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Integrating Photorewritable Fluorescent Information in Shape-Memory Organohydrogel Toward Dual Encryption

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As one of the severe global problems, counterfeiting information has brought a huge negative impact on every aspect of human society. Though various anticounterfeiting strategies, including fluorescent materials, are widely developed for dealing with the above problem, the information security level needs to be further improved due to sophisticated hacking techniques. In this study, an organohydrogel is designed by constructing interpenetrating organohydrogel networks, in which naphthalimide moieties (DEAN, green-yellow emission) are introduced in hydrophilic poly(*N*,*N*-dimethylacrylamide) (PDMA) hydrogel network and anthracene units (blue emission) are copolymerized in hydrophobic polystearate methacrylate (PSMA) organogel network. Triggered by UV light of 365 nm, the unimer–dimer transition occurs and leads the fluorescent color of organohydrogel to change from blue to faint yellow, making secret information be stored with the assistance of photomasks. Furthermore, by combining crystallization-induced shape memory performance, dual encryption can be achieved. This fluorescent organohydrogel provides a new idea for fabricating smart materials with the ability of encryption–decryption, which is of great significance in information security protection.

1. Introduction

The rapid development of information technology has provided convenience to people's lives, but also brings potential information security risks in the fields of pharmaceutical, food industries, and so on. Thus, rather than simply storing information, the ability to encrypt information has more important significance in information security. Nowadays, many scientists focus on the development of late-model anti-counterfeiting materials, and corresponding strategies,[1–4] including bar code, two-dimensional code, holograms, luminous materials, and so on. Among them, luminous materials can be one of the best candidates for anti-counterfeiting due to their responsiveness to UV light,[5,6] the diversity of vivid colors[7,8] as well as various luminescence modes.[9,10]

Given the excellent modifiability, containment and unique stimulus-responsiveness of polymeric hydrogels,[11–14] the combination of luminescence materials and intelligent hydrogels can make the stored information dynamic and improve security level. Up to now, a large number of studies have focused on information storage or information encryption[15–18] on the basis of fluorescent hydrogels. For example, Ji's group[19] fabricated a self-assemble hydrogel with polyvinyl alcohol (PVA) hydrogel doping three kinds of aggregation induced emission (AIE) fluorescent monomers. The prepared multifluorescent hydrogel could store a large amount of information by introducing 1D barcodes or 2D codes and arraying the fluorescent blocks. Saunders and coworkers[20] employed microgels (MG) as

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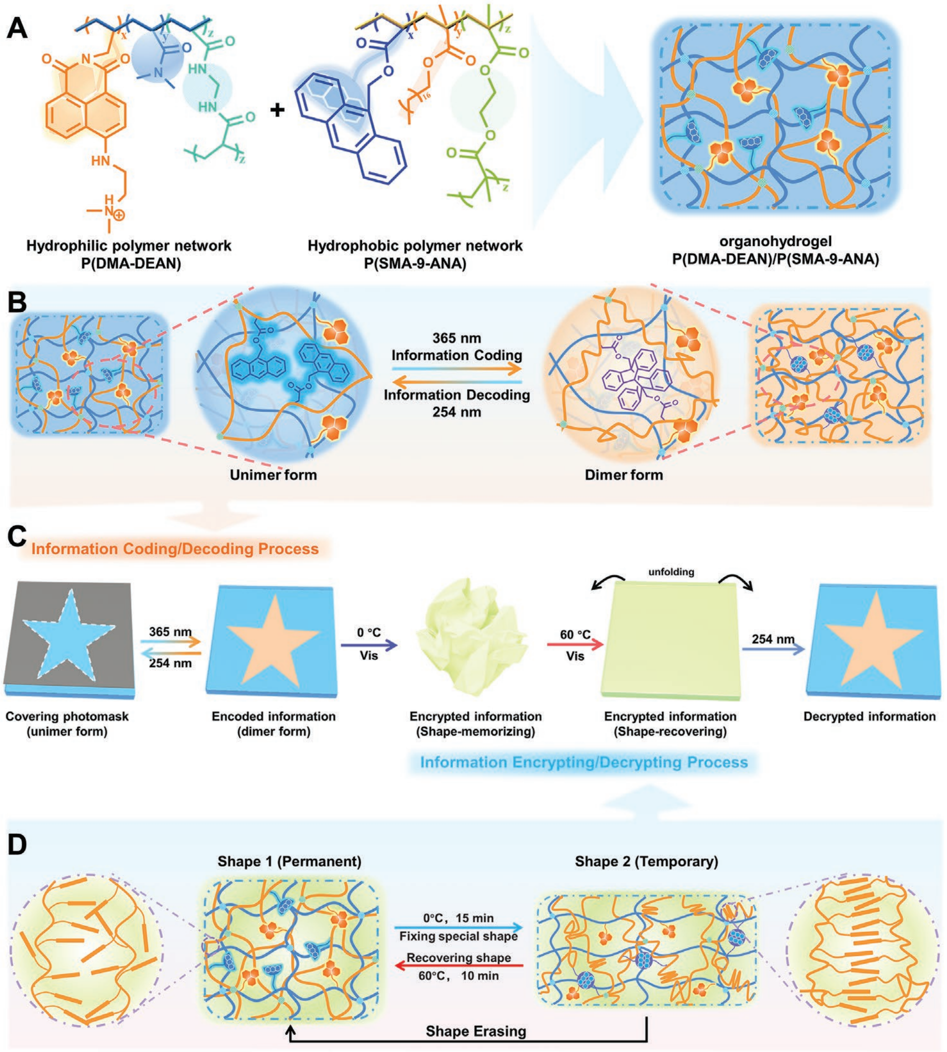
the building blocks, containing photo-crosslinking/cleaving methylcoumarin (CMA) moieties, through which the fluorescent hydrogel could change color and store information. Zhao's group[21] constructed a hybrid hydrogel within lanthanide complexes and diarylethene photochromic unit. Under the stimulus of UV or Vis, the diarylethene rings on the polymer chains display open–close behavior, leading or inhibiting fluorescence resonance energy transfer with the lanthanide complex thus could be used for information storage. These elegant attempts prove that fluorescent hydrogel could be a suitable candidate for information storage, but still lacks security level due to direct deciphering under UV light.

Owing to the coexistence of hydrophobic and hydrophilic polymer networks in the developed organohydrogel, both hydrophilic and hydrophobic fluorescent monomers can be introduced, which provides a new inspiration for obtaining novel cryptographic material with a variety of colors. Herein, we report an anthracene organohydrogel with recodable fluorescent patterns and reconfigurable stereostructure. The organohydrogel is fabricated by interpenetrating hydrogel and organogel polymer networks (**Scheme 1**A), in which the green-yellow-emitting fluorescent unit (DEAN) is in the hydrophilic poly(*N*,*N*-dimethylacrylamide) (PDMA) polymer chains, and photo-switchable anthracen-9-yl acrylate (9-ANA) is in the hydrophobic poly(methacrylate stearate) (PSMA) polymer chains. In the system, 9-ANA exhibits on-off switching of blue emission and goes through unimer-to-dimer conversion when exposed to 365 nm UV light (Scheme 1B). In the meanwhile, the inherent crystallization property of PSMA endows the organohydrogel with a temperature-mediated shape-memory effect (Scheme 1D). Therefore, various fluorescent patterns can be coded on the surface of organohydrogel with the assistance of photomasks, after which the organohyrogel is subsequently deformed into specific configurations to complete the process



Scheme 1. Schematic illustration of fluorescent organohydrogel P(DMA-DEAN)/P(SMA-9-ANA) for dynamic information coding/decoding process. A) The fluorescent organohydrogel was comprised of fluorescent monomer (DEAN, green-yellow) copolymerized with DMA in hydrogel network and fluorescent monomer (9-ANA, blue) copolymerized with SMA in organogel network. B) Schematic illustration of the information coding/decoding process of fluorescent organohydrogel P(DMA-DEAN)/P(SMA-9-ANA). C) The procedures of information-coding, dual-encryption, and dynamic fluorescent decryption. D) Schematic illustration of the shape memorizing/recovering process of fluorescent organohydrogel P(DMA-DEAN)/P(SMA-9-ANA).

of information encryption. The hidden information can be decoded by sequential steps of the shape recovery process and UV light irradiation (Scheme 1C). Such an organohydrogel with outstanding shape-memory ability and tunable fluorescent pattern has been developed for dual-encryption, showing a much higher security level.

2. Results and Discussion

2.1. Fabrication of P(DMA-DEAN)/P(SMA-9-ANA) Organohydrogel

The fluorescent monomer 4-(*N,N*-dimethylaminoethylene) amino-nallyl-1,8 naphthalimide (DEAN) was firstly prepared according to previous work.[16,22] Meanwhile, we synthesized an anthracene fluorescent monomer with double bonds, 9-ANA, for the first time (Figure S1, Supporting Information). Specifically, the fluorescent monomer 9-ANA shows excellent unimer–dimer transition via the switch of UV light between 365 and 254 nm[23,24] (Figures S2–S6, Supporting Information). The hydrogel polymer network was constructed through copolymerization of DMA and slight DEAN in the presence of N,N′-Methylene bis(acrylamide) (BIS, crosslinker) and ammonium persulfate (APS, initiator). After being fully dehydrated in acetone, P(DMA-DEAN) hydrogel was immersed in an oily precursor solution containing stearyl methylacrylate (SMA), 9-ANA (UV-responsive fluorescent monomer), ethylene dimethacrylate (EGDMA, crosslinker) and 2,2-diethoxyacetophenone (DEAP, photoinitiator). Triggered by UV light, the hydrophobic network was polymerized in the presence of the hydrophilic polymer network. As a result, P(DMA-DEAN)/P(SMA-9-ANA) organohydrogel was obtained after removing unreacted monomers in ethanol and fully swelling in the deionized water (Figure S7, Supporting Information). Then the aimed dry P(DMA-DEAN)/P(SMA-9-ANA) organohydrogels were got by air-drying at room temperature and put in an 80 °C oven for achieving a complete unimer–dimer transformation of 9-ANA.

2.2. Structural Characterization of Prepared Organohydrogel Before and After UV Irradiation

Due to the photocontrolled unimer–dimer transition of anthracene moieties, the prepared organohydrogel showed changes in both network structure and fluorescence properties when exposed to UV light with different wavelengths (**Figure 1**). When UV light of 365 nm was applied, the dimer structure of anthracene moieties was obtained, leading to the formation of new chemical crosslinking points. As shown in Figure 1A,B, the original P(DMA-DEAN)/P(SMA-9-ANA) organohydrogel owns a porous structure with sizes of 5–10 nm, which were occupied by smaller pores (Figure 1B) after UV irradiation for 2 h through scanning electron microscope (SEM). What's more, anthracene moiety, emitting blue fluorescence, gradually weakens its fluorescent intensity after the transition to a dimer structure under UV light (365 nm), which can be observed by confocal laser scanning microscopy (CLSM). The unimer–dimer transition of the P(DMA-DEAN)/P(SMA-9-ANA) can be further verified by the changes in dynamic mechanical properties such as the storage modulus (*G*') (Figure 1C). After UV irradiation for 2 h, the organohydrogel got an order of magnitude improvement in *G*', indicating the increase of crosslinking points.

To verify the chemical composition and structure of the hydrogel and organohydrogel, P(DMA-DEAN) hydrogel and P(DMA-DEAN)/P(SMA-9-ANA) organohydrogel before and after UV irradiation (365 nm) were measured by attenuated total reflection Fourier transformed infrared spectrometer (ATR-FTIR). As shown in Figure 1D, with similar spectra, some new peaks appeared in that original P(DMA-DEAN)/P(SMA-9-ANA) organohydrogel (yellow line). The emerging absorption peaks at 1729.84 cm^{-1} was attributed to the presence of C=O groups in PSMA polymer chains, and significantly enhancing peaks at 2923.5 cm^{-1} was caused by the asymmetric stretching of –CH2– groups belonging to hydrophobic PSMA polymer chains, which illustrates the successful introduction of PSMA into P(DMA-DEAN) hydrogel matrix. Moreover, the clear identifiable signals of organohydrogel at 1540.85 cm^{-1} (yellow line) and 1467.56 cm^{-1} (red line), corresponding to the benzene group of the anthracene, show an obvious shift and indicate the unimer–dimer transition. As illustrated by the Raman spectra (Figure 1E), some typical bands in the range of 1000–1200 cm^{-1}, belonging to the aromatic anthracene backbone, disappear after dimerization. Specifically, original multiple peaks at 1026, 1039, 1061, 1083, 1105, 1128, and 1150 cm^{-1} converted into two board peaks (1143 and 1077 cm^{-1}) for anthracene moieties, which correspond to C–C and/or ring deformation modes.[25,26] To further certify the mechanism of the unimer–dimer transition of anthracene moieties, density functional theory (DFT) calculations were employed to analyze the energy gaps and orbital energy levels of the fluorescent monomers involved (Figure 1F). The separation degree of electron cloud between HOMO and LUMO of unimer form is larger than that of dimer form, and the energy gap (ΔE) between HOMO and LUMO of dimer form (1.049 eV) was less than that of unimer form (2.256 eV), indicating that it is more susceptible for unimer to be excited and switched to dimer under 365 nm. Once the unimer of anthracene moieties in the organohydrogel is switched to the dimer, the blue fluorescent emission of the original P(DMA-DEAN)/P(SMA-9-ANA) organohydrogel can be decreased.

2.3. Dynamic Fluorescence Color Variation of P(DMA-DEAN)/P(SMA-9-ANA) Organohydrogel

In our organohydrogel system, the original anthracene moieties (unimer form) emit blue fluorescence can switch to none fluorescence state (dimer form) by applying UV light of 365 nm. Through the coupling of fluorescent colors with DEAN (yellowish-green fluorescence), the organohydrogel could achieve the dynamic fluorescent change from blue to yellow by the irradiation of UV light (365 nm). Determined by the characteristic peak of DEAN and 9-ANA on UV–vis absorption spectra (Figure S8, Supporting Information), the organohydrogel can emit blue and yellowish-green fluorescence in response to a certain excitation wavelength (254 nm). The PL mapping spectrum (**Figure 2**A) revealed that there are two emission centers

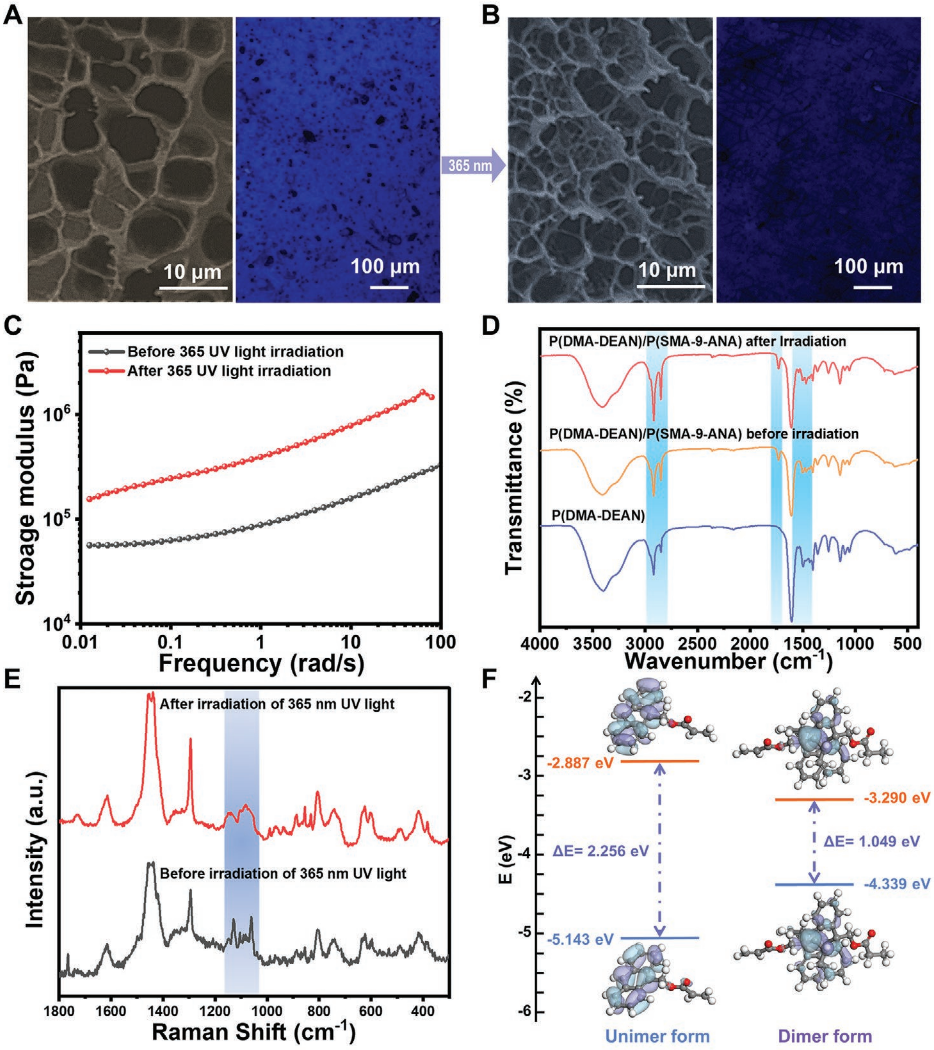


Figure 1. Characterization of P(DMA-DEAN)/P(SMA-9-ANA) organohydrogel before and after exposure to UV lamp (365 nm) for 2 h. SEM and CLSM images of P(DMA-DEAN)/P(SMA-9-ANA) organohydrogel A) before UV irradiation and B) after UV irradiation. C) Storage modulus (G') of P(DMA-DEAN)/P(SMA-9-ANA) organohydrogel before and after UV irradiation in the range of 0.01 to 100 rad s^{-1} at a constant shear strain of 1% and temperature of 25 °C. D) FT-IR spectra and E) Raman spectra of pure P(DMA-DEAN) hydrogel and P(DMA-DEAN)/P(SMA-9-ANA) organohydrogel before and after UV irradiation. F) The energy of highest occupied molecular orbital (HOMO) and lowest unoccupied molecular orbital (LUMO) of unimer form and dimer form of 9-ANA, respectively.

(blue and yellowish-green) in the P(DMA-DEAN)/P(SMA-SMA) organohydrogel (left). Relatively, UV light (365 nm) triggered the unimer–dimer transition process of anthracene moieties leads the weakening of both centers to varying degrees, among which the former was more attenuated.

As the coupling fluorescent color could be tuned by adjusting the proportion of the two fluorescent monomers, a variety of fluorescent color variations could be achieved under the irradiation of UV light (365 nm). As shown in Figure 2B, when keeping the content of 9-ANA in the oily precursor as 1 mg mL^{-1}, the fluorescent organohydrogels displayed a rich color variation by controlling the UV irradiation time and the amount of DEAN. When the amount of DEAN was 0.1 mg mL^{-1}, the organohydrogel shows the most obvious color change from blue to faint yellow with the extension of UV irradiation, which was selected for further study. In addition, the color variation could also be monitored by the fluorescence spectra (Figure 2C; Figures S9–S12, Supporting Information), in which there is a distinct weakening in the blue fluorescence with the extension of UV irradiation (365 nm). The Commission Internationale de L' Eclairage (CIE) coordinates of the selected organohydrogel before and after UV irradiation are (0.2463, 0.3202) and (0.3040, 0.4421), corresponding to blue and faint yellow (Figure 2D). It is worth noting that our organohydrogel shows excellent cyclic stability through exposure to UV light with different wavelengths (365 and 254 nm), which were tested by the fluorescence spectra (Figure 2E; Figure S13, Supporting Information). The fluorescent intensity of 9-ANA at 417 nm gradually decreased when the exposure time (365 nm) extended from 0 min to 60 min. In contrast, when exposed to UV light at 254 nm, the fluorescent intensity located at 417 nm increased. Meanwhile, the fluorescent intensity of DEAN at 515 nm was

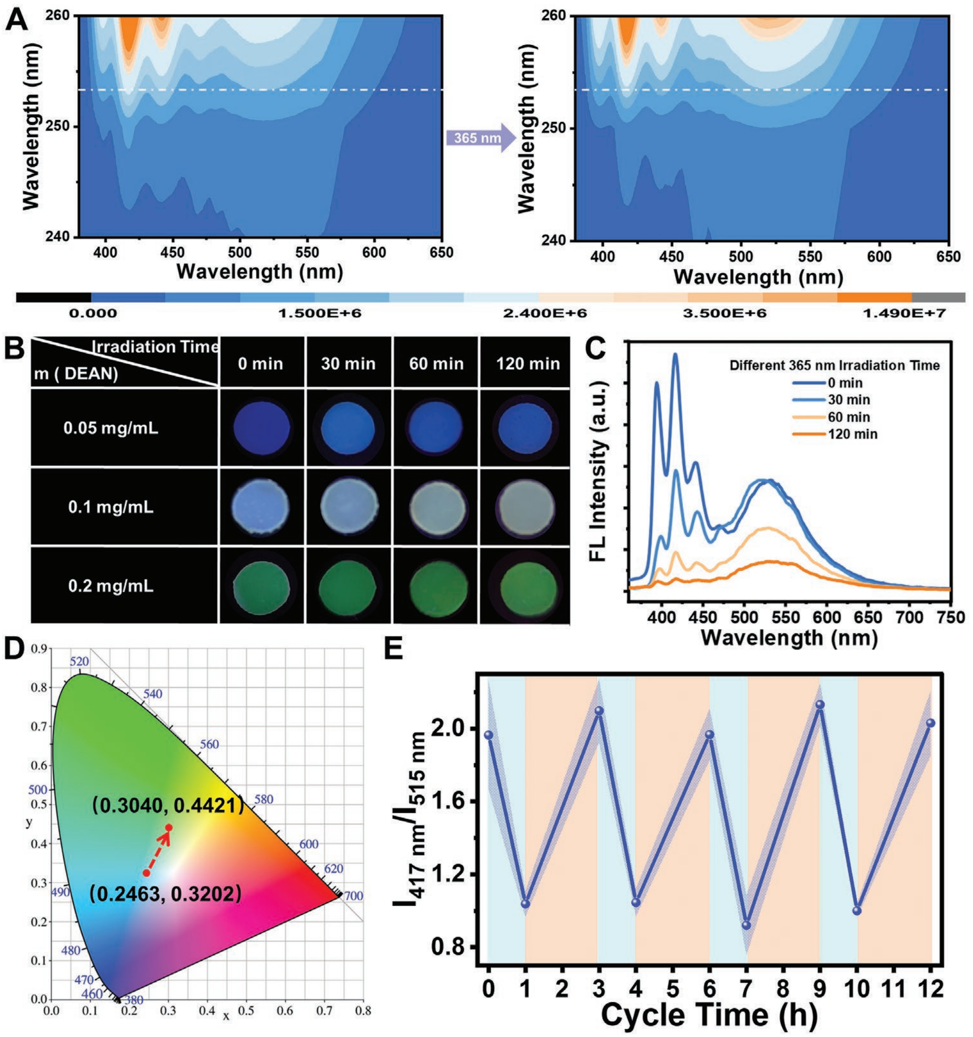


Figure 2. Realization of dynamic fluorescent change. A) PL mapping spectra of P(DMA-DEAN)/P(SMA-9-ANA) organohydrogel before and after UV irradiation (365 nm, 2 h). B) The array of fluorescent colors of P(DMA-DEAN)/P(SMA-9-ANA) organohydrogel with different content of DEAN (0.05, 0.1, and 0.2 mg mL^{-1}) and various irradiation time (0, 30, 60, and 120 min) under 365 nm UV lamp. C) Fluorescence spectra P(DMA-DEAN)/P(SMA-9-ANA) organohydrogel (with 0.1 mg·mL^{-1} DEAN) upon irradiation of 365 nm UV light with different time (0, 30, 60, and 120 min). D) Fluorescence color change of P(DMA-DEAN)/P(SMA-9-ANA) organohydrogel before and after 2 h UV irradiation of 365 nm UV light at a CIE (1931) chromaticity diagram. E) The circular changes of fluorescence intensity ratio $I_{417\ \mathrm{nm}}/I_{515\ \mathrm{nm}}$ through 1 h irradiation of 365 nm UV light and 2 h irradiation of 254 nm UV light.

almost constant, which can also be observed through the fluorescent intensity ratio at 417 nm and 515 nm ($I_{417\ \mathrm{nm}}/I_{515\ \mathrm{nm}}$).

2.4. Dual Information Coding, Encryption, and Decryption based on P(DMA-DEAN)/P(SMA-9-ANA) Organohydrogel

Given the perfect fluorescence reversibility, the P(DMA-DEAN)/P(SMA-9-ANA) organohydrogels were first cut into the shape of ginkgo-leaf and dolphin. With the irradiation time (365 nm) increasing, the orgnaohydrogels adjusted their fluorescent color from blue to faint yellow (**Figure 3**A,B), which could make a full recovery under UV light of 254 nm. Thus, cyclic light-writing/light-erasing processes can be achieved with the assistance of programmed photomasks (Figure 3C). As shown in Figure 3D, several Olympic sports icons such as wrestling, tennis and volleyball were written and erased in sequence via adopting UV light at 365 and 254 nm. One thing that needs to point out is that the patterned organohydrogel could be stored for more than two months in dark (Figure S14, Supporting Information).

As our organohydrogel possesses a hydrophobic network, which was made up of SMA that can form micro-crystalline domains at a certain temperature.[27–29] From the differential scanning calorimetry (DSC) curve of the P(DMA-DEAN)/P(SMA-9-ANA) organohydrogel (Figure S15, Supporting Information), crystallization and melting temperature were anchored at 14.8 and 38.5 °C, which means the organohydrogel could carry out shape memory for fixing specific shapes. Thus, the combination of fluorescence color change and shape memory properties can offer a new way for dual information encryption. As shown in **Figure 4**A, UV light of 365 nm was firstly utilized for information coding, after which written information can further be encrypted via shape memory process at low temperature. Only when the fixed shape was unfolding

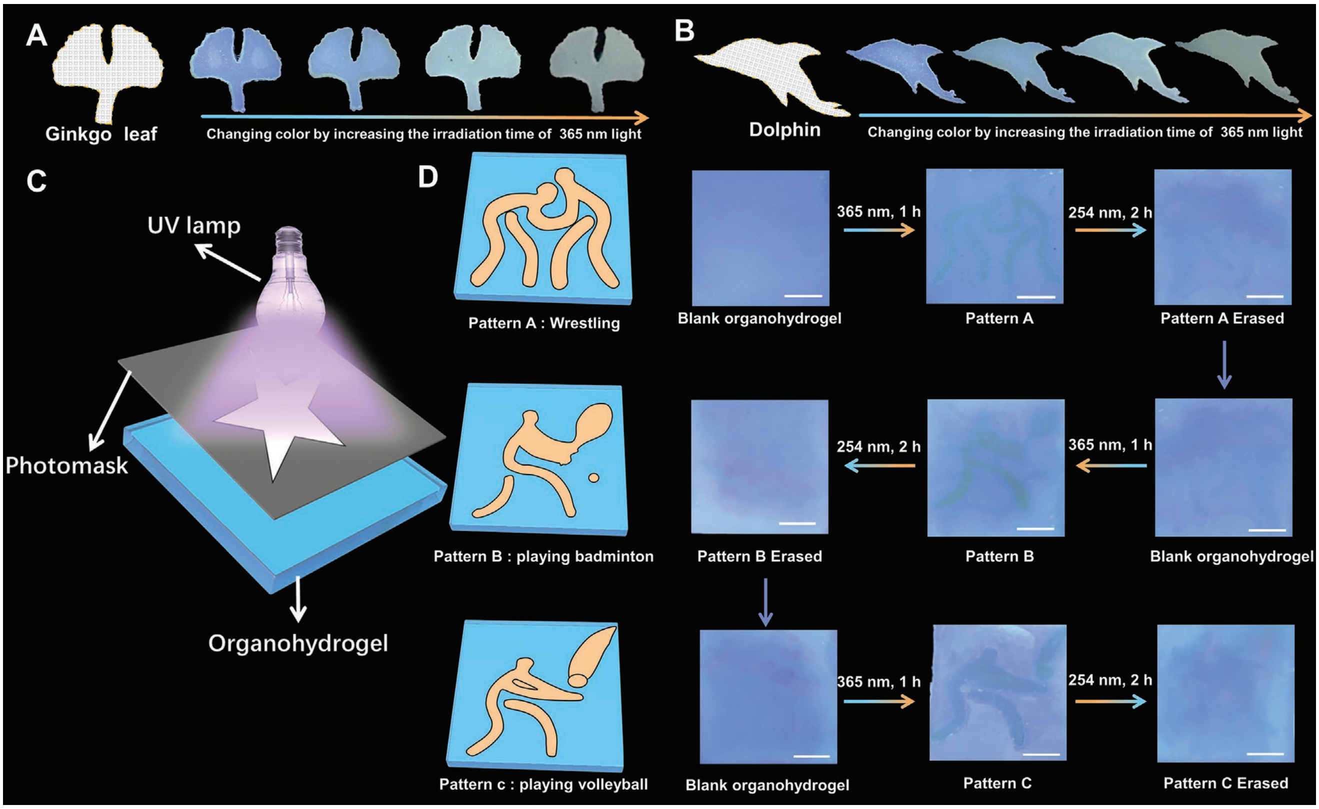


Figure 3. Circular information coding/decoding process based on the unimer–dimer conversion of anthracene moieties in the P(DMA-DEAN)/P(SMA-9-ANA) organohydrogel. A) The Ginkgo leaf shape and B) the dolphin shape of P(DMA-DEAN)/p(SMA-9-ANA) organohydrogel exhibit gradually fluorescent change. C) Schematic illustration of patterning achieved by partially irradiated by 365 nm UV lamp via the assistance of programmed masks. D) Photos showing the "light-writing/light-erasing" cycles through UV light irradiation at 365 and 254 nm, respectively (scale bar: 0.5 cm).

and the organohydrogel was exposed to UV light of 254 nm can the secret message be decrypted. As a proof-of-concept of dual anticounterfeiting, the organohydrogel with the message of "grapes" was encrypted by being folded into the shape of paper cranes. When being put in a 60 °C oven for 15 min, the hidden information on the flat organohydrogel can be read under the irradiation of UV light (254 nm). With the increase of the irradiation time, the message can be erased and ready for new information coding. Subsequently, patterns such as "bamboos" and "flowers" were hidden in the shapes of aircraft and flower, which can be decrypted and erased under certain conditions.

3. Conclusion

In summary, we fabricated a fluorescent organohydrogel possessing hydrophilic and hydrophobic polymer networks, in which DEAN was introduced into the former network and 9-ANA were copolymerized in the later network. Owning to the photoswitching properties of the anthracene moieties, the fluorescent organohydrogel could tune color from original blue to faint yellow, which could recover to blue fluorescent color when exposed to UV light of 254 nm. Besides the color change, the crystallization property of the hydrophobic polymer network in the organohydrogel system also could provide shape-memory ability, leading to dual encryption of information. Only being unfolded and exposed to UV light of 254 nm can the information on the organohydrogel be decrypted and read. In a word, our strategy provides a new method for designing and fabricating novel fluorescent dual-encrypted materials, improving the information security level.

4. Experimental Section

Materials: *N*,*N*-Dimethylacrylamide (DMA), ammonium persulfate (APS), *N*,*N*″-methylene bis(acrylamide) (BIS), *N*,*N*,*N*″,*N*′-tetramethylethylenediamine (TEMED), 2,2-diethoxyacetophenone (DEAP), ethylene dimethacrylate (EGDMA), acryloyl chloride were commercially provided by Aladdin. Anthracen-9-ylmethanol (9-AN), (*N*,*N*-dimethylamino) pyridine (DMAP), stearyl methylacrylate (SMA), anhydrous dichloromethane (DCM) were bought from J&K Chemical Co., Ltd. HCl, $NaHCO_3$, ethanol, anhydrous magnesium sulfate were purchased from Sinopharm Chemical Reagent Co., Ltd. DEAN as a fluorescent comonomer was synthesized according to our previous report. All reagents were used without any treatment or purification.

Synthesis of Fluorescent Monomer Anthracen-9-yl Acrylate (9-ANA): The synthetic route of 9-ANA was shown in Figure S1 (Supporting Information). Firstly, under N_2 protection, 0.416 g (2 mmol) 9-AN and 0.024 g (0.2 mmol) DMAP were dissolved in 100 mL dry dichloromethane and 0.404 g (4 mmol) triethylamine in the ice water bath. Then, a mixture solution of 0.416 g acryloyl chloride and 5 mL dry dichloromethane was added dropwise. The product was obtained by stirring for 4 h at room temperature. Dichloromethane was poured into the obtained mixture

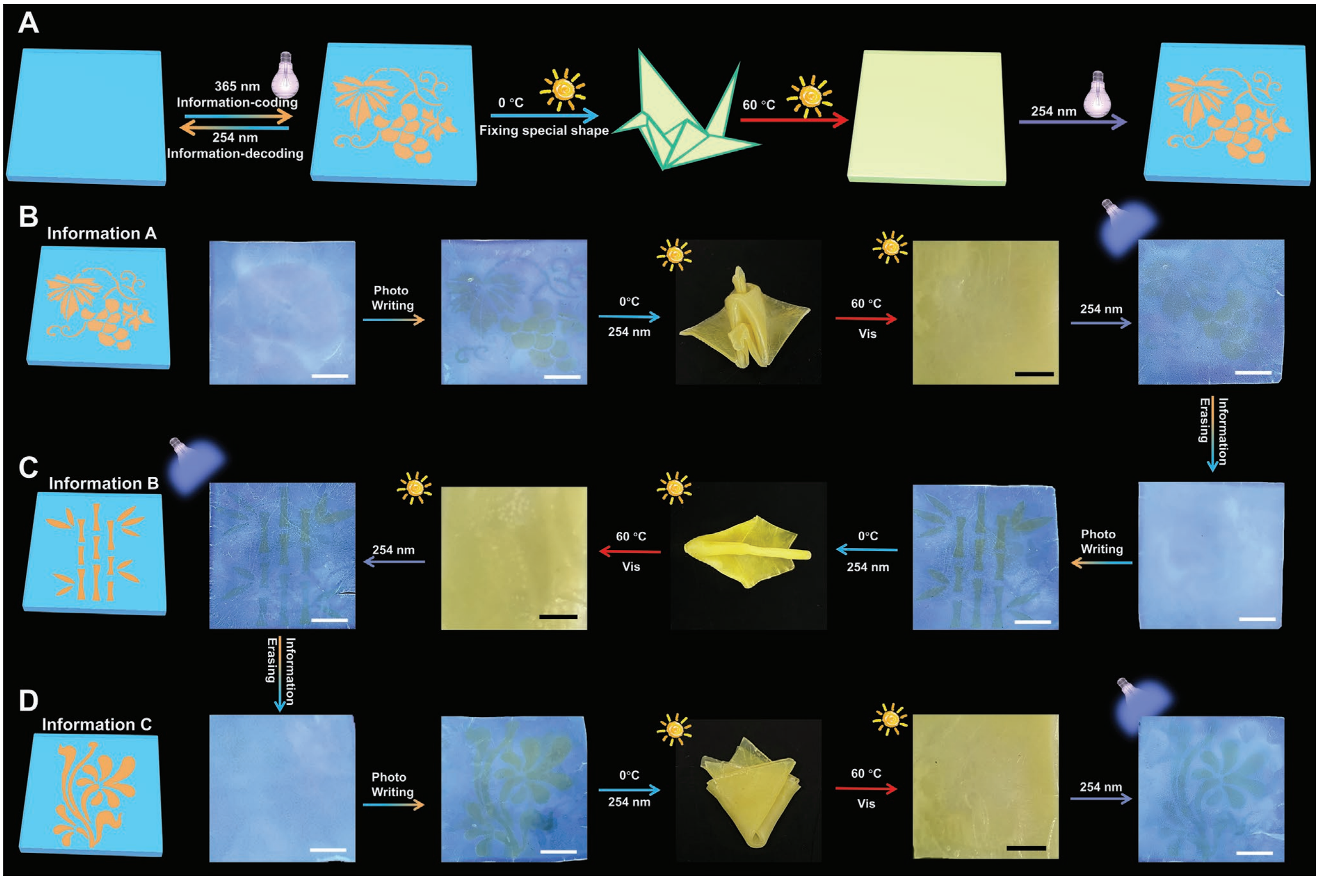


Figure 4. Advanced dual information coding, encryption and decryption based on fluorescent organohydrogels with dynamic fluorescence change and shape memory performance. A) Schematic diagram of information coding, dual encryption and decoding process. B–D) Various patterns such as grapes (B), bamboos (C), and flowers (D) were written through the irradiation of 365 nm UV light, then the coded information were further encrypted by the process of shape memory. The hidden information could be obtained by shape recovery and exposure to 254 nm UV light (scale bar: 0.5 cm).

liquid to dilute it, and then added 0.02 M hydrochloric acid and saturated sodium hydrogen carbonate to washing mixture respectively. Finally, the solvent was removed by rotary evaporator to obtain the product.

Preparation of P(DMA-DEAN) Hydrogel: 2.5 g DMA, 2.5 mg BIS (0.1 wt% of DMA), 25 mg APS (1 wt% of DMA) and certain amount of DEAN (0.5, 0.1, or 0.2 mg mL^{-1}) were first dissolved in 10 mL deionized water. After the solid was completely dissolved, 10 µL TEMED was added and oscillated rapidly before being transferred into molds. After about 8 h, the P(DMA-DEAN) hydrogel was put into acetone to stir and dehydrate as well as remove the unreacted monomers.

Preparation of P(DMA-DEAN)/P(SMA-9-ANA) Organohydrogels: In order to obtain organohydrogels displaying various colors, as-prepared gels with different amounts of DEAN were respectively immersed in oily precursor solutions containing 20 mL SMA, 20 mL ethanol, 300 µL EGDMA, 0.2 g DEAP, 40 mg 9-ANA. Being kept in the dark for 12 h in a 35 °C oven, the gels were sandwiched between two pieces of quartz glasses and irradiated under UV light for 30 min (365 nm, 50 W). Finally, prepared organohydrogels were put into ethanol for getting rid of unreacted monomers and then swelled in deionized water. Then, the swollen organohydrogel was taken out and dried in air. Finally, the drought organohydrogel was put in an 80 °C oven (24 h) for converting dimer-form to unimer-form of 9-ANA.

Information Loading, Decryption Procedure, and Erasing: The obtained organohydrogel was covered by a black PDMS mask and then irradiate under 365 nm UV lamp for about an hour for loading precise information. The encrypted information can be further encrypted by fixing special shapes sandwiched by two pieces of ice block for 10 min. The encrypted information can be read by a two-step method. First, the shape-fixed organohydrogel unfolds in the 60 °C oven for 15 min; then, the unfolded organohydrogel are read under 254 nm UV lamp. The erasing procedure was that the encrypted organohydrogel was illuminated where sandwiched in two 254 nm UV lamps about 2 h.

Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

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Conflict of Interest

The authors declare no conflict of interest.

Data Availability Statement

The data that support the findings of this study are available in the supplementary material of this article.

Keywords

anthracene, anti-counterfeiting, fluorescent organohydrogel, information encryption, shape memory

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