



# Visible Light Rewritable and Long-Lived Colors in Cholesteric Liquid Crystals: A Facile Co-Doping Strategy

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**Photoresponsive cholesteric liquid crystals (CLCs) are able to selectively reflect colors upon light exposure. Yet, it still remains a formidable challenge to realize simultaneous rewriting and long-life color in CLCs using visible light. Herein, guided by time dependent density functional theory (TD-DFT) computation, an octafluorinated binaphthyl azobenzene is synthesized to achieve the fast response and long-life color upon visible light exposure. Subsequently, based on the solubility parameter, uniform CLCs are formulated through a facile co-doping strategy. Interestingly, the CLCs change reflection colors from blue to green, red, and then into the near infrared region in seconds upon 550 nm light illumination. The completely reversible process is readily accessible upon 450 nm irradiation. More importantly, each color is independently stable for  $\approx 24$  h in the dark.**

Photoresponsive liquid crystals (LCs) have drawn extensive attention in recent years,<sup>[1–6]</sup> because new LC behaviors can be enabled spatially and temporally upon light exposure. In particular, the visible light-responsive cholesteric LCs (CLCs) have attracted numerous cutting edge efforts,<sup>[7–9]</sup> since the reflection colors are readily modulated and controlled using less hazardous, deeper tissue-penetrating, less scattering, larger window of available wavelength than the ultraviolet irradiation.<sup>[10,11]</sup> These efforts are not only of fundamental

significance to mimic the light-triggered chirality change in nature, but also promise advanced color display and information storage applications. Visible light-responsive CLCs are typically comprised of visible light sensitive chiral dopants (e.g., binaphthyl azobenzene) and nematic LC host. Binaphthyl azobenzenes are extensively investigated since they are able to afford a helical twisting power (HTP) up to  $304 \mu\text{m}^{-1}$ .<sup>[12]</sup> To boost the visible light-response capability, azobenzenes have been functionalized with methoxy, fluorine, chlorine, and other groups.<sup>[13–15]</sup> Due to such functionalization, the  $n$  and  $\pi^*$  orbital energies and distributions for the *cis*-isomers can be distinguished from those of *trans*-isomers.<sup>[16,17]</sup> More electron-

withdrawing groups in one azobenzene-derivative are generally able to significantly decrease the  $n-\pi^*$  transition gap and then to offer much faster visible light response and higher *cis*-configuration stability.<sup>[17,18]</sup> In particular, fluorinated azobenzene-derivatives are reported to show outstanding advantages in boosting the visible light response capability and the *cis*-configuration stability.<sup>[17,19]</sup> The small radius of fluorine atom helps maintaining the conjugated planar geometry of azobenzene and the large electronegativity of fluorine atoms displays a strong electron-withdrawing effect.<sup>[20–22]</sup> Recently, Yu and co-workers elegantly linked the fluorinated and non-fluorinated azobenzene in one binaphthalene dopant via chemical bonding, and achieved red, green, and blue—three primary colors with a black background (RGBB)—on the basis of piecewise reflection tuning of the doped CLCs.<sup>[23]</sup> Nevertheless, to realize this fantastic effect, stringent and skillful chemical synthesis is highly required because only a low yield (<2%) for such kind of newly tristable chiral switch can be obtained after five-step reactions and subsequent purifications. Also, the *cis*-configuration of non-fluorinated azobenzene group in this switch will fast and spontaneously back to the *trans*-configuration under ambient conditions, which gives rise to the instability of the pitch and the corresponding colors of CLCs. Nowadays, it still remains a grand challenge to explore visible light rewritable and long-lived CLCs through a simple method where the color range can be varied and long-lived as demanded.

Herein, as a proof of concept, we synthesize an octafluorinated binaphthyl azobenzene (namely D-4F, **Scheme 1**) to realize the fast response and color long-living upon visible light exposures. The synthesis is easy to control through Mills

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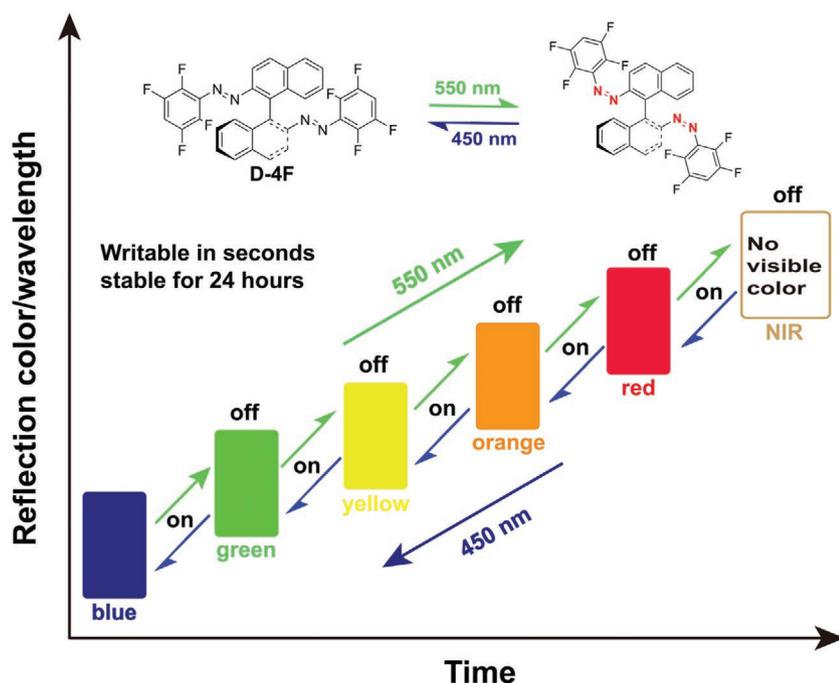
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**Scheme 1.** Illustration of the reflection color change in CLCs upon exposure to 450 and 550 nm lights. The color change due to reversible *trans*-*cis* isomerization was implemented in seconds while each color was stable in dark for around 24 h.

reaction with a yield up to 23% (Figure S1, Supporting Information).<sup>[22]</sup> In addition, we employ a solubility parameter-oriented co-doping strategy to overcome the poor miscibility of D-4F with the non-fluorinated LC host. Interestingly, our CLCs are able to change reflection colors from blue to green, red, and then into the near infrared region in seconds upon 550 nm light illumination. In addition, the completely reversible process is easily accessible upon 450 nm light illumination. More significantly, each color is stable in the dark for around 24 h.

Proton and fluorine nuclear magnetic resonance (Figures S2 and S3, Supporting Information) and mass spectroscopy (see the Supporting Information) confirm the successful synthesis of D-4F. Notably, the reaction time control is crucial to improve the yield of D-4F. D-4F can be achieved with 16 h of aniline oxidation before reacting with the binaphthyl diamine, without any production of tetrafluorinated binaphthyl azobenzene (S-4F). On the contrary, only S-4F can be obtained if the oxidation time is reduced to 4 h (Figure S1, Supporting Information).

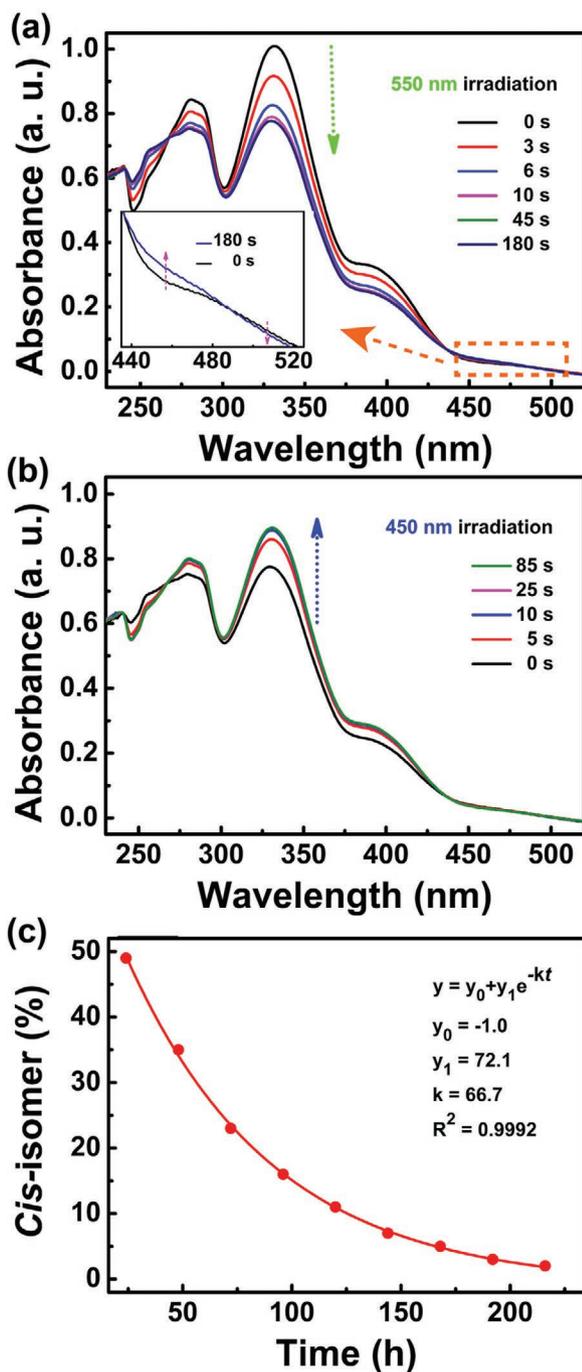
Time-dependent density functional theory (TD-DFT) calculations and natural transition orbital (NTO) analysis are implemented to predict the  $n-\pi^*$  transition behaviors of D-4F (Figures S4 and S5, Supporting Information).<sup>[24–27]</sup> A dominant visible absorption at  $\approx 500$  nm is calculated for *trans*-D-4F, due to the electron excitation into two independent but close singlet excited states (e.g.,  $S_1$  with an energy gap of 2.453 eV and  $S_2$  with an energy gap of 2.455 eV, Figure S4a, Supporting Information). In contrast, there are two distinct visible absorptions (e.g., 500 and 478 nm) for *cis*-D-4F (Figure S4b, Supporting Information) due to the electron excitation into the  $S_1$  and  $S_2$  with an energy gap of 2.477 and 2.592 eV, respectively. The results imply that the  $n-\pi^*$  transition absorption at  $\approx 478$  nm

will increase during the *trans*→*cis* isomerization. Experimental results show that the absorption increases at  $\approx 476$  nm upon exposure to 18 mW cm<sup>-2</sup> of 550 nm irradiation (Figure 1a), in good agreement with the calculation. No significant absorption change at 500 nm is noted during the *trans*→*cis* isomerization, in coincidence with calculation. Interestingly, the *trans*→*cis* isomerization process is fast with a pseudo-first-order rate of 0.144 s<sup>-1</sup> and saturated in 180 s. After separating the *trans*- and *cis*-isomers using high-performance liquid chromatography (HPLC, Figure S6, Supporting Information), we know that nearly half (49%) D-4F isomers hold the *cis*-configuration. The *cis*-configuration tends to isomerize back to the *trans*-configuration with an absorption increase in the wavelength range from 270 to 430 nm, upon exposure to 9 mW cm<sup>-2</sup> of 450 nm light (Figure 1b). This process is much faster than the *trans*→*cis* transformation, and is completed within 85 s. This is reasonable because the transformation from *cis*- to *trans*-configurations is thermodynamically favorable.

Interestingly, the *cis*-configuration of D-4F can be stable in dark for a long time.

After reaching the saturation upon 550 nm light exposure, the D-4F *cis*-configuration tends to isomerize into the *trans*-configuration to release the thermodynamic energy. Based on the HPLC analysis, we are able to trace the *cis*→*trans* isomerization (Figure 1c and Figure S6, Supporting Information). Notably, the isomerization process in dark follows the pseudo-first-order exponential decay, with a half-life time of 67 h for the *cis*-configuration. This value is over 2.5 times larger than previously reported (usually less than 27 h).<sup>[14]</sup> We expect a much faster response to visible light and a longer lifetime of *cis*-configuration in D-4F in comparison with S-4F, since D-4F holds double fluorine and azobenzene groups. Indeed, D-4F holds a three-time larger *trans*-to-*cis* isomerization rate than S-4F (Figure 1 and Figure S5, Supporting Information). In addition, only 15% of *cis*-configuration isomers can be obtained in S-4F, which is much lower than that in D-4F. The strong intermolecular hydrogen bonding (2.48 Å) and  $\pi-\pi$  interactions (3.46 Å) in S-4F are added reasons to hinder the isomerization process (Figure S7, Supporting Information).

In spite of the astonishing fast response to visible light irradiation and the long lifetime of the metastable *cis*-configuration, we encounter a poor solubility problem when doping D-4F in a nematic LC host (e.g., P0616A, Figure 2a) to form the CLCs.<sup>[28,29]</sup> Severe precipitation of D-4F is clear when cooling down the homogenous D-4F/P0616A mixture from 120 °C to room temperature. We turn to the solubility difference to understand their physical interactions. The solubility parameter difference can be estimated as  $\Delta\delta = \sqrt{\sum(\delta_{j,1} - \delta_{j,2})^2}$ , where  $\delta_j$  refers to the contribution of dispersion force ( $\delta_d$ ), polar force ( $\delta_p$ ) and hydrogen bonding ( $\delta_h$ ), respectively. It is worth noting that P0616A has a clear point at 58 °C which

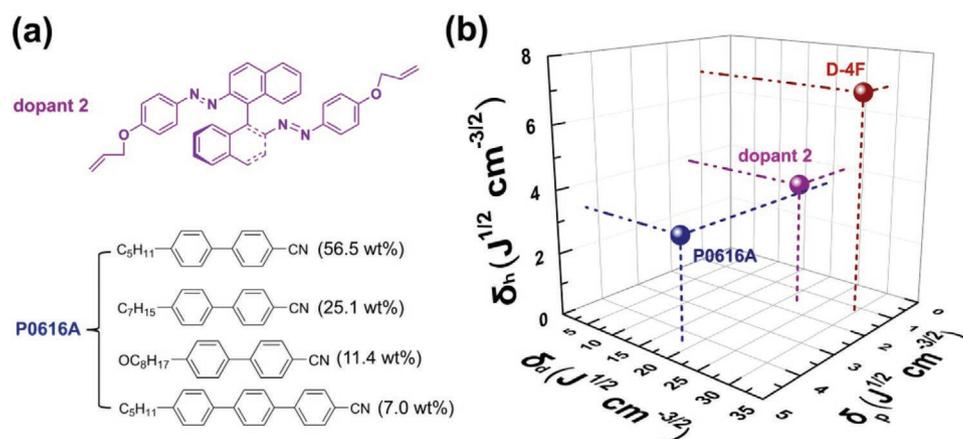


**Figure 1.** UV-vis absorption spectra of D-4F in acetonitrile upon a) 17 mW cm<sup>-2</sup> of 550 nm and b) 9 mW cm<sup>-2</sup> of 450 nm irradiations, respectively. c) plot of *cis* content versus time for D-4F in dark at 25 °C.

can hold LC phase stable even in hot weather. Moreover, the definite chemical components and percentages are in favor of calculating its solubility parameter on the basis of group contribution. The  $\Delta\delta$  value between D-4F and P0616A is calculated to be 11.09 J<sup>1/2</sup> cm<sup>-3/2</sup> (Figure 2b), implying a poor miscibility with each other. It is reported that fluorinated molecules usually exhibit poor miscibility with other

nonfluorinated molecules, due to their low surface energy, high bond energy, and low polarizability of C-F bonds.<sup>[30]</sup> On the other hand, the complete rigidity of D-4F, which is quite different from the nematic LC host bearing some flexible end groups, may be another important reason for the poor miscibility. Fortunately, solubility parameter is a powerful tool to bridge the miscibility gap between two immiscible compounds. Since  $\delta_d$ ,  $\delta_p$ , and  $\delta_h$  are 29.58, 1.15, and 6.93 J<sup>1/2</sup> cm<sup>-3/2</sup>, and 19.63, 4.56, and 3.42 J<sup>1/2</sup> cm<sup>-3/2</sup> for D-4F and P0616A, respectively, the dispersion force dominates the solubility difference. Thus, we expect a boosted miscibility after incorporating a second chiral dopant which holds a  $\delta_d$  between 19.63 and 29.58 J<sup>1/2</sup> cm<sup>-3/2</sup>. To demonstrate a proof of concept, we employ a non-fluorinated binaphthyl azobenzene (e.g., dopant 2, after D-4F, Figure 2a) reported in our recent work,<sup>[31]</sup> with a synthesis yield of 60%. The dopant 2 without fluorine groups holds a  $\delta_d$ ,  $\delta_p$ , and  $\delta_h$  of 21.82, 1.56, and 3.93 J<sup>1/2</sup> cm<sup>-3/2</sup>. Thus, it displays improved miscibility with both P0616A ( $\Delta\delta$ : 3.75 J<sup>1/2</sup> cm<sup>-3/2</sup>) and D-4F ( $\Delta\delta$ : 8.33 J<sup>1/2</sup> cm<sup>-3/2</sup>). Obviously, the dopant 2 with *S*-chiral configuration can act as a bridge compound to alleviate the solubility parameters mismatch between P0616A and D-4F. More importantly, the dopant 2 exhibits a high molar HTP (55.2  $\mu\text{m}^{-1}$ ) in P0616A.<sup>[32]</sup> In addition, the dopant 2 needs indispensable UV light for its *trans*→*cis* isomerization, so we expect that it has rare impact on the isomerization of D-4F upon visible light irradiations and the stabled *cis*-isomer of D-4F. Thus, more dopant 2 is expected not to deteriorate the visible light response capability of D-4F based CLCs. Fortunately, although the P0616A/D-4F mixture cannot form any CLCs, the ternary mixture of dopant 2 (up to 20 wt%), D-4F (up to 12 wt%), and P0616A is homogenous and stable even for more than one year after cooling down to room temperature. This solubility parameter-oriented co-doping approach can be widely applied to other dopants (Figure S8, Supporting Information). Furthermore, this approach is much easier to implement as compared to approaches that involve covalent bonding.

Visible light rewritable CLCs can be obtained by co-doping D-4F and dopant 2 into P0616A. Herein, parallel rubbed cell (purchased from E.H.C.) coated with planar polyimide (PI) layer in the inner surfaces was used for generating a uniaxially aligned CLC. As expected, when fixing the dopant 2 content at 12 wt% while increasing the D-4F content from 2 to 4, 6, and 9 wt%, consecutive blue, green, yellow, and red colors are reflected (Figure S9, Supporting Information). The increased reflection wavelength indicates a decrease of HTP by D-4F. Since both the dopant 2 and D-4F hold the identical *S*-chiral configuration, the decreased HTP is primarily caused by the poor miscibility of D-4F with the LC host. Small aggregation may disturb the uniform helical structures. Thus, the formulation with 2 wt% D-4F and 12 wt% dopant 2 in P0616A is utilized to understand the visible light responsive reflection. Upon exposure to 27 mW cm<sup>-2</sup> of 550 nm light, the reflection pattern changes from blue to green, orange, red, and eventually colorless within 60 s (Figure 3a). In other words, we can take advantage of a simple codoping strategy to achieve RGB three primary colors in convenience by adjusting the content of both dopants and the exposure time of visible lights. UV-vis

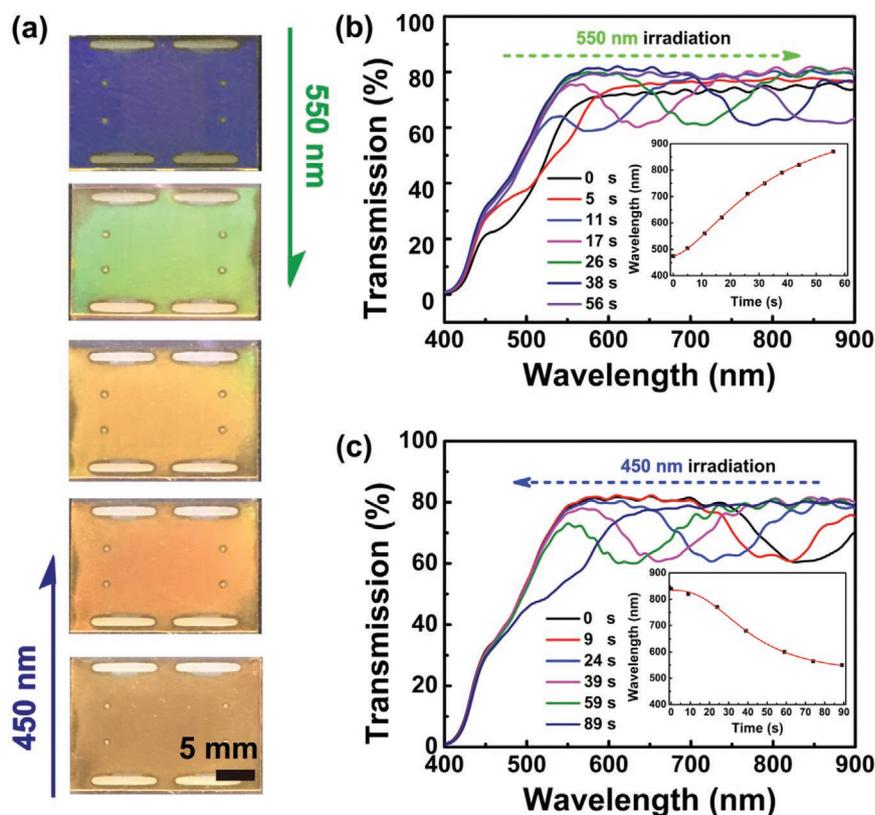


**Figure 2.** a) Chemical structures and b) theoretical solubility parameters of D-4F, P0616A, and dopant 2.

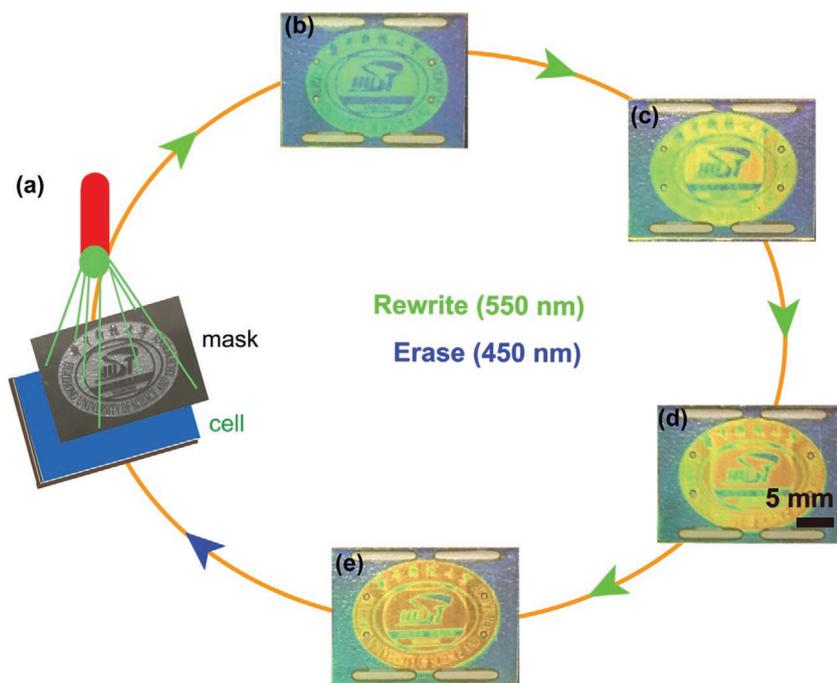
transmission spectra confirm the reflection wavelength change from 560 nm visible to the  $\approx 1 \mu\text{m}$  near-infrared (NIR) region (Figure 3b). The red shift of reflection color and wavelength are caused by the decreased HTP during the *trans*→*cis* isomerization.<sup>[32]</sup> Interestingly, this process is fully reversible. For instance, upon exposure to  $15 \text{ mW cm}^{-2}$  of 450 nm irradiation, the reflection color goes back to blue (Figure 3c). According to the quantitatively time-dependent reflection wavelength upon 550 and 450 nm irradiations, one specific color can be obtained with a specific exposure time (insets of Figure 3b,c).

The fast response of our CLCs to visible light and their outstanding thermodynamic stability promise visible light rewriting and color long-living. As a proof of concept, we use the 550 nm light to pattern letters on the CLCs through a mask of “HUST” (Figure 4a). Before irradiation, blue color reflection is clear. When increasing the exposure time, the irradiated region turns to green, yellow, red, and eventually colorless (Figure 4b–e). The blocked region by mask remains blue. It is noteworthy that the patterned images readily identifiable by the naked eye even after 122 h in dark at room temperature (Figure S10, Supporting Information). Namely, it takes about 5 days for the recovery of blue color from NIR reflection, that is, each color range is stable for an average period of 24.4 h in dark under ambient conditions. The thermodynamic stability of our visible light responsive CLCs is much better than those in literatures.<sup>[12,23,33]</sup> In addition, the exposed patterns are easy to erase by 450 nm light and are rewritable by 550 nm light, showing at least five consecutive cycles without

fatigue. The stability issue of the CLC film in bright condition is also conducted due to the system is not self-illuminating. Upon the incandescent light ( $\approx 0.02 \text{ mW cm}^{-2}$ ) irradiation, the blue color red-shifted to red color within 45 h (Figure S11, Supporting Information). However, the color of “HUST” barely



**Figure 3.** a) Reflection colors, and corresponding transmission spectra of the ternary mixture (2 wt% D-4F, 12 wt% dopant 2, and other P0616A) confined in a  $5 \mu\text{m}$  thick parallel cell upon b) 550 nm and c) 450 nm irradiations, respectively. The background was black. The light intensities were 27 and  $15 \text{ mW cm}^{-2}$  for 450 and 550 nm, respectively. The inset curves in (b, c) show the time-dependent reflection wavelength of CLCs upon 550 and 450 nm irradiations.



**Figure 4.** a) Illustration of rewriting patterns on our visible light responsive CLCs (2.0 wt% D-4F, 12 wt% dopant **2**, and other P0616A). The rewriting was implemented using 550 nm light. The image contrast increased clearly when increasing the irradiation time from b) 5 s, c) 17 s, d) 32 s to e) 54 s. The sample thickness was controlled to be 5  $\mu\text{m}$ . Dark background was used to take the picture. Longer exposure time upon 550 nm resulted in a red-shift of the reflection color and visible transparency. The patterned images were easy to erase by 450 nm light and rewritable by 550 nm light.

displayed any change until merged. As shown in Figure 1a, the isobestic point in UV-vis spectra for D-4F is 490 nm. The wavelength range of visible lights (>490 nm) for *trans*→*cis* isomerization of D-4F is broader than that for *cis*→*trans* isomerization. As a result, the *cis*-isomer dominates the photostationary state in bright condition, which is very different from the case in dark. It is obvious that the non-fluorinated dopant **2** has rare influence on the reversible *trans*-*cis* isomerization of fluorinated D-4F switch only upon visible light irradiations.

In summary, a visible light responsive axially chiral dopant octafluorinated binaphthyl azobenzene (D-4F) was selectively synthesized through the controlled Mills reaction with a high yield. D-4F dissolved in acetonitrile exhibited a fast response to 550 nm light with an isomerization rate of 0.144  $\text{s}^{-1}$  during *trans*→*cis* isomerization. The *cis*-isomer of D-4F showed a long thermodynamically half-life time of 67 h in dark. A 450 nm irradiation could significantly accelerate the *cis*→*trans* isomerization. Subsequently, by employing a solubility parameter oriented co-doping strategy, we successfully achieved visible light rewritable and long-lived CLCs based on the D-4F and a non-fluorinated chiral dopant. The rewriting could be completed in seconds, while the long-lived colors were able to be stable for around 24 h in the dark under ambient conditions. These findings open up a myriad of opportunities to explore visible light response CLCs for meeting the complex requirements of active realms from color display to information storage and optical switching.

## 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.

## Keywords

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