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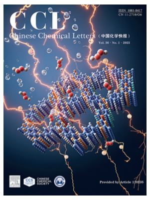

Constructing diverse switchable circularly polarized luminescence *via* a single azobenzene polymer film

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ABSTRACT

Recently circularly polarized luminescence (CPL) materials have attracted significant interest. Introducing reversible dynamic property to these materials has been a key focus in cutting-edge fields, such as in high-level information encryption. Here, we provided a novel and general strategy involving handedness-selective filtration and ground-state chiral self-recovery (CSR) in double film system to address this issue. Based on this strategy, we achieved CPL switch through the reversible modulation of ground-state chirality including absorption and scattering circular dichroism (CD) signals over the full UV-visible wavelength range (365-700 nm) in a single azobenzene polymer (PAzo) film. More importantly, by flexibly changing the type of fluorescent films, it is convenient to achieve general excited-state CSR, that is reversible switching of full-color including ideal white (CIE coordinate (0.33, 0.33)), as well as room-temperature phosphorescent CPL. All these CPL signals without almost any intensity decay after three cycles of on-and-off switching. Experimental results indicated that the *trans-cis* isomerization and ordered rearrangement of azobenzene units in PAzo film were the fundamental reasons for realizing CPL switching. Finally, based on this system we achieved dynamic visual encryption and decryption process including multiple decryption methods. This study provides an effective method for constructing a universally applicable chiroptical switch in excited state.

With the development of technology, the world has entered the information age, and information has become one of the three major resources in the world [1]. In this context, information security issues have received more and more attention. Circularly polarized luminescence (CPL), due to its additional polarization dimension compared to common fluorescence [2], is one of the important tools [3,4] for developing information encryption and has great application prospects in the field of information security [5-7], which necessitates CPL materials to exhibit flexible dynamic control. In the CPL system, chirality and fluorescence are two indispensable factors [8-10], which are usually connected by covalent [11-14] or non-covalent [15-18] interactions to obtain CPL activity. However, because of the difficulties in accurately identifying their relationship, aggregation-induced fluorescence quenching and chirality transfer [19-21] are frequently accompanied by uncertainty. Therefore, achieving dynamic signal modulation of CPL through rational molecular or supramolecular design is still a challenge.

Furthermore, the dynamic controllability of switchable CPL [22,23] has always been a topic of mutual contradiction with its own high stability. In fact, chiral structure of the majority chiral substances is often easily disrupted by external stimuli such as light [24], heat [25], or solvents [26], leading to further decay [27,28] or loss of CPL signals [29]. Therefore, while ensuring good dynamic controllability, it is also necessary to pay attention to the stability of its own luminescence and chiral information. Chiral self-recovery (CSR) strategy [30,31] is an effective method to enhance the stability of chirality during dynamic control processes through covalent cross-linking [32]. The chirality of assemblies can be effectively stored within the cross-linking units, allowing complete self-recovery even after disruption. But currently, this strategy is limited to the modulation of ground-state chirality. In

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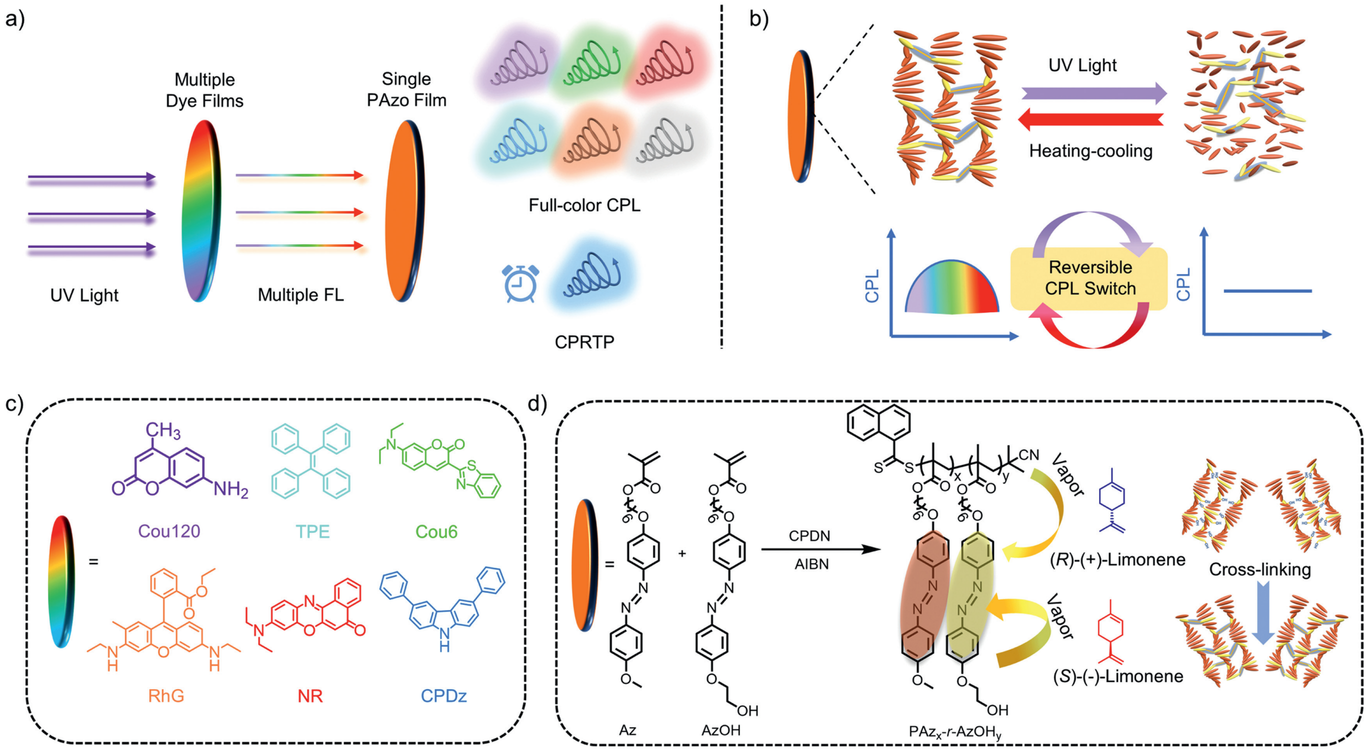

Scheme 1. Schematic illustration of the convenient strategy for constructing switchable CPL. (a) Generation of full-color CPL, white CPL and CPRTP: Ordered combination of fluorescent film (the former) and cross-linking PAzo film (the latter). (b) Self-recovery of CPL signals and construction of reversible CPL switch: Without any external chiral source, polymer chains undergo a transformation from UV-induced disorder to helical reorder through the heating-cooling treatment, and CPL signal transition from existence to silence and back to existence. (c) Chemical structural formulas and abbreviations for typically various color luminescence component used in this experiment. (d) Preparation process of the cross-linking film including polymerization reaction, chiral induction, and cross-linking fixation.

addition, recently, methods based on matching rules [33,34] to achieve CPL-activity have been developed. In this method, the chiral component and the fluorescent component are usually separated, indicating that the relationship between the two is deterministic and the fluorescent component is typically stable. Therefore, the selective filtration effect of the chiral component plays a decisive role in the dynamic control of CPL. Based on this design, we expect to achieve the same control over the excited-state chirality, namely CPL, simply by dynamically controlling the ground-state chirality while ensuring CPL stability.

Herein, we provided a new strategy involving matching rule and ground-state CSR to successfully constructed reversible full-color CPL in an achiral PAzo system, including white CPL and even circularly polarized room temperature phosphorescence (CPRTP). Moreover, the chirality of the excited-state has achieved fixed and self-recovery. As is well known, Azo chromophore exhibits non-fluorescent under usual circumstances [35,36]. However, as shown in Scheme 1, combining diverse non-chiral fluorescent films with chiral PAzo films can induce CPL due to the remarkable activity over the full UV-visible wavelength range (365-700 nm). By changing the emission wavelength of the fluorescent film, CPL covering the entire color spectrum from violet to visible red including ideal white emission as well as CPRTP can be achieved. There were hydroxyl groups at the end of side-chain PAzo to carry out acetal reaction. After completely removing the chiral source and inter-chain cross-linking, good storage of excited-state chiral information was achieved. It is worth noting that the presence or absence of chiroptical activity in the PAzo film plays a crucial role in CPL. CPL signal becomes silent when the external UV light destroys the supramolecular helix structure of the PAzo film. Thanks to the CSR property, the disrupted chiral information can be fully restored through a simple heating-cooling treatment, and the previously silent CPL signal recovered to the initial level. Based on this strategy, this switchable CPL can be used to construct reversible CPL switches, and all of these CPL signal exhibit almost no intensity decay after undergoing three cycles of "on" and "off" switches.

Referring to previous study [30], we first synthesized side-chain Azo monomers with terminal methoxy and cross-linkable hydroxyl groups spectra (Fig. S1 in Supporting information). Subsequently, we employed the reversible addition-fragmentation chain transfer (RAFT) polymerization method to synthesize side-chain Azo copolymers with appropriately proportioned hydroxyl end groups. The structure and molecular weight of the polymer were determined using ^{1}H NMR spectra and GPC curves (Fig. S2 in Supporting information). The glass transition temperature (T_g) and clearing temperature (T_i) of the polymer measured by differential scanning calorimetry (DSC) are 87 °C and 133 °C, respectively (Fig. S3a in Supporting information), which establishes the induction temperature for subsequent chiral induction experiments.

Supramolecular chiral structure of the PAzo films was induced by Limonene vapor deposition at high temperature as previously reported [30]. After investigating the effect of PAzo film thickness on chiroptical properties (Fig. S4 in Supporting information), we selected the PAzo film spin-coated three times as the subject of our study. The corresponding CD and UV-vis spectra of the PAzo films were illustrated in Fig. 1a, which performed annealing-cooling treatment in the presence of *R* or *S* limonene vapor. We conducted CD tests on the chiral film at different angles to exclude the influence of linear polarization (LD), and the CD signal remained essentially unchanged (Fig. S5 in Supporting information). The chiral induced PAzo films exhibit mirror-symmetrical Cotton effects in the CD spectra. For the chiral film induced by *S*-type limonene, the positive exciton coupling with a trough at 290

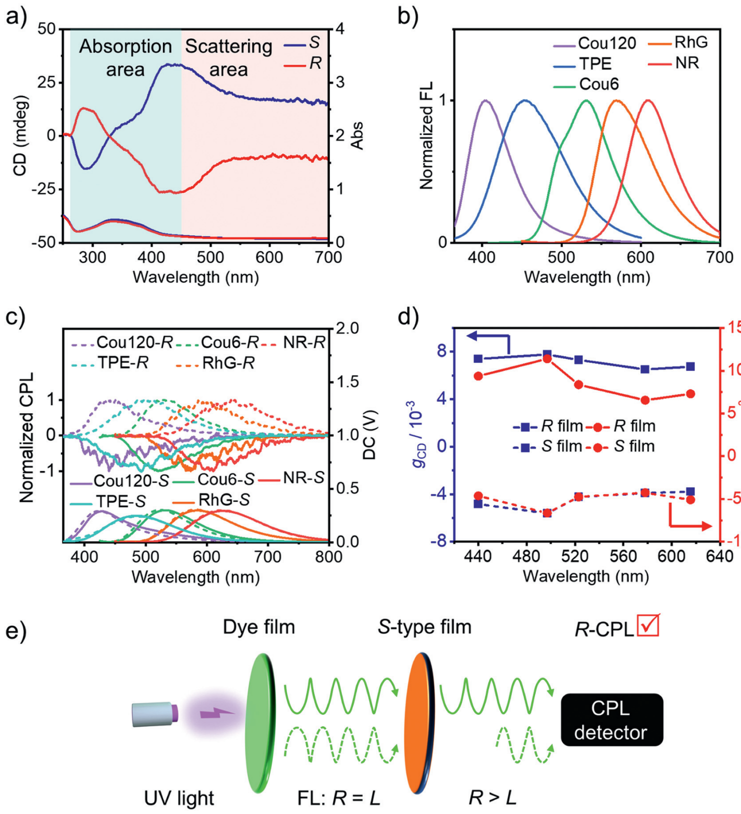

Fig. 1. (a) CD and UV-vis spectra of the PAzo film after chiral induction. (b) Normalized fluorescence spectra of various color fluorescent films. $\lambda_{ex} = 365$ nm but 320 nm for Cou120 and TPE. (c) Normalized CPL and DC spectra of various color fluorescent films orderly combined with chiral films. (d) g_{lum} values of CPL at their respective maximum emission wavelengths for various color fluorescent films and g_{CD} values for chiral PAzo films. (e) Schematic explanation of the mechanism of CPL generation.

nm and a peak at 420 nm corresponds to the main absorption peak in the UV spectrum. According to the exciton coupling theory [29,37], this positive exciton coupling indicates the formation of a right-handed helical structure in the *S*-type PAzo film. In contrast, *R*-type limonene induced a left-handed helical structure. This result demonstrates that chirality is successfully transferred from solvent vapor molecules to the Azo side-chain of polymer. It should be noted that in addition to the main absorption peak, a significant broad CD signal appears in the 420-800 nm region. This result is attributed to the circularly polarized scattering effect of the PAzo film [38], and based on this unique chiroptical active property, the general strategy for constructing full-color CPL can be realized by only using the single PAzo film.

Inspired by the unique chiroptical active property (the absorption CD [39] in the short wavelength and the circularly polarized scattering CD in the long wavelength range) of chiral PAzo films, we rationally selected five different fluorescent molecules with varying emission wavelengths (Fig. 1b), in sequence: coumarin 120 (Cou120), stilbene (TPE), coumarin 6 (Cou6), rhodamine 6G (RhG), nile red (NR). Combining with PAzo film orderly (Fig. S6 in Supporting information), they acquire CPL activity, taking the example of the Cou6 fluorescent film (Fig. 1c), CPL signals with different handedness and maximum emission were measured at 530 nm. The induced CPL signal is exactly opposite to the corresponding CD signal. Specifically, *R*-type chiral films exhibit a negative Cotton effect but a positive CPL signal, while *S*-type chiral films show a positive Cotton effect but a negative CPL signal. Similar results were obtained when other fluorescent films were tested in combination with the same chiral PAzo film. It is noteworthy that the magnitudes of CPL g_{lum} values for each color are positively correlated with the circularly polarized scattering CD magnitudes of chiral films. As shown in Fig. 1d, with the increase of wavelength, the overall trend of g_{CD} values first increases and then decreases. Correspondingly, the g_{lum} values also show a similar trend, with the blue CPL at a wavelength of 487 nm reaching its maximum ($|g_{lum}| = 1.14 \times 10^{-2}$). The result indirectly indicated that the intensity of CPL originates from the chiroptical activity of the chiral PAzo film. Thus, we have successfully achieved full-color CPL in the same PAzo film conducted.

We explored the mechanism of CPL generation. To eliminate interference, we conducted CPL tests on single PAzo films and single fluorescent films, respectively. The results show no CPL in any case (Fig. S7 in Supporting information), which indicates that CPL is the result of the light-chiral matter interaction between fluorescent films and PAzo film. Combining the photoluminescence quantum yield tests of the fluorescent films and its composite films (Fig. S8 in Supporting information), we speculate, as shown in Fig. 1e, when incident light excites the fluorescent film, it emits fluorescence with equal amounts of polarized light including left and right-handedness. When this equivalent photon irradiates the PAzo film, the circularly polarized scattering CD of PAzo films causes asymmetric filtering effect of left and right-handed CPL [29,34]. Taking the *S*-type film as an example, with a positive circularly polarized scattering CD, this PAzo film will selectively filter out left-handed CPL while having no effect on right-handed CPL. Circularly polarized fluorescence in the transmitted fluorescence through the PAzo film, thus presenting a negative CPL. The *R*-type film, similarly, exhibits a positive CPL. This effectively rules out the circularly polarized reflection effect of chiral liquid crystals [40,41]. In our previous discussion, the *S*-type film was found to have a right-handed helical structure, which should reflect right-handed CPL and transmit left-handed CPL. Similarly, the *R*-type film transmits right-handed CPL. This contradicts the experimental observations. Additionally, the transmission curve of the PAzo film further

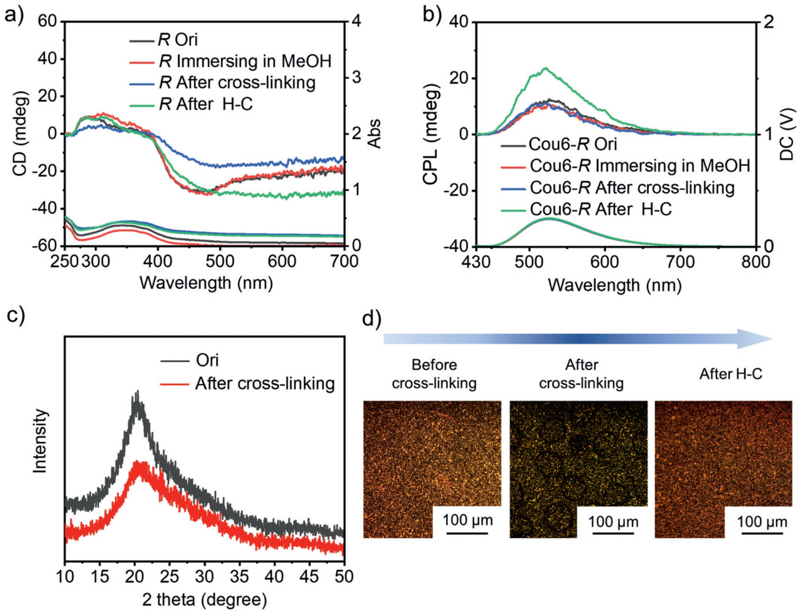

Fig. 2. (a) CD and UV-vis spectra during the preparation process of the cross-linking film ("H-C" stands for heating-cooling treatment), and (b) CPL and DC spectra combined with Cou6 fluorescent film. (c) XRD profiles of the *R*-film recorded before (black line) and after cross-linking (red line). (d) Changes in POM images of the polymer film before, after cross-linking, and after heating-cooling.

ruled out this effect (Fig. S9 in Supporting information). In summary, based on the combining of absorption CD and circularly polarized scattering CD, we have achieved the generation of full-color CPL using only *via* a single PAzo film.

Additionally, when we swap the positions of the fluorescent film and the chiral film and the CPL signal is silent (Fig. S10 in Supporting information). This result also confirms our previous speculation. Interestingly, based on the CSR strategy, we can achieve dynamic switching of the CD signal by covalently cross-linking the chiral-induced PAzo film after chiral fixation. Due to the decisive role of the CD of the PAzo film in determining the CPL, we can obtain switchable full-color CPL using the same method.

Subsequently, we tracked the changes in CD and CPL during the chiral fixing process of the PAzo film. Initially, we employed a methanol immersion method to remove residual chiral limonene from the PAzo film [42]. After immersing for 10 h, the signal of chiral limonene could no longer be detected (Fig. S11 in Supporting information), indicating the complete removal of the chiral source. During this process, the intensity of the CD of the PAzo film and the CPL of the bilayer film remained relatively unchanged (Figs. 2a and b, Figs. S12 and S13 in Supporting information), suggesting that the unfavourable dissolution immersion had no impact on the chiral structure of the PAzo film.

We performed an acetal reaction on the hydroxyl groups at the ends of the polymer to achieve chiral fixing. X-ray photoelectron spectroscopy (XPS) tests indicate that the C-OH in the polymer terminal has been transformed to C-O-C, indicating chemical changes caused by cross-linking (Fig. S14 in Supporting information). However, before and after chiral fixing, both the CD signal of the PAzo film and the CPL signal of the bilayer film showed a certain degree of decrease. XRD testing indicates that after cross-linking, the peaks in the wide-angle region become lower and wider (Fig. 2c), indicating a change in the spacing between adjacent mesogens within the PAzo film. Combining polarizing optical microscope (POM) (Fig. 2d), it was observed that this decrease was attributed to cross-linking leading to the destruction of the PAzo structure. Fortunately, we achieved dual recovery of CD and CPL through a pre-treatment process involving heating and cooling of the PAzo film. The CPL signal intensity maybe showed some improvement, possibly due to the more complete structure of the PAzo film after the heating-cooling process. It is noteworthy that in this system, there is no longer any presence of chiral sources or chiral centres. Thus, we have successfully constructed full-color CPL in an achiral PAzo system and achieved fixation of excited-state chirality.

The *trans-cis* photoisomerization of Azo units has been proven to trigger the chiral "on" and "off" switching of side-chain polymers in solution [43] or film [31] states. We irradiated the PAzo film with 365 nm UV light, as shown in Fig. 3a, and its CD gradually decreased. Taking the Cou6 fluorescent film as an example (Fig. 3b), the corresponding CPL of the bilayer film also gradually decreased and completely disappeared after 30 min. After a heating-cooling treatment in the absence of any external chiral source, both the absorption CD and circularly polarized scattering CD of the PAzo film could almost recovery completely over to the initial level. This indicates that the previously almost silent full-color CPL can also be restored to its initial level (Figs. S15 and S16 in Supporting information). Based on the reversible *trans-cis* photoisomerization of Azo units, we have successfully achieved the *in situ* switchable CPL from "on" to "off" and back to "on". Experimental results demonstrate that this reversible cycling process of CPL can be repeated more than three times without almost intensity decay (Figs. 3c and d, Fig. S17 in Supporting information). From this, we have successfully constructed reversible CPL switches of full-color *via* a single PAzo film.

For the uncross-linking PAzo film, under 365 nm UV light irradiation, both its CD and the corresponding CPL of the bilayer film gradually decreased to complete disappearance. However, after the same heating-cooling treatment, neither the CD nor the corresponding CPL of the bilayer film could recover (Fig. S18 in Supporting information). This result indicates the necessity of the intermolecular cross-linking units of Azo for the *in situ* switchable CPL.

The preparation and application of white CPL devices have attracted widespread attention in the field of optoelectronic materials [44-46]. In our previous studies, we found that a combination of three monochromatic fluorophores could be used to produce white fluorescence [47]. By adjusting the mass ratio of the blue

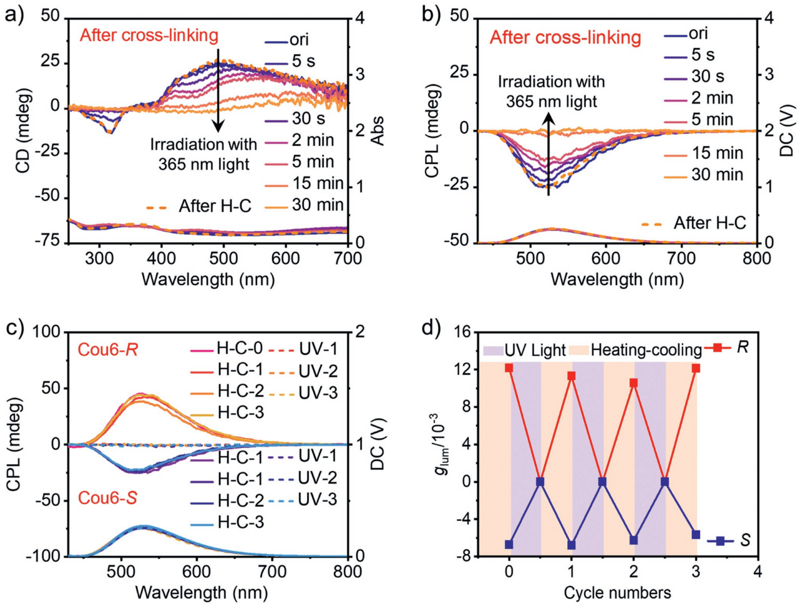

Fig. 3. (a) CD and UV-vis spectra of the cross-linking film during 365 nm UV light irradiation and heating-cooling treatments, along with (b) CPL and DC spectra combined with Cou6 fluorescent films. (c) CPL and DC spectra of Cou6 during three repeated cycles of UV irradiation and heating-cooling treatment. (d) Corresponding cycling tests of the erasure and self-recovery behavior of CPL.

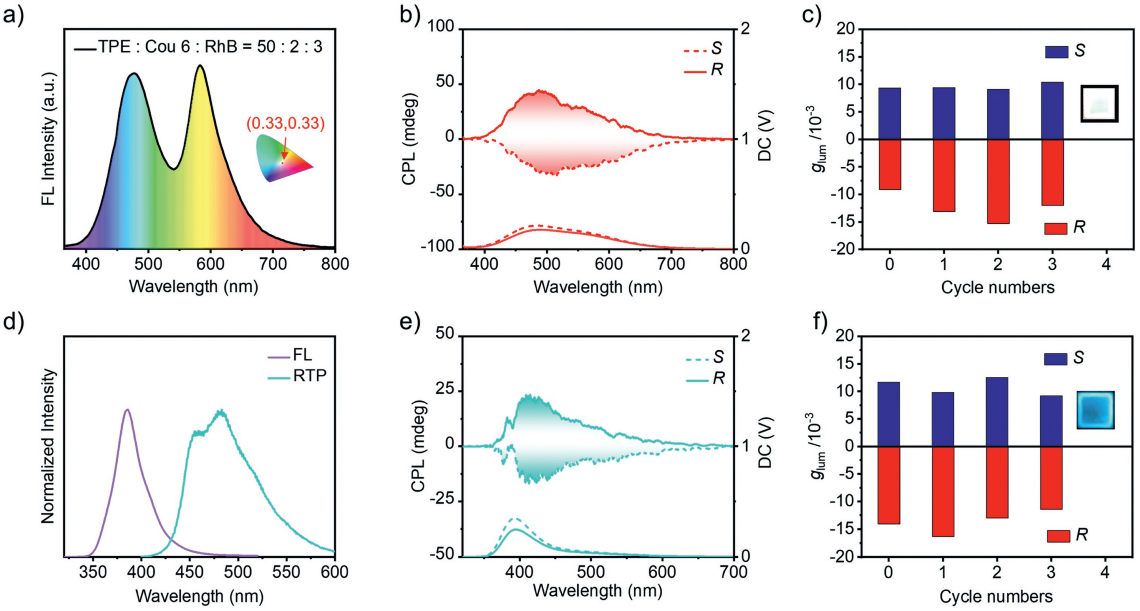

Fig. 4. (a) Fluorescence spectra of the white film and (b) CPL and DC spectra combined with the PAzo film. $\lambda_{ex} = 320$ nm. During the 365 nm light irradiation and heating-cooling treatments of the cross-linked film, (c) the g_{lum} value of white CPL and (f) the g_{lum} value of CPRTP (g_{lum} values calculated according to Fig. S19 in Supporting information) are presented. The inset in Fig. 4c shows a digital image of the white fluorescent film under 365 nm UV excitation, and the inset in Fig. 4f shows a digital image of the long afterglow of the room temperature phosphorescent film. (d) Steady-state (fast) fluorescence spectra (purple line) and delayed phosphorescence spectra (blue line) of DPCz film. $\lambda_{ex} = 272$ nm. (e) CPL and DC spectra of DPCz film combined with the PAzo film.

fluorophore TPE, the green fluorophore Cou6, and the red fluorophore rhodamine B (RhB) to 50:2:3, we prepared an ideal white fluorescent film with Commission International d'Eclairage (CIE) 1931 coordinates of (0.33, 0.33) (Fig. 4a). After combining it with the PAzo film, we successfully obtained white CPL (Fig. 4b). As expected, this white CPL also possesses the ability of self-recovery and reversible switch. After three cycles of "on" and "off" switching, its luminescence intensity and chiral signal did not show almost intensity decay (Fig. 4c), indicating that we have successfully constructed a switchable white CPL switch.

To validate the universality of this strategy and CPL switch, we attempted to combine room-temperature phosphorescent luminophore with PAzo films. Firstly, we doped of 3,6-diphenylcarbazole (DPCz) [48,49] in polyvinyl alcohol to prepare a long-lived room-temperature phosphorescent film and conducted fluorescence and phosphorescence tests. After combining it with the PAzo film, CPRTP was achieved. As shown in Fig. 4d, the steady-state (fast) fluorescence spectrum exhibited a major fluorescence emission peak at 385 nm, while the time-resolved (delayed) spectrum revealed a distinct room-temperature phosphorescent emission peak at 485 nm. Additionally, after turning off the excitation light, the persistence of the room-temperature phosphorescent film exceeded 21 s (Fig. S20 in Supporting information). After combining it with PAzo films, we successfully

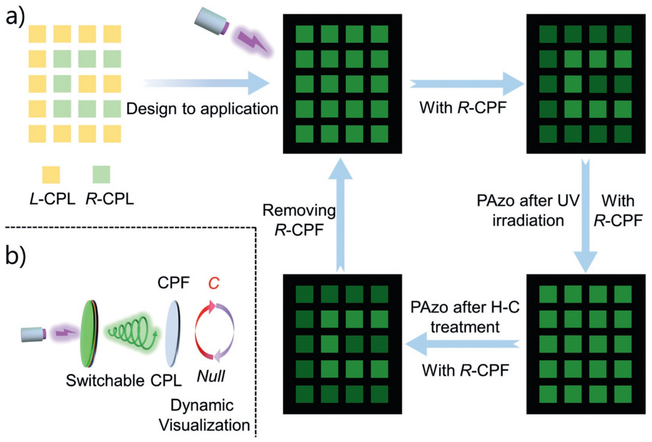

Fig. 5. Design and application of dynamic and anti-counterfeiting materials through switchable CPL. (a) Diagram illustrating the process of storing, encrypting, and decrypting pattern information. (b) Diagram illustrating the reversible change of CPL after passing through CPF under UV irradiation.

obtained CPRTP. As shown in Fig. 4e, the CPL signal after 450 nm is mainly attributed to the CPRTP. Excitingly, this CPRTP material also has chiral memory function (Fig. 4f) and can successfully construct a switchable CPRTP switch, indicating that this strategy is also applicable to room-temperature phosphorescence, with superior universality and potential.

Based on this switchable CPL, we can directly construct dynamic encryption and anti-counterfeiting materials by introducing circularly polarized filter (CPF) (see Fig. S21 in Supporting information for relevant principles). As shown in Fig. 5a, we arrange switchable CPL-active films with opposite handedness in a certain order according to the designed pattern (the letter C) and the relevant information will be encrypted and stored within it. Under UV excitation, the pattern information is encrypted and cannot be read. Subsequently, when *R*-CPF is introduced (Fig. 5b), the pattern information will be decrypted and read. After irradiation with 365 nm UV light on the PAzo film, the pattern information is re-encrypted and cannot be read. This encryption can be deciphered by the heating-cooling treatment of the PAzo film, reproducing the letter C.

In summary, we have successfully achieved diverse CPL including UV-vis full-color, white CPL and CPRTP only through a single PAzo film without any chiral source, which is attributed to the handedness-selective filtration effect of the PAzo film by the absorption and scattering CD in the wide wavelength range. Among them, the $|g_{\text{lum}}|$ value of the blue CPL could reach as high as 1.14×10^{-2}. What is more, based on the CSR strategy, we achieve chiral fixation through inter-chain covalent cross-linking of PAzo. Excited-state chiral information has also been effectively stored during this process. When UV light exposure suppresses the stored CPL signals, a simple heating-cooling procedure can fully recover them. Meanwhile, "on" and "off" CPL switches can be reversibly repeated more than three times without almost significant attenuation. Finally, we design and construct the dynamic encryption and anti-counterfeiting display by introducing CPF, which is readable for multiple times. Our proposed strategy not only presents a general way to construct CPL switch, which has important application value for high-level information storage and encryption, but also provides new insight into the light-chiral matter interaction.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

CRediT authorship contribution statement

Xiang Wang: Writing – original draft, Investigation, Formal analysis. **Qingping Song:** Investigation. **Zixiang He:** Formal analysis. **Gong Zhang:** Writing – review & editing, Funding acquisition, Formal analysis. **Tengfei Miao:** Formal analysis. **Xiaoxiao Cheng:** Investigation, Funding acquisition. **Wei Zhang:** Writing – review & editing, Supervision, Funding acquisition.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.cclet.2024.110047.

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