.: Experimental design, supervision, writing – review, editing, and funding acquisition.

Use of AI statement

None.

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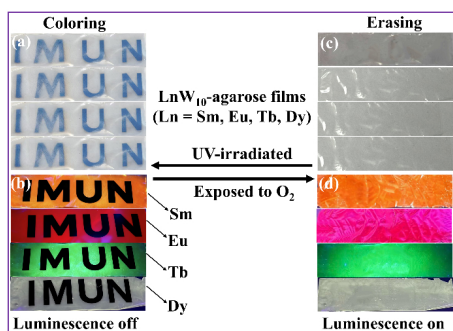


Figure 8 Digital photographs of LnW_{10} ($\text{Ln} = \text{Sm}, \text{Eu}, \text{Tb}, \text{Dy}$) films: (a) coloring and (b) luminescence quenching after UV irradiation through a mask; (c) erasing and (d) luminescence recovery after reoxidation in oxygen.

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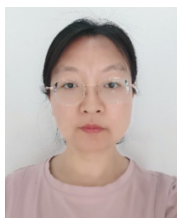
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