



Evaluation of Photoswitching Properties for Hockey Stick-Shaped Mesogens Bearing Azo Benzene Moieties

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In this paper, we report the photoresponsive behavior of hockey stick-shaped mesogens bearing azo wing with different terminal alkoxy chains at one terminal end. Except for the compound E16, which exhibits SmC along with nematic phase, all other mentioned compounds exhibit nematic phase alone. Influence of chain length on the photophysical properties were investigated using UV-Vis spectroscopy. It is observed here that influence of chain length is negligible on thermal back relaxation time. Spectroscopic investigation with variable intensities of UV light studies reveals that reverse *cis-trans* isomerization process was inversely proportional to the intensity of illuminated light. The present study also reveals that the structure-property relationship plays a dominant role on shape anisotropic structures. A spectroscopic study of the solid sample using guest-host mixture was also carried out and the compilation of results forecast these mesogens as ideal candidates for optical storage devices.

Keywords: azobenzene, photoisomerization, optical storage devices, thermal back relaxation, hockey stick

INTRODUCTION

The chromophore moiety, such as azobenzene, is an interesting functional group owing to their reversible photoisomerization between the more stable *trans* to metastable *cis* isomers upon irradiation with UV or visible light. This property of photoisomerization could bring about important changes in properties of the azobenzene chromophore, such as a large change in the molecular shape anisotropy and dipole moment. These changes could be imparted to liquid crystals when azobenzene is a part of their structure and even makes the mesogens more interesting and drives towards the applications [1–7]. Also, it is reported that photoisomerization effects are large in the case of liquid crystalline systems composed of azo substituted molecules when compared to azo-dye-doped systems [8–12]; azo-substituted LCs use light as a powerful external stimulus to control the molecular geometry and this subtle change is capitalized upon in optical storage technology. The aforesaid azobenzene undergo *trans* (*E*) to *cis* (*Z*) isomerization when illuminated with UV light of an appropriate wavelength and the isomers could switch from one state to the other with a particular wavelength of light corresponding to the energy gap of $\pi-\pi^*$ or $n-\pi^*$ transition. Upon illuminating with UV light (~360 nm) the energetically, more stable *trans* form of -N=N- linkage gets converted into a *cis* form. The *cis* form of -N=N- linkage can be converted back to the *trans* form either by illuminating with visible light (~450 nm) or in the “dark” by a process known as thermal back relaxation. This photoisomerization can bring about the phase transition in liquid crystalline system

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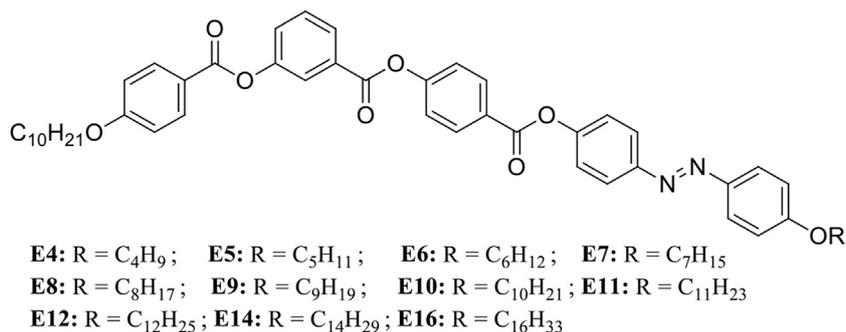


FIGURE 1 | Molecular structure of the HSLCs (**E**) compounds [18].

TABLE 1 | Phase Transition temperatures (°C) of the mentioned compounds while cooling.

Compound	R	Cooling
E4	C ₄ H ₉	139.6 N 125.1 Cr
E5	C ₅ H ₁₁	134.0 N 122.0 Cr
E6	C ₆ H ₁₃	136.3 N 116.2 Cr
E7	C ₇ H ₁₅	130.6 N 114.1 Cr
E8	C ₈ H ₁₇	135.2 N 112.1 Cr
E9	C ₉ H ₉	132.8 N 113.0 Cr
E10	C ₁₀ H ₂₁	130.5 N 111.8 Cr
E11	C ₁₁ H ₂₃	133.7 N 117.5 Cr
E12	C ₁₂ H ₂₅	132.3 N 115.7 Cr
E14	C ₁₄ H ₂₉	132.8 N 115.7 Cr
E16	C ₁₆ H ₃₃	130.0 N 125.6 SmC 119.7 Cr

due to the occurrence of ordering and disordering during this process. The time taken for the phase transitions to occur via this isomerization of the photoactive (azobenzene) behavior is significant from an application point of view. The time required for the conversion of *trans* to *cis* is faster since it can be controlled using UV intensity; however, the long thermal back relaxation is essential for optical storage devices [13–17].

The present investigation is focused on the photo-switching studies on a series of hockey stick-shaped (**E**) liquid crystalline azo compounds previously reported by us [18]. These compounds exhibit nematic mesophases. The liquid crystalline

system that exhibits nematic–isotropic (*N–I*) phase transitions is one of the simplest phase transitions to be brought about by photoisomerization of the photoactive chromophore and thus it is well studied in such systems. The lowering of the transition temperature, T_{N-I} , could induce an isothermal *N–I* transition. The photochemically induced transition is promising in order to develop an optical image storage system and their optical rewritable ability [19–25]. Hockey stick-shaped mesogens often exhibit unusual mesomorphic properties due to their frustration between bent-core and linear shape [26–28]. The molecular structure of the HSLCs (**E**) is shown in **Figure 1** and their complete synthesis and spectral characterization are reported [18].

EXPERIMENTAL

Photoswitching studies were performed by UV-Vis spectrophotometer using a Ocean Optics HR-2000+. The samples were prepared in chloroform solution at fixed concentration 1×10^{-5} molL⁻¹. Quartz cuvette (optical length; 1 cm) is used for the measurement of the UV-Vis spectra. The blank tests were performed in chloroform solution at room temperature ($27 \pm 1^\circ\text{C}$). Then the prepared sample was taken in cuvette and placed into the sample holder, which was irradiated with UV light at about 1 mW/cm² intensity. The

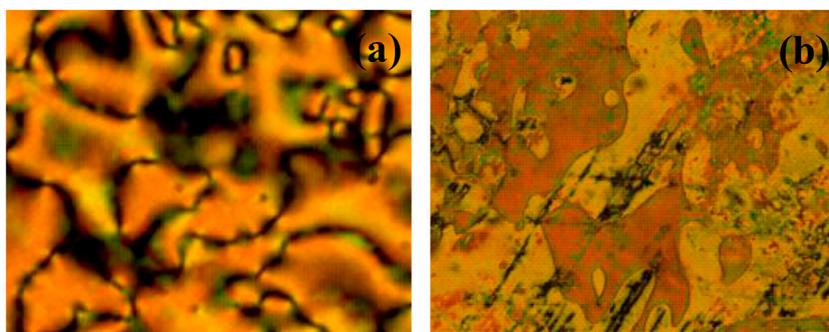
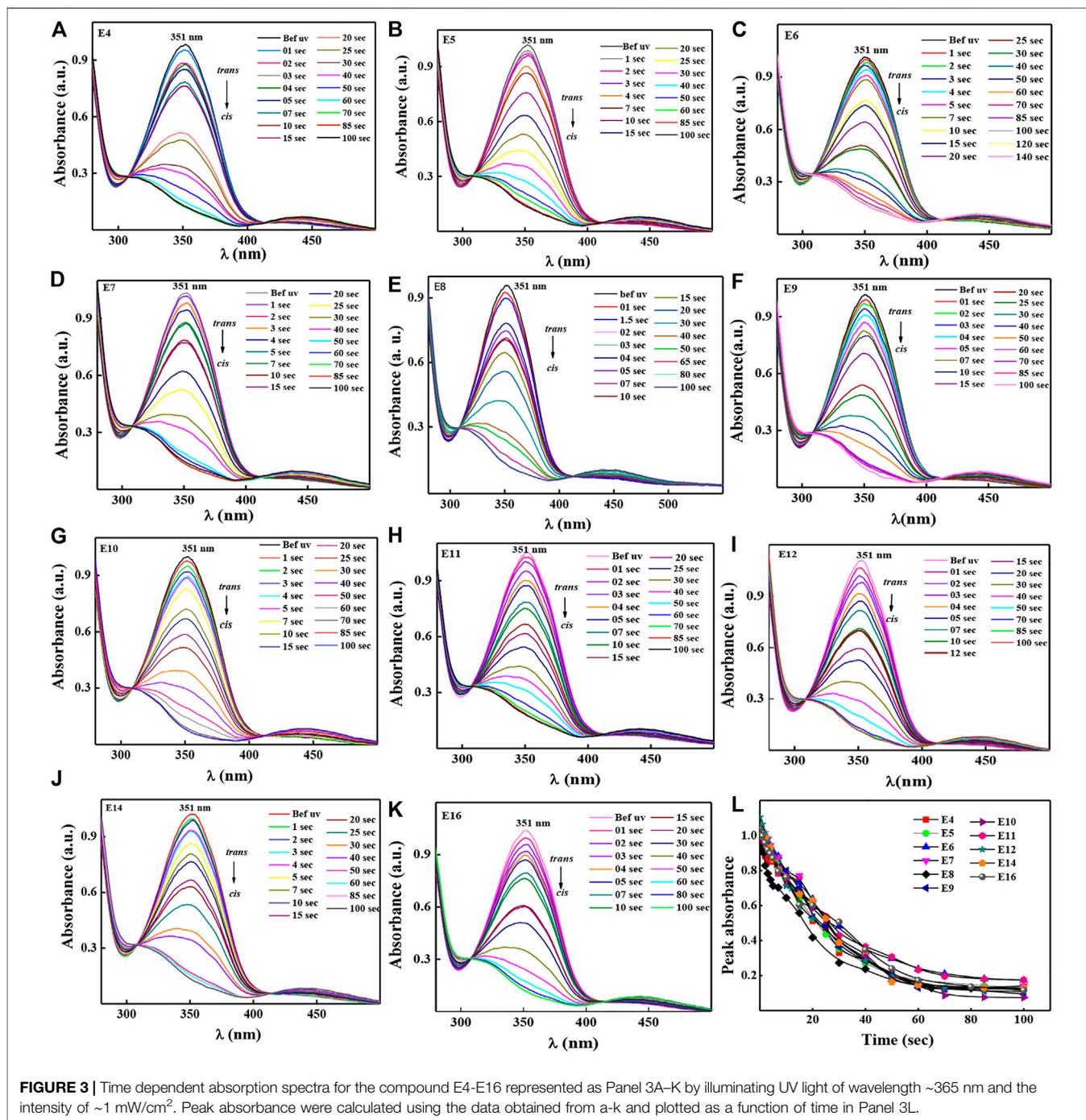


FIGURE 2 | Polarizing Optical Microscopy texture of compounds E8 and E16 on cooling from isotropic liquid under the crossed polarizers: one can see N mesophase at 128°C for compound E-8 (**A**); N mesophase at 127°C for compound E-16 (**B**). Reproduced the part of the figure from [18].



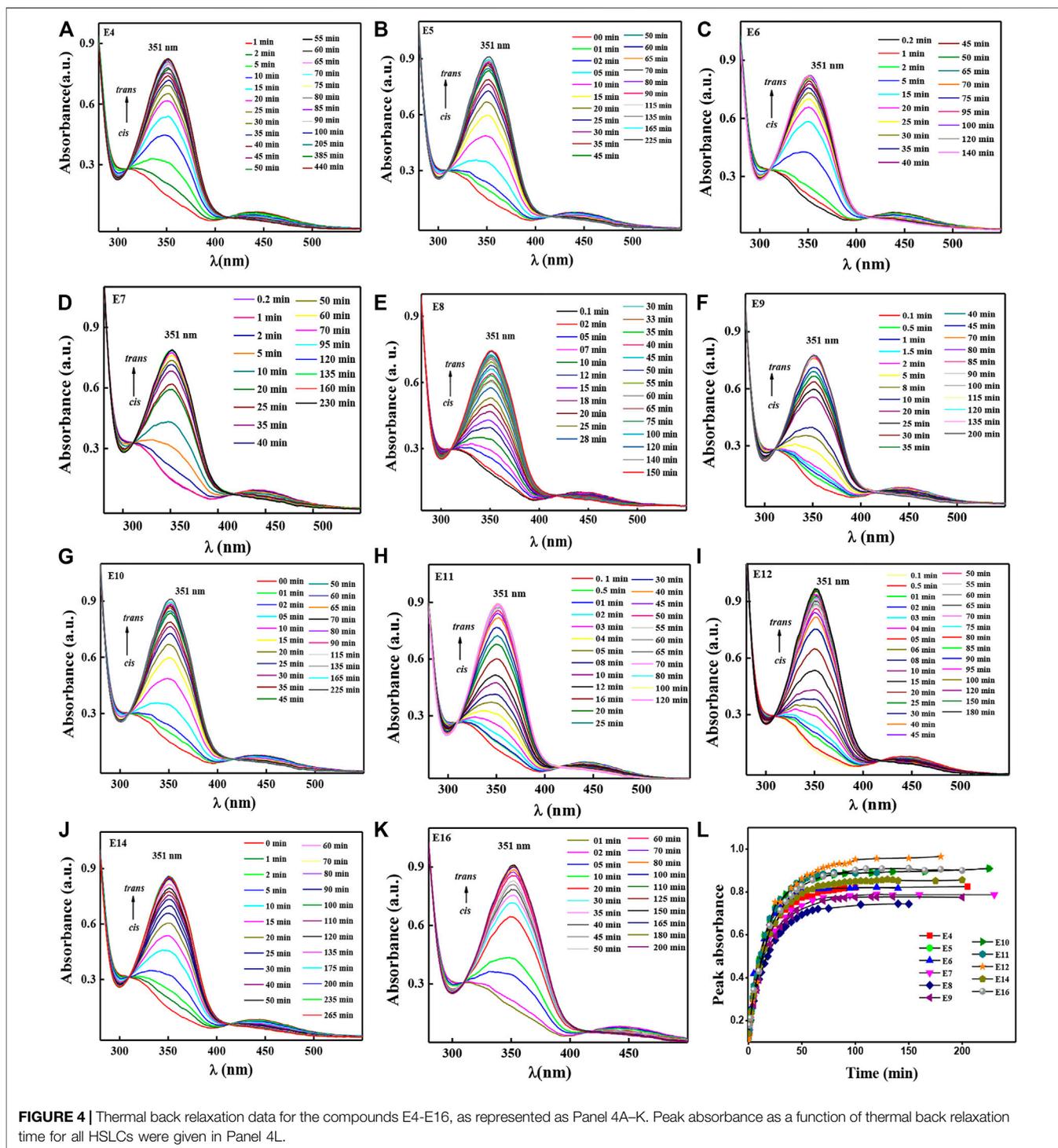
Omniscure series 2000 UV was used for illumination along with 365 nm UV filter and heat filter was equipped between the sample and the source to avoid heat radiation from the source. The intensity was calibrated using UV meter (UV513AB) and adjusted for 1mW/cm². The time dependent absorption spectra of HSLCs were recorded at time zero and at different intervals of irradiation time until photostationary state (PSS) was achieved. After PSS, the thermal back relaxation time as a function of time was determined by keeping the sample in

dark. The photoconversion efficiency for *trans-cis* isomerization at PSS were determined by using Eq. (1).

$$CE = \frac{A_{(t_0)} - A_{(t_{\infty})}}{A_{(t_0)}} \times 100\% \quad (1)$$

where *CE* is conversion efficiency and $A_{(t_0)}$, and $A_{(t_{\infty})}$ is absorbance before and after UV illumination, respectively [29].

The first-order kinetics for thermal back relaxation of *cis-trans* isomerization were measured using Eq. (2).



$$-K_{c \rightarrow t} t = \ln \frac{A_{(\infty)} - A_{(t)}}{A_{(\infty)} - A_{(0)}} \quad (2)$$

where A_t , A_0 , and A_{∞} are the absorbance with respect to peak wavelength at time t , time zero, and infinite time respectively. τ is relaxation time of *cis* isomer [30].

The solid cell has been fabricated from the series of HSLCs using precoated ITO glass substrates. The cleaned ITO glass

substrate was coated with polyimide solution and rubbed unidirectionally using a rayon cloth. The thickness of cell was maintained about $5 \mu\text{m}$ using glass spacers. The guest-host mixture was prepared by physical mixing where 5 wt% of HSLCs and 95 wt% of room temperature nematic liquid crystal E7 (now onwards called as NLC) was mixed and then the cell was filled by capillary action using guest-host mixture. The liquid crystal mixture aligns along the rubbing direction. The

TABLE 2 | Photo conversion efficiency of hockey-stick compounds during UV illumination.

Compound code	Photoconversion efficiency (%)
E4	87.67
E5	85.00
E6	80.80
E7	87.00
E8	85.00
E9	89.00
E10	90.10
E11	85.42
E12	87.60
E14	86.10
E16	89.10

spectroscopic investigation was carried out at room temperature by placing a previously prepared solid cell in sample holder between detector and source. The 1 mW/cm^2 intensity of UV light was used for illumination at different intervals of time.

RESULTS AND DISCUSSIONS

Mesophase Characterization

It is important to know the mesophase properties of the liquid crystalline compounds. It is not necessary that all the photosensitive compounds should exhibit liquid crystalline properties. One can see from **Figure 1** that when $-N=N-$ linkage is away from the central phenyl ring, we get mesomorphicity in all the mentioned compounds here, unlike in the case when $-N=N-$ linkage was directly attached to the central phenyl ring [18] where one gets non-mesomorphic nature. **Table 1** shows the phase transition temperatures and liquid crystalline mesophases for all the mentioned compounds in this study. One can also note that all the compounds exhibit nematic phase except for the compound E16, which exhibits the

SmC phase along with the nematic phase. To understand further, we took polarizing optical microscopy (POM) textures for the representative compounds, mainly on E8 and E16, which exhibits nematic phases. **Figure 2** represents POM textures of the compound E8 1) and E16 2) while cooling from the isotropic phase. It is evident from the figure that the characteristic nematic phase is present in both of them. A detailed investigation on mesomorphic effect can be found in our earlier work [18].

Photoswitching Studies

The photoswitching studies of HSLCs were performed on quartz cuvette using chloroform as the solvent. These hockey-stick shaped mesogens bearing azo wing at one terminal with constant C_{10} chain at another end were investigated for their photosensitive behavior. The azo wing has a different alkoxy chain at terminal and another end attached to center core through ester linkage. The photoswitching properties of these compounds were examined by UV light illumination at different time intervals. The wavelength of the UV light used was $\sim 365 \text{ nm}$ and the intensity used was $\sim 1 \text{ mW/cm}^2$.

A heat filter was inserted between the UV source and the sample to avoid any heat radiation arising from the source. Figures 3A–K represents the UV absorption spectra as a function of time for the respective compounds E4–E16, which was evaluated using UV-Vis spectrophotometer in solution. **Figure 3L** represents the peak absorbance data obtained from 3a–3k and is represented with respect to illumination time. The strong absorption band at $\sim 351 \text{ nm}$ in the UV region, which corresponds to the symmetry, allowed $\pi-\pi^*$ transition; $n-\pi^*$ weak transition band, meanwhile, appears in the visible region ($\lambda \sim 450 \text{ nm}$). All the compounds show similar absorption spectra and there was also no significant changes in the photosaturation time (PSS). One can see from the figure that all HSLCs took $\sim 60 \text{ s}$ to photo saturate. The maximum absorption peak gradually falls down at wavelength $\sim 351 \text{ nm}$ while UV light illumination is ON and intensity of the lower band at $\sim 450 \text{ nm}$ slightly increases. Tacitly, these changes indicate transformation of energetically

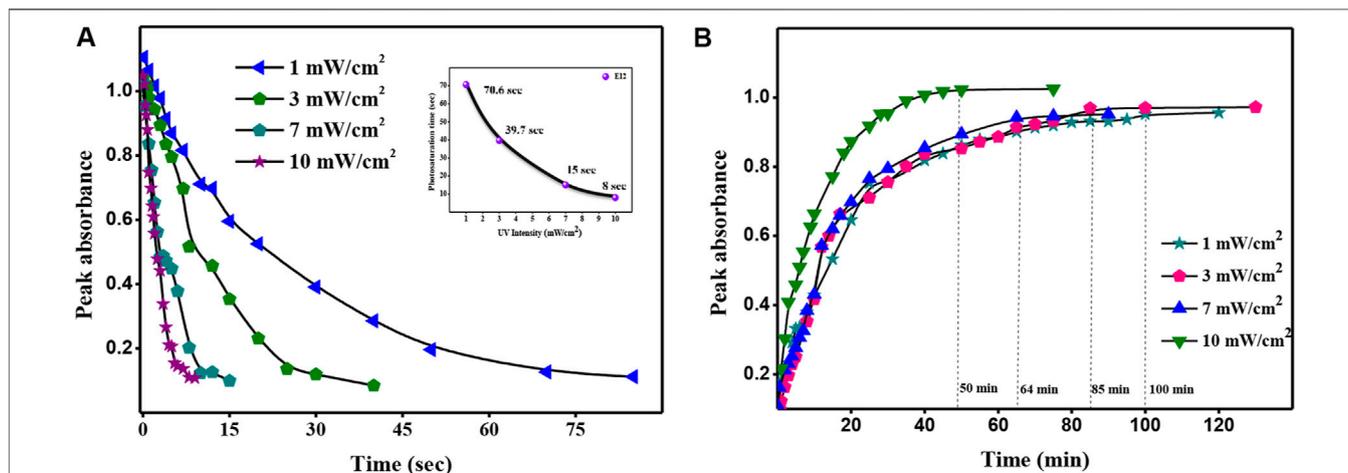
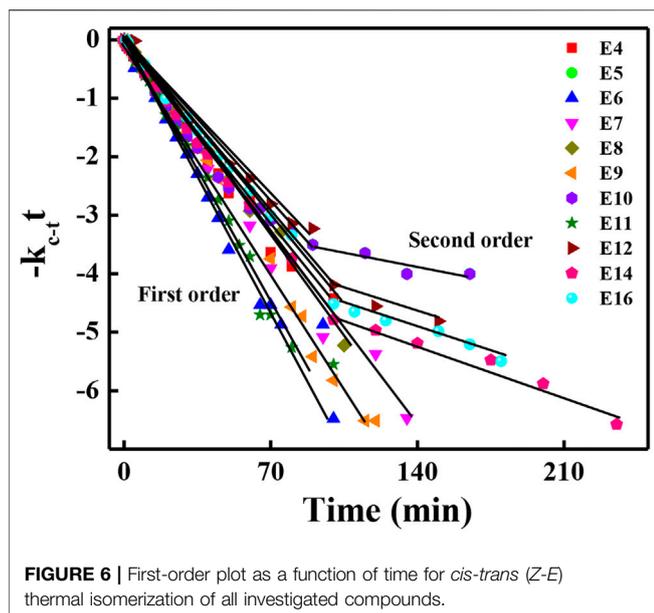


FIGURE 5 | (A) Photosaturation time versus UV intensity plot for *E-Z* isomerization and (B) Thermal back relaxation or *Z-E* isomerization of compound **E12** with different intensities.



stable *trans* isomer to unstable *cis* isomer during suitable UV light illumination. Whereas with thermal back relaxation, which occurs when UV light is turned off, energetically unstable *cis* converts to *trans* configuration without any external influence, which could be utilized in optical storage devices.

The photoactive behavior of hockey-stick compounds was determined using Eq. 1 and photoconversion efficiency was given in Table 2. The photoconversion efficiency of all investigated compounds in solution state exhibits good *E-Z* conversion upon UV illumination. In PSS, more than 85% of *trans* isomers exchanged to unstable *cis* isomer at ~70 s of UV irradiation time.

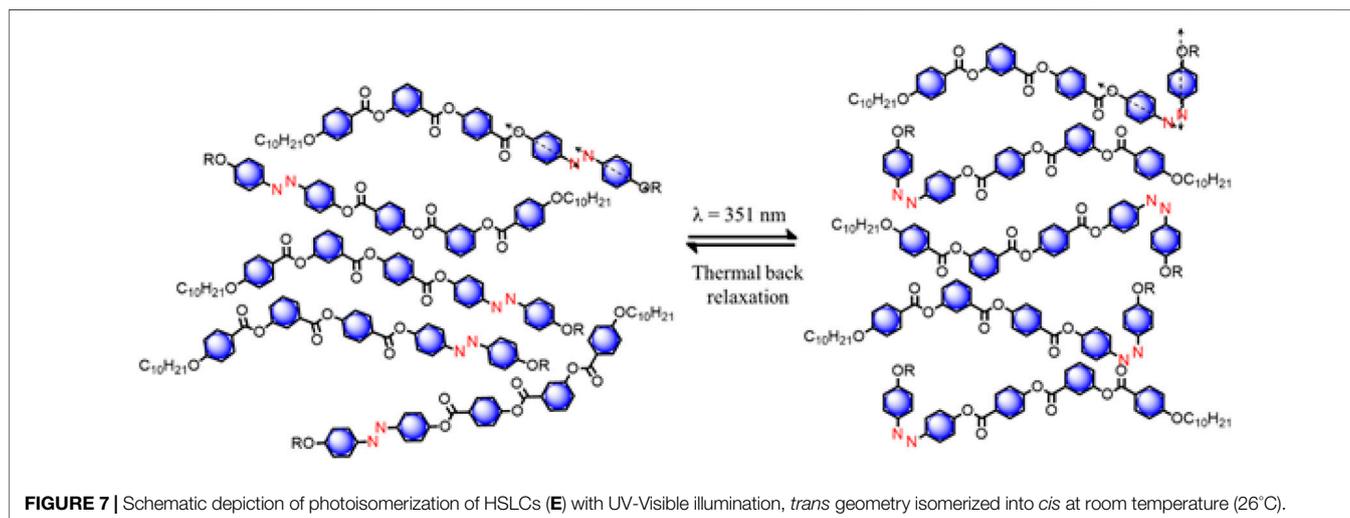
After reaching the PSS, molecules will start to reverse back to their original *trans* configuration since PSS is an unstable state. The process to relax back with temperature as the only parameter is known as the thermal back relaxation. One can also bring back

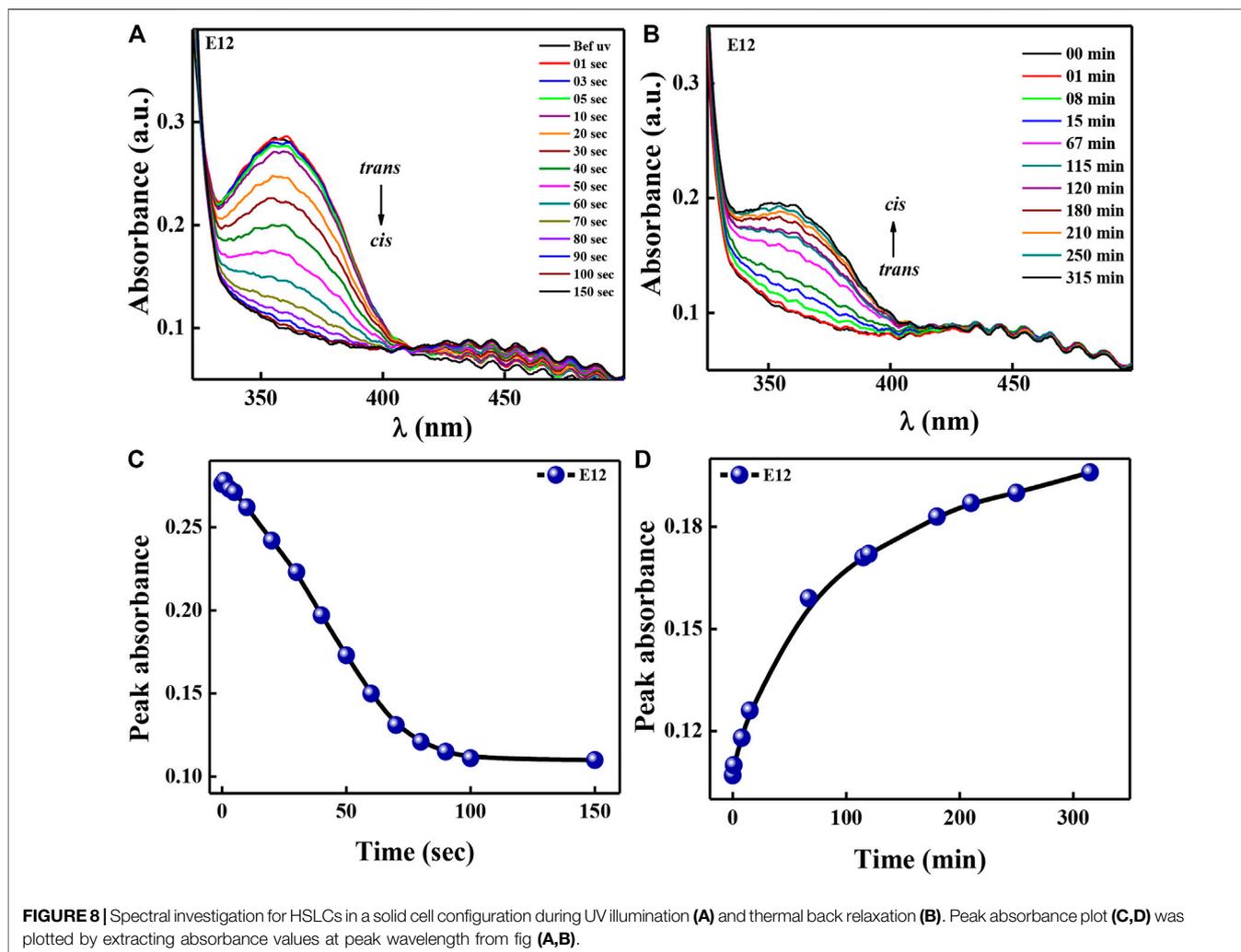
the system to its original configuration quickly by shining the wavelength of light ~450 nm.

Since the ideal situation is to get longer thermal back relaxation time, one must not use any external sources other than the temperature to return it to the original configuration. The higher the thermal back relaxation, the better the device is. Figures 4A–K represents the thermal back relaxation process after the system attains the photo saturation level. Peak absorbance as a function of back relaxation time is plotted using Figures 4A–K data and is shown in figure 4L. One can see from this figure that almost all the compounds relax back to their original *trans* configuration within 150 min.

As photo conversion efficiency is mainly dictated by the intensity since it is a controllable parameter, we did the variation of intensity vs time to understand the influence of intensity on photosaturation time. Figure 5A shows the influence of intensity on photo saturation time for compound E12. One can see that, photo saturation time is inversely proportional to the intensity of UV light. As the intensity of UV light increases, photo saturation time decreases and vice versa. The compound E12 took 70.6 s to reach its photoequilibrium state at 1 mW/cm² intensity, whereas, it takes 8 s at 10 mW/cm². Hence, one can easily notice the influence photoactive azo derivatives with respect to the intensity.

We also studied the influence of intensity with respect to the back relaxation time. As expected, the longer thermal back relaxation time was observed for lower UV intensity illuminated samples; higher intensities of UV light shined samples showed the shortest time. The observed back relaxation spectra show different thermal back relaxation times such as 100, 85, 64, and 50 min in solution at room temperature with respect to different intensities (Figure 5B). Concurrently, the observed results agreed with the previous results [16]. Therefore, intensity has a profound influence on the thermal back relaxation time. For any optical storage devices, lower intensity is ideal since it is not only cost effective but also user friendly.





Kinetic Studies

It is obligatory to determine the first-order kinetic plot for *trans-cis-trans* photoisomerization [29]. The first-order plot for unimolecular thermal *cis-trans* isomerisation obeys Eq. (2). Figure 6 shows the first-order plot which was studied by furnishing the obtained experimental data to Eq. (2) at room temperature. The indicated time region in the graph obeys first order, later, it is slightly deviated from first order to second order due to experimental conditions (since this experiment is carried out in solution). The deviation was observed from E10 to E16, which bears a higher number of the carbon atom in alkoxy chain at one end of HSLCs. As the carbon atoms increase in alkoxy group at one end of HSLCs, the thermal back relaxation slows down due to its bulkier nature. These results suggest that lower alkoxy chain bearing HSLCs obey first order, whereas the higher the number of carbon atom present in alkoxy chain (more than 10 carbon atoms) the more deviation will be shown from first order to second order with respect to molecular structure [30].

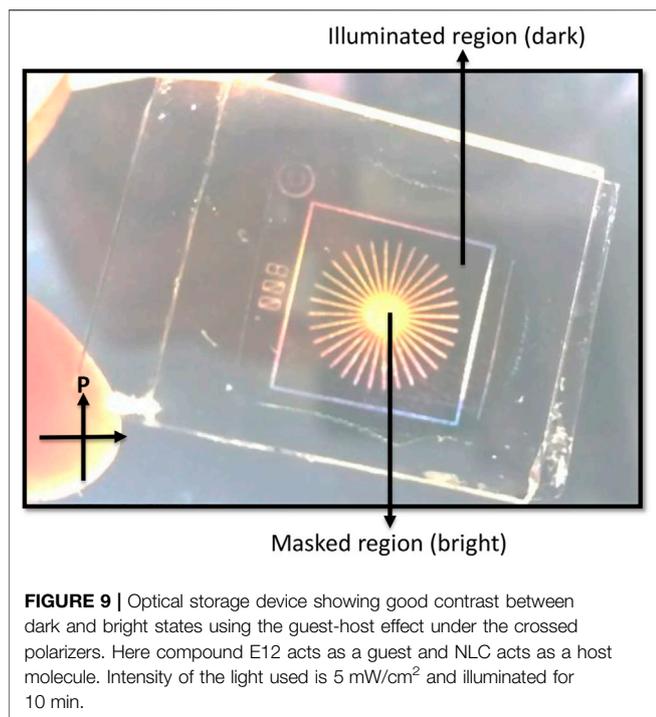
Reason Behind the Phenomena

Although the exact reason for such a shorter thermal back relaxation is not clear we speculate the following. The HSLCs,

in their *trans* configuration, are more anisotropic. But as soon as UV light of wavelength ~ 365 nm is shined on the system, a kink is observed near to azobenzene moiety due to *trans-cis* conformational change in molecules. Apart from this, the rest of the molecules remain in the same state where not much change in shape anisotropy takes place (Figure 7). Because of this, guest azo molecules are not creating any hindrance for the liquid crystal host molecules to return from *cis* to *trans* configuration in a real device.

Optical Storage Device Studies

It is ideal to check the liquid crystal device characteristics in the actual device to see the potential of the said material. Compound E12 is chosen for this purpose since almost all the compounds exhibit the same mesomorphic behavior. Liquid crystal guest-host mixture is prepared using NLC as the host and E12 as the guest in the ratio 95:5. The freshly prepared guest-host mixture was then filled in the solid cell through capillary action and placed solid cell in the sample holder. Thickness of the cell was maintained at $\sim 5 \mu\text{m}$. The UV light intensity of 1 mW/cm^2 was illuminated on solid cell with different intervals of time.



The time dependent absorption spectra for UV illumination were recorded (Figure 8A) until photosaturation state took place. Then, absorption spectra of thermal back relaxation were recorded as shown in Figure 8B. The maximum peak absorbance values are noted from Figures 8A,B and the corresponding peak absorbance graph (Figures 8C,D) was plotted as a function of time. From these results, we can infer that *trans* isomer reaches its photostationary state was 100 s during UV light illumination and returns to its original state within 5.3 h at room temperature.

To see the potential of the said material, an optical storage device was constructed by using guest-host mixture. Since all the compounds exhibit similar characteristics, we chose E12 as the representative compound. Previously prepared LC cells were filled with guest-host mixture where E12 acted as a guest and NLC acted as a host material. Suitable masks were kept above the cell and UV intensity of 5 mW/cm² was shined for 10 min. One can see the clean optical storage device under the crossed polarizers (Figure 9) with dark and bright states. The dark region corresponds to the illuminated area where the system changes from nematic state to isotropic and the bright region

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corresponds to the masked area where the system remains in the nematic state.

More investigations on changing the different functional groups and azo substitutions at different places in the structure are needed to understand more about the structure property relationships.

CONCLUSION

We report on the photoactive behavior of HSLCs with variable alkoxy chain at terminal end. All the HSLCs exhibit nematic phase at shorter intervals except for E16 which exhibits SmC as well. All HSLCs exhibit the similar *cis-trans* isomerization behavior due to similar rigid center cores. The *trans* (*E*)—*cis* (*Z*) isomerization occurs at around 70 s and thermally reversible *cis* (*Z*) to *trans* (*E*) isomerization in solution occurs ~150 min. In solid cell, photosaturation time was about 100 s, whereas it took ~5.3 h to reach its original state at room temperature. Spectroscopic investigation with variable intensities of UV light studies reveals that reverse *cis-trans* isomerization process was inversely proportional to the intensity of illuminated light. The prevailing tuning of the intensity of UV light shows promise in thermal back relaxation time.

DATA AVAILABILITY STATEMENT

The raw data supporting the conclusion of this article will be made available by the authors, without undue reservation.

AUTHOR CONTRIBUTIONS

MM synthesized the compounds and BS did the photoswitching measurements. Both MM and BS wrote part of the manuscript. GS analyzed the results and edited the manuscript. GH conceived the idea and edited the manuscript. VP analyzed the results and edited the manuscript. All authors have approved the final version of the manuscript.

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