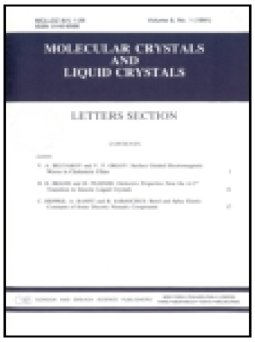

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Molecular Crystals and Liquid Crystals

ISSN: 1542-1406 (Print) 1563-5287 (Online) Journal homepage: http://www.tandfonline.com/loi/gmcl20

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To cite this article: A. R. Yuvaraj, Mohd Yusoff Mashitah & Rahman Lutfor (2016) Synthesis of bent-shaped azobenzene liquid crystals derived from resorcinol for optical storage properties, Molecular Crystals and Liquid Crystals, 631:1, 21-30, DOI: 10.1080/15421406.2016.1147328

To link to this article: http://dx.doi.org/10.1080/15421406.2016.1147328

MOLECULAR CRYSTALS AND LIQUID CRYSTALS
2016, VOL. 631, 21–30
http://dx.doi.org/10.1080/15421406.2016.1147328

Synthesis of bent-shaped azobenzene liquid crystals derived from resorcinol for optical storage properties

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ABSTRACT
The liquid crystalline and light-induced properties of resorcinol spacer based dimeric azobenzene were investigated. First, the compound was synthesized and elucidated the molecular structure by spectral analysis such as NMR, IR, and UV–VIS. polaring optical microscope studied showed that compound exhibit nematic and smectic A phases. Then, the photoisomerization effect was evaluated in solution and also in the solid state. The photosaturation occurred exactly at 18 sec whereas thermal back relaxation was observed at around 11 hr. Long duration of the thermal back relaxation is due to the presence of rigid resorcinol spacer substituted in the bent-shaped molecules. This compound is useful in the fabrication of optical storage device and molecular switches.

KEYWORDS
Azobenzene; liquid crystal; optical storage; photoisomerization; synthesis

Introduction

The information can be optically stored via light irradiation method. This process is called optical storage and the gadget is known as optical storage device. Many photo-sensitive organic compounds were used to evaluate the light-induced behavior of optical data storage device. Tacitly, the molecular structure and properties of illuminated light are responsible for the performance of optical storage device [1, 2]. The photoisomerization and molecular reorientation are depend on chromophores present in the chemical structures [3–5]. Azobenzene is a good candidate among other chromophores to evaluate light-induced effects, due to its promising light sensitivity [1–4]. The liquid crystalline and photoisomerization properties of azobenzene derivatives were investigated by many research groups [1, 2–5]. The liquid crystallinity of the bent-core molecules were first reported by Vorlander et al. [6]. However, banana-shaped liquid crystals are also interested widely, due to its unique electro-optic and photo-induced characteristics [7, 9]. Even though, banana-shaped liquid crystals have a significant role in the guest–host system. It means these liquid crystals (LC) can be mixed as a guest with commercial liquid crystal (host) to elucidate the device characteristics.

Azobenzene derivatives have delocalized electronic charge distribution between accepter and donor atom or group of atoms substituted with it. Nevertheless, *trans–cis* isomerization of azobenzene derivatives have a significant importance in the field of optics. This photophysical property plays the main role in the optical data storage. In particular, *trans–cis* isomerization is important especially in the research areas, such as holographic media [10], optical storage [11], and photoalignment of LC systems [12].

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The spacer effect is significant in the symmetric bent-core LC molecules [13]. Recently, we have reported the behavior of aliphatic and aromatic spacer incorporated in the azobenzene dimeric system [14]. It is clear that different spacers exhibit different optical activities [14]. Hence, back relaxation of azobenzene dimers are quite interested, due to its dramatic variation with respect to spacers present at the molecular center. *trans–cis* isomerization takes place in the energetically more stable *cis* isomer of the azobenzene, when light of suitable wavelength (say 365 nm) is illuminated on the molecular system [1–4, 15]. Then, the reverse process is spontaneous and it is called thermal back relaxation [2]. *Cis–trans* isomerization occurs in two ways; one is by keeping the excited trans isomer in dark place (thermal back relaxation) and other one is by shining 450 nm white light on the molecular system [16]. Among these, the thermal back relaxation is considerable because of its spontaneity.

In this report, we have synthesized bent-shaped azobenzene liquid crystals having short alkyl chains (C_6), which is so far new compound. We also have investigated the mesomorphic (LC) and light-induced isomerization properties of resorcinol-based azobenzene liquid crystals, which gave interesting photoisomerization properties. This new compound shows suitable photochromic and optical data storage properties.

Experimental

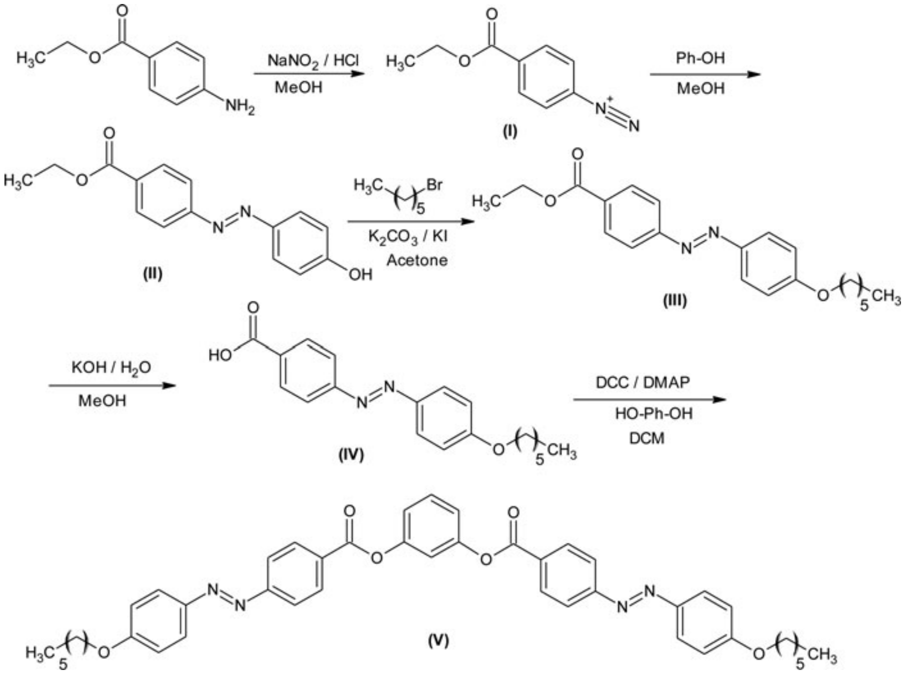
Raw materials and instruments

Ethyl-4-aminobenzoate (Fluka), sodium nitrite (Aldrich), hydrochloric acid (Fluka), sodium hydroxide (Fluka), methanol (Fluka), phenol (Aldrich), 1-bromo hexane (Fluka), potassium carbonate (Aldrich), potassium iodide (Aldrich), acetone (Fluka), potassium hydroxide (Aldrich), 1, 3-dicyclohexylcarbodiimide (DCC) (Fluka), 4-(N, N-dimethylamino)pyridine (DMAP) (Fluka), dichloromethane (Fluka), Resorcinol (Aldrich). Acetone was dried over phosphorus pentoxide (Fluka) and dichloromethane was dried over calcium hydride (Fluka) and distilled out before the use. Other solvents and chemicals were used as such.

Instrumental details are as follows: IR spectra were recorded using a Perkin Elmer (670) FTIR spectrometer. ^{1}H NMR (500 MHz) and ^{13}C NMR (125 MHz) were recorded in Bruker. The photoswitching study was performed using UV–Visible spectrophotometer from Ocean Optics (HR2000+). For photoswitching studies in solutions, the compounds were dissolved in chloroform at fixed concentration of $C = 1.1 \times 10^{-5}$ mol L^{-1}. Photoisomerization of these compounds were investigated by illuminating it with an OMNICURE S2000 UV source equipped with a 365 nm filter and heat filter was used to avoid unwanted heat from the radiation. The intensity used for measuring E/Z is around 5.860 mW/cm^2 measured using UV meter. The photoswitching behavior of this compound was studied by shining UV light of suitable wavelength. Then, mesomorphic property was investigated using polarized optical microscopy (Nikon, Olympus BX 51 POL) along with linkam hotstage. Although both heating and cooling cycles were measured using DSC by NETZSCH DSC214 Polyma. The X-ray diffraction (XRD) was measured using Cu-Kα radiation with $\lambda = 1.54$ Å and rotation anode generator of 4,000 W (Rigaku Ultrax-18) connected with graphite crystal monochromator. The sample was heated to obtain the mesophase in Hampton research capillaries with 0.5 mm diameter.

Synthesis and elucidation

The resorcinol spacer substituted azobenzene dimer (**V**) can be synthesized by five steps. First, 4-ethyl aminobenzoate was diazotized using sodium nitrite in the presence of acid medium.



Scheme 1. Synthesis of bent-shaped azobenzene liquid crystals.

Then, the diazonium ion (**I**) produced in the first step was coupled by phenol to get **II**. Next, free hydroxyl group of the compound **II** was methylated with bromo-alkene in the presence of mild basic medium. On the other hand, the substituted ester group was hydrolyzed using potassium hydroxide and water. Lastly, two molecules of **IV** were coupled with one molecule of resorcinol by DCC coupling reaction. The synthetic strategy is given in Sch. 1.

General procedure for diazotization reaction (II)

The solution of 4-ethyl amino benzoate (7.59 g, 46.00 mmol) was prepared by methanol (40 mL) and cooled to 2°C. 25% HCl was added dropwise to the solution at 2°C. The solution of sodium nitrite (3.03 g, 44.6 mmol) was prepared in water and added to the reaction mixture dropwise at 2°C for 15 min to get **I**. Then, phenol (4.19 g, 44.6 mmol) was dissolved in methanol and added to the reaction mixture at 2°C. Next, 50% NaOH solution was added to the reaction mixture to increase the pH (>9.0) and reaction mixture was stirred for 4 hr. Finally, the reaction mixture was poured into the beaker containing distilled water (2 L). Solution is filtered to collect the reddish brown precipitate and solid **II** was recrystallized from methanol.

A reddish yellow colored solid; yield: 82%; melting point: 160.2–160.5°C; IR (KBr Pellet) $\gamma_{\max}$ in cm^{-1}: 3321 (OH), 1728 ($CO_2C_2H_5$), 1602, 1484, 1248, 1140 (C=C aromartic), 829 (C–H); ^1H NMR (500 MHz, acetone-*d6*): δ 8.12 (d, $J = 10$ Hz, 2H, Ar), 7.92 (d, $J = 5$ Hz, 2H, Ar), 7.88 (d, $J = 5$ Hz, 2H, Ar), 7.14 (d, $J = 5$ Hz, 2H, Ar), 5.54 (s, 1H, OH), 4.42 (q, $J = 12.2$ Hz, 2H, CH_2CH_3), 1.44 (t, 3H, CH_2CH_3).

General procedure for etherification reaction (III)

The solution of **II** (4.77 g, 18.5 mmol) and 1-bromohexane (6.12 g, 37.1 mmol) was prepared by acetone (30 mL). The catalytic amount of potassium iodide (50 mg) and also potassium carbonate (2.55 g, 18.5 mmol) were added to the reaction portionwise and refluxed. The reaction was completed after 24 hr and it was confirmed by TLC. Lastly, the reaction compound **III** was dried and used it for the next step.

General procedure for hydrolysis of ester (IV)

Dried compound **III** from the previous step was dissolved in methanol (100 mL) and the solution of potassium hydroxide (3.48 g, 62.2 mmol) in water (10 mL) was added dropwise. The reaction mixture was refluxed for 4 hr and the completion of the reaction was confirmed by TLC. Then, the reaction mixture was washed with *n*-hexane to remove the nonpolar impurities. Dilute HCl was added to the crude solution until the pH of the solution become 3. The compound was extracted with ethyl acetate and brine wash was given to remove ionic impurities. The crude solution was dried over anhydrous sodium sulfate and solvent was removed by rotor vapor. The crude solid obtained was recrystallized from methanol.

A dark yellow colored solid; yield: 75%; melting point is 173.2–173.6°C; IR (KBr pellet) γ_{max} in cm^{-1}: 1681 (COOH), 2942, 1421, 1600, 1289,, 1138 (C=C aromatic), 1248, 1025 (C–O), 837 (C–H); ^{1}H NMR (500 MHz, DMSO): δ 8.12 (d, $J = 5$ Hz, 2H, Ar), 7.93 (d, $J = 5$ Hz, 2H, Ar), 7.91 (d, $J = 5$ Hz, 2H, Ar), 7.14 (d, $J = 10$ Hz, 2H, Ar), 4.09 (t, $J = 15$ Hz, 2H, OCH$_2$), 1.74 (q, $J = 15$ Hz, $J = 10$ Hz, 2H, CH$_2$), 1.43 (q, $J = 5$ Hz, $J = 10$ Hz, 2H, CH$_2$), 1.32 (m, $J = 5$ Hz, 4H, CH$_2$ ×2), 0.89 (t, $J = 5$ Hz, 3H, CH$_3$); ^{13}C NMR (125 MHz, DMSO): δ 166 (COOH), 162, 154, 146, 132, 130, 125, 123, 122, 119, 114, 68 (OCH$_2$), 30, 28, 25, 22, 13.

General procedure to esterification by DCC coupling reaction (V)

The solution of **IV** (200 mg, 0.49 mmol) and resorcinol (30 mg, 0.27 mmol) was prepared with dichloromethane. The reaction mixture was stirred for 20 min. Then, catalytic amount of DMAP (10 mg) and also DCC (84 mg, 0.81 mmol) was added portionwise. The reaction mixture was stirred for 9 hr and the completion of reaction was confirmed by TLC. The reaction mixture was quenched with water (10 mL) and also washed with dilute acetic acid. Then, the compound was extracted with ethyl acetate and dried over anhydrous sodium sulfate. Ethyl acetate was removed by rotor vapor and the crude sample was purified by column chromatography with the mobile phase of 80% ethyl acetate in *n*-hexane and finally the target compound (**V**) was recrystallized from methanol.

A pale yellow colored solid; yield: 35%; melting point is 173.5–173.8°C; IR (KBr) cm^{-1}: 1735 (C = O), 2923, 2859, 1597, 1654, 1283, 1107 (C=C aromatic), 1253, 1025 (C–O), 841 (C–H); ^{1}H NMR (500 MHz, CDCl$_3$): δ 8.33 (d, $J = 10$ Hz, 4H, Ar), 7.89–7.99 (m, $J = 5$ Hz, 8H, Ar), 7.67 (d, $J = 5$ Hz, 4H, Ar), 7.52 (s, 1H, Ar), 7.02 (q, $J = 5$ Hz, 4H, Ar), 6.77 (d, $J = 5$ Hz, 2H, Ar), 4.07 (t, $J = 10$ Hz, 4H, OCH$_2$ ×2), 2.22 (q, 4H, CH$_2$ ×2), 2.01 (m, 4H, CH$_2$ ×2), 1.35 (m, 8H, CH$_2$ ×4), 0.92 (q, 6H, CH$_3$ ×2).

Result and discussion

Mesophase characterization

Differential Scanning Calorimetry (DSC) provides the information about phase transition temperature and change in enthalpy for the liquid crystal compound **V** (Fig. 1). Both heating and cooling cycles of DSC were measured with the constant rate of 10°C min^{-1} with mainly

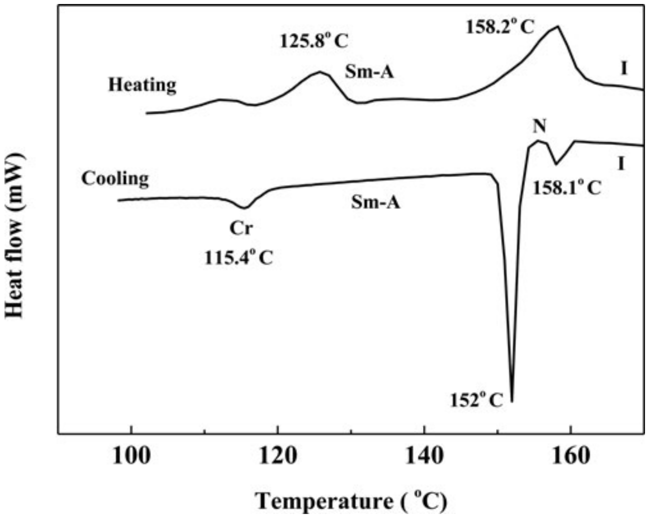


Figure 1. DSC heating and cooling cycles for **V** (10°C min^{-1}).

second heating and cooling cycle was considered in order to investigate the mesomorphic properties.

In compound **V**, endothermic peaks were found at 125.8°C and 158.2°C. Then, the sample was cooled spontaneously from the isotropic phase and the exothermic peaks were observed at 158.1°C and 152°C before crystallization (115.4°C). Therefore, the phase sequence for **V** is Cr 125.8, SmA 158.2 on heating and I 158.1, N 152, SmA 115.4, Cr on cooling mode.

The structure of the mesophase was captured using polaring optical microscope (POM, Fig. 2) and the hot stage was used to control the temperature during the observation of mesophase. The temperature rate of 5°C min^{-1} was maintained in the hot stage during the determination of liquid crystallinity of **V**.

The mesophase was further evaluated by X-ray diffraction method for the compound **V**. The X-ray diffraction was measured using Cu-Kα radiation with $\lambda = 1.54$A° and rotation anode generator of 4,000 W (Rigaku Ultrax-18) connected with graphite crystal monochromator. The sample was heated to obtain the mesophase in Hampton research capillaries with 0.5 mm diameter. The assignment of mesophase was confirmed by X-ray analysis and the intensity verses 2θ was plotted using the diffraction pattern of the compound **V** as shown in Fig. 3 (ii). The X-ray diffraction was studied at mesophase via cooling mode from the isotropic

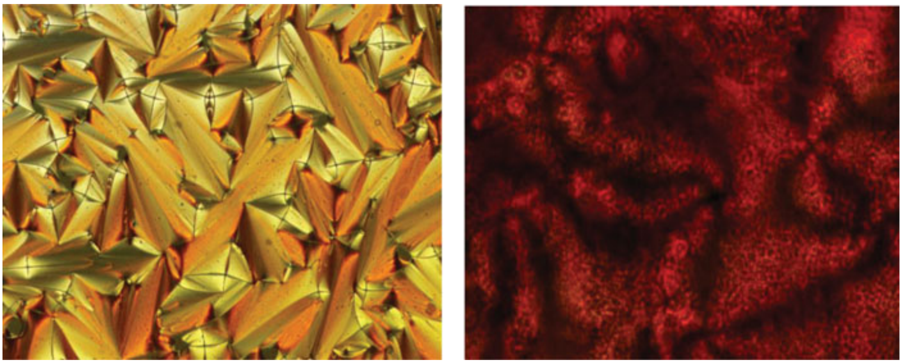


Figure 2. Microscopic images were captured by POM for and **V** (left SmA at 152°C and right N phase at 158°C).

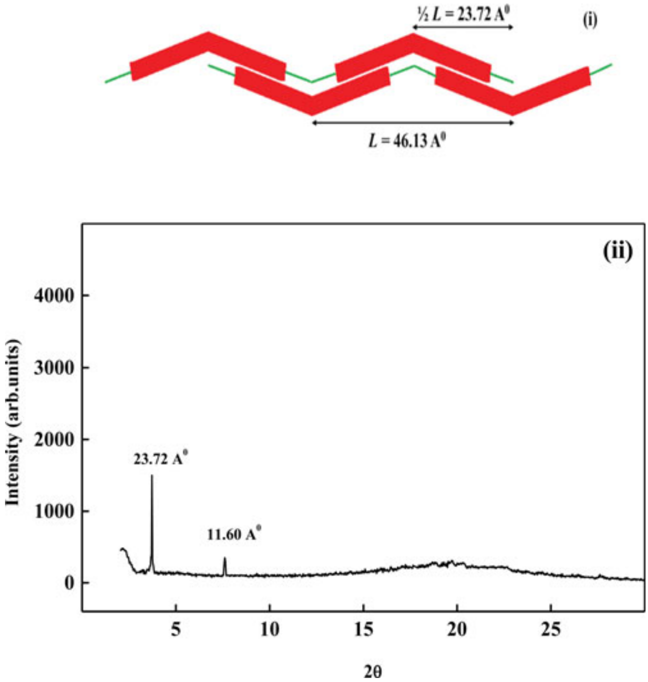


Figure 3. (i) The schematic picture of **V** at smectic A phase and (ii) the intensity versus 2θ plot derived from X-ray diffraction pattern of **V** at 145°C.

phase. A sharp reflection was found at 23.72 Å and small reflection at 11.6 Å, also a wide angled diffused scattering was observed at ˜4.45 Å at 145°C as shown in Fig. 3 (ii). The minimum conformation of the compound **V** is bent shape with respect to the molecular length 46.13 Å (Fig. 3 (i)). The peak at 23.72 Å corresponds to half of the total length of Y6c and also, the wide angled diffused scattering at ˜4.45 Å revealed the planar structured arrangement in the fluid phase. Therefore, V consists of the intercalated smectic A phase. The diffusion scattering at 11.6 Å is almost one-fourth of total length of LY 6c as shown in Fig. 3 (i) and this is due to the shrinkage of molecular order in the fluid phase. It means that there is a compressed arrangement of **V** molecules using intercalated packing system, when they are near to the isotropic phase via phase transition.

Photoswitching properties

The compound **V** is showing the absorption maxima at 356.14 nm. Here, the nature of the spacers is responsible for the variation in photo-reactivity of the compounds [14]. Tacitly, the nature of the spacers involves flexibility, elasticity, and free rotation parameters [2, 17–20].

Ostensibly, the photoisomerization was analyzed using solution to get the idea about the structure–property relationship of the spacer substituted azobenzene dimers. The *trans* to *cis* isomerization of **V** is given in Fig. 4. The isomerization started when the UV light of threshold energy 5.5848×10^{-19} J was illuminated on the system of molecules. The solution of the compound **V** was prepared with chloroform with the concentration of 1.1×10^{-5} mol L^{-1}.

Photoswitching properties were investigated with the fixed intensity of 5.86 mW cm^{-2} and heat filter was used to avoid the thermal *trans* to *cis* isomerization. The compound was illuminated with the UV light to reach the photostationary state. The photostationary state was

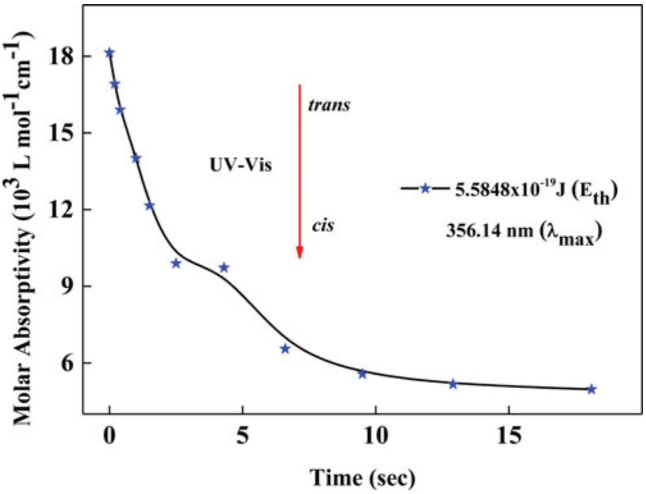


Figure 4. *Trans* to *cis* isomerization of **V**.

found at 18 sec for the compound **V**. The absorption maxima was decreased stepwise with the irradiation of UV light [4, 21] and the absorption parameters were recorded during systematic illumination of light.

Flexibility and rigidity of the spacers are an important factor for free rotation and molecular movements [22]. In most of the cases, the thermal back relaxation is spontaneous and it does not need an external stimuli [23–25]. Nevertheless, back relaxation is very fast when the light of 450 nm is illuminated on the azobenzene derivatives [4, 26], but thermal back relaxation is interesting due to the variation in the duration [14]. Absorption spectra were recorded simultaneously from the point of photosaturation to the original state. The back relaxation was determined finally for **V** with the values 10.95 hr (Fig. 5).

Tacitly, spacers play an important role in the thermal back relaxation and the factor affecting the process is flexibility [27]. The flexibility of spacer mainly has an impact on free rotation and mobility of the molecules [22]. Thermal back relaxation is longer in case of these resorcinol spacer substituted azobenzene compound. This is due to the rigid core of resorcinol spacer

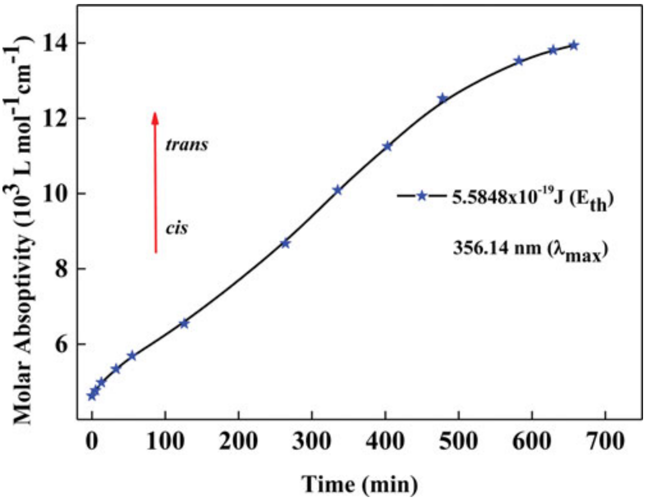


Figure 5. Thermal back relaxation of **V** after photostationary state.

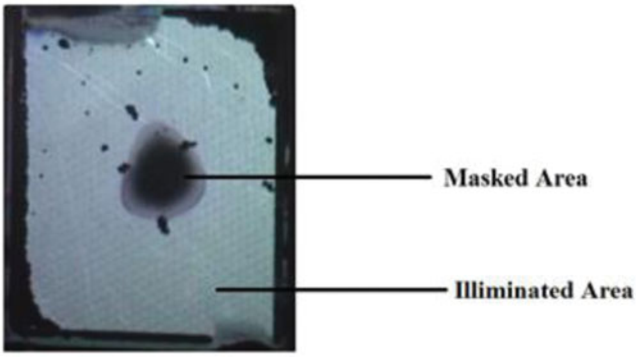


Figure 6. Liquid crystal prototype cell image and it is constructed by **V**.

that disturbs the mobility [14] and free rotation [22] of molecules in the system. Usually, the mobility of the molecules increases [2, 14] and the configuration of them also changes [28] when sufficient amount of energy is supplied to the molecular system. Generally, light is a form of energy and it changes the molecular configuration and increases the mobility of each molecule [29–30]. Therefore, dimeric azodyes with resorcinol rigid spacers provide long thermal back relaxation. So, the activity of the compounds can be tuned by incorporating various spacers in the chromophoric and light sensitive compounds.

Exactly, 5% of **V** was mixed with commercial liquid crystal 5CB and this homogeneous mixture was eluted into an ITO coated, unidirectionally rubbed cells (Fig. 6). Initially, absorption parameters of liquid crystal prototype cell were recorded and the UV light (intensity = 5.86 mW cm^{-2} and $E_{\text{th}} = 5.5848 \times 10^{-19}$ J) was illuminated on it. Absorption spectra were recorded simultaneously during the systematic irradiation of UV light. The photostationary state was found at 14 sec and the thermal back relaxation was observed after 6.14 hr from photosaturation. Interestingly, disorder to order state transition was observed in this case and it is confirmed by further investigation.

Again, 9% of the compound **V** was mixed with commercially available liquid crystal 5CB and coated on the previously glass substrates using spin coater. Then, the polarized images were captured from the microscope before and after illuminating UV light for 1 min of intensity 20 mW cm^{-2}. The mixture showed the disordered state before UV light irradiation and interestingly the clear ordered texture was observed after UV light irradiation. Prasad et al. proved this phenomenon earlier [31]. Even though, this disorder to order transition is not

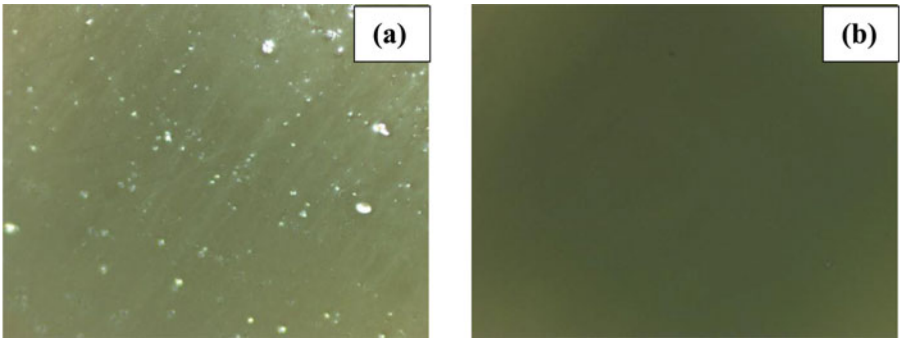


Figure 7. Disordered to ordered molecular orientation of compound **V**.

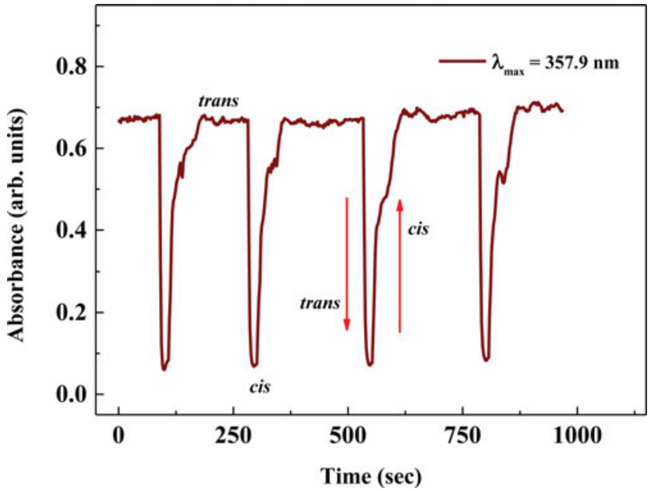


Figure 8. The determination of photo-stability of **V**.

common in light sensitive compounds and it is rarely found in nature. In Fig. 7, (*i*) before illuminating UV light (*disordered state*) and (*ii*) after illuminating the UV light (*ordered state*).

Figure 8 shows the photo-stability of the compound **V**. Multiple cycles of photoisomerization are considered here, irrespective of time of *trans–cis* and *cis–trans* isomerization. The compound **V** is dissolved in chloroform with the concentration 1.1×10^{-5} mol L^{-1}. The UV light of intensity 10 mW cm^{-2} was illuminated on the solution continuously, until photostationary state. Immediately, visible light of 10 mW cm^{-2} (450 nm wavelength) was irradiated on the solution for the back relaxation process after photosaturation state. This phenomenon was continued several times to evaluate the photo-stability of the compound. Thus, these compounds are stable toward light and does not degrade via light illumination. Also, one can reuse the compound several times in the photo-induced process without discording the compound as a waste. Therefore, these siloxane substituted azodyes are suitable in optical storage device industries, due to the excellent light-induced characteristics. Mainly, industries may reduce the chemical wastes by the usage of these compounds.

Conclusion

The new dimeric azodye with resorcinol spacer was synthesized. It was showed nematic and smectic A phases in the mesomorphic investigation. Next, mesophases were studied in detail using DSC, POM, and XRD. The presence of spacer in the molecule plays an important role in photoisomerization. This compound is suitable for photochromism studies under UV irradiation. It is clear that the effect of resorcinol spacer is considerable in the study of optical properties of the compounds due to the rigid core. The photoswitching properties of compound show *E–Z* isomerization at 18 sec, whereas the reverse process takes place at around 11 hr in solution. Solid study showed *E–Z* photoisomerization takes around 14 sec and the reverse to the original *Z–E* takes around 6 hr. Thus, the photoswitching behavior of this material may be suitably exploited in the field of optical data storage devices and in molecular switches. Interestingly, order to disorder state transition was found in the prototype device evaluation. Also, the photo-stability of this compound was checked and the compound was not degraded in many cycles of prolonged irradiation of UV light. Hence, this compound is suitable for industrial use.

Acknowledgments

This research was supported by FRGS research grant (RDU 130121). Thank Mrs. K. N. Vasudha for supporting this work.

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