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Analysis of birefringence property of three different nematic liquid crystals dispersed with TiO₂ nanoparticles

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ABSTRACT

In the present work TiO₂ nanoparticles (NPs) have been dispersed into three different nematic liquid crystals (2020, 1823A and 1550C) in different concentration. The value of the birefringence (Δn) has been calculated by the transmitted intensity method at a 632.8 nm wavelength. NLC 2020 used in the present study is a high birefringent material ($\Delta n=0.44$), NLC 1550C is a low birefringent material ($\Delta n=0.067$) and NLC 1823A is a mid birefringent material ($\Delta n=0.14$). An increased value of birefringence has been found after dispersion of TiO₂ NPs in all three NLCs but this increment depends upon the concentration of the dopant material, temperature range and chemical character of the mixtures. It is suggested that this LC materials can be applicable in making of phase shifters, compensators and many more photonic devices.

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Introduction

Liquid crystal materials having high birefringence (Δn) are very useful because of their application in liquid crystal displays (LCDs), as well as in non-display devices. One of the most basic and important property of nematic liquid crystals is optical birefringence. In the twisted – nematic (TN) mode (and similarly in other important electrooptical mode such as IPS, VA) the gap of cell (d) is directly related to the birefringence and response time [1,2]. Generally for display devices we need high resolution and high contrast ratio and this can be done by carefully adjustment of birefringence (Δn). Birefringence of liquid crystal mixtures can be changed by proper selection of components from different chemical structures or by doping polymers (a polymer network formulation), solvents, dyes or nanoparticles. It is not a problem to tailor low and medium birefringence LC crystal media, because many families of LCs with low and medium viscosity are known. The greater task is to obtain high or very high birefringence LCs because highly conjugated structures are necessary to be used here and they are often very viscous or show smectic feature [3]. Therefore, the use of nanoparticles may be alternative choice. They increase the value of birefringence by the change of order parameter (S). Their concentration is very small so the increase of viscosity is also small or even it may be decreased

[4–6]. Order parameter S is directly correlated with birefringence [7,8]. Enhanced birefringence for metallic nanoparticles doped liquid crystal has been studied by Li et al. [9]. Some also important study about the effect of birefringence after dispersion of polymer and quantum dot into the nematic liquid crystal has already been described by our group [10,11].

There are many advantages of liquid crystal devices which are developed by materials having a high optical birefringence. The nematic liquid crystals which are polar in nature and have low viscosity have found many applications in GHz and THz ranges of electromagnetic radiation [12–14]. The phase shift introduced by nematic liquid crystal in the cell of thickness d can be measured as:

$$\Delta\phi = \frac{2\pi}{\lambda} \Delta n d.$$

Where d is the thickness of the cell, Δn is the birefringence and λ is the wavelength of light.

There are many applications of nematic liquid crystal (NLC) having high birefringence value and broad nematic range [15–20]. The applications of liquid crystalline materials lie only when they are aligning in the particular direction because these materials are highly anisotropic in nature. Since for liquid crystals, having positive dielectric anisotropy, the value of electric permittivity along molecular axis ($\epsilon_{||}$) will be greater than as compared to perpendicular (ϵ_{\perp}) to it (i.e., $\epsilon_{||} > \epsilon_{\perp}$). Here ϵ_{\perp} will show the minimum value for a perfectly aligned NLC. But if we disturb the alignment of molecules of NLC, then ϵ_{\perp} will show an increased value due to

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$\varepsilon_{||}$. The disturbance in the alignment of NLC molecules will lead to the change in dielectric anisotropy ($\Delta\varepsilon = \varepsilon_{||} - \varepsilon_{\perp}$), as well as birefringence $\Delta n = n_e - n_o$ (where n_e and n_o are the extraordinary and ordinary refractive indices).

There are several techniques used for measurement of the birefringence value such as total reflection method [21,22] and hollow prism method [23]. Calculation of phase difference of monochromatic light when passed through cell has been proposed by Wu et al. [24]. In the present investigation the voltage-dependent transmission technique has been used for the measurement of birefringence.

In this work we have measured the birefringence of NLCs after dispersion of TiO_2 NPs by transmittance technique. For this we have dispersed same anatase TiO_2 NPs in three different nematic liquid crystals (2020, 1823A and 1550C) in different concentration. We have measured the birefringence of pure and all the mixtures with the variation of temperature. All three nematic liquid crystals used in this study have different birefringence values. NLC 2020 is a high birefringence material having $\Delta n > 0.4$, NLC 1550C is a very low birefringence material having $\Delta n < 0.1$ and NLC 1823A is a mid birefringence material having $\Delta n > 0.1$. Nematic liquid crystal materials having low and medium birefringence values have many display applications [25–27]. These types of materials are used in large TV screens, flat panel mobiles phones and in many projectors. Colour sequential LCDs produce a better quality picture [28,29], but they need fast operating materials, which can be ensured only by the high birefringence LCs with a small gap [30]. The value of ordinary refractive index (n_o) for NLC 1550C is less than as compared to silica glass in high temperature range. It enables to observe the change of light preparation mechanism in a photonic liquid crystal fiber from photonic band gap (PBG) [31].

Experimental details

Here we have used three different types of liquid crystals having same dopant. We are using three different nematic liquid crystals: NLC mixture 2020 [Eq. (1)] which consists of 4'-alkyl-4-isothiocyanatotolanes and 4'-alkylphenyl-4-isothiocyanatotolanes having in the lateral positions fluorine atoms [32]. NLC mixture 1550C [Eq. (2)] which consists of 4'-(trans,trans-4-alkylbicyclohexyl)carbonates and 4'-(trans,trans-alkyl)-4-alkenylbicyclohexane and 4'-(4-(trans,trans-4-propyl)-4-cyanobicyclohexane, NLC mixture 1823A [Eq. (3)] which consists of 4-(4-trans,trans-cyclohexyl)- and 4-(trans,trans-bicyclofluorobenzenes and isothiocyanatobenzenes [33], received from Institute of Chemistry, Warsaw, Poland.

NLC 2020 has the phase sequence as follows:



And $\Delta n = 0.45$ (589 nm), $\Delta\varepsilon = 15.5$, $\eta = 24.6$ mPas, $\gamma = 224.7$ mPas at 20°C

NLC 1550C has the phase sequence as follows:



And $\Delta n = 0.059$ (589 nm), $\Delta\varepsilon = 3.2$, $\eta =$ mPas at 20°C

NLC 1823A has the phase sequence as follows:



And $\Delta n = 0.1697$ (589 nm), $\Delta\varepsilon = 8.10$, $\eta = 10.26$ mPas at 20°C

The dopant we are using here is a TiO_2 nanoparticle. Size of TiO_2 nanoparticle used here is spherical in shape with a diameter less than 25 nm [34–36].

In this study TiO_2 NPs have been dispersed in three different concentrations 0.1 wt% (mix 1), 0.25 wt% (mix 2) and 0.5 wt% (mix 3) in all three different NLCs i.e. NLC 2020, 1550C and 1823A. Here TiO_2 NPs have been dispersed in propanol –2 for proper mixing

and stirred for 6 hours. Propanol-2 owing to hydroxy group can absorb itself strongly on a TiO_2 surface via hydrogen bonds and, therefore it can be difficult to be removed totally from surface during evaporation process. Also some alcohols have ability to react with NCS group towards thiocarbonates. To exclude that, it does not occur in this case, we have used acetone as another solvent. Then, a proper volume concentration is mixed with NLCs and solvent was evaporated at its isotropic temperature. In this investigation planar aligned cells of thickness 8 μm have been used to determine the birefringence property for all the samples. To achieve the planar aligned cell we have coated nylon (6/6) on the two ITO coated glass plates and rubbed unidirectionally. Then, we have placed the mylar spacer in between the plates. Now we sealed these two plates by UV sealant. The cells have been calibrated using an AR grade benzene. Next, cells have been filled with pure and dispersed LC above their isotropic temperature by the capillary method. The temperature has been controlled with an INSTEC mk2000 hot plate with an accuracy of $\pm 0.001^\circ\text{C}$. Process of preparation of an LC cell has been described in detail by our group [37,38].

Optical birefringence measurement (transmittance technique)

Figure 1 shows the experimental set-up of birefringence for the study of pure NLC and NPs dispersed system. Here, first we set the polarizer and analyzer in cross position. Now we have placed the planar aligned LC cell in between the crossed polarizer and analyzer for minimum intensity. In this experiment the He-Ne laser beam, having a wavelength (λ) 632.8 nm and a power of 5 mW, falls onto the planar aligned LC cell of a thickness of 8 μm . Now we have rotated the LC cell at an angle of 45° in either direction. Then, we have heated the LC cell up to isotropic temperature of nematic. Here we have measured the intensity I_{\min} and I_{\max} for crossed and parallel conditions of polarizer and analyzer. Here output intensity has been measured in terms of voltage with the help of photo detector (Instec – PD02-P) which is attached to a digital storage oscilloscope (Tektronics TDS-2024C). Here temperature is varied by a temperature controller hot plate (mk2000, Instec Co USA). As we can see from this figure that a phase shift is producing within the cell. Here a linear polarized light will reach the photo detector after passing through the analyzer. The intensity of this linear polarized light will depend upon the profile of elliptical light. Now we have plotted the transmittance of Vs temperature curve. In this plot each peak corresponds to a phase difference of π between E and O rays. Now we have calculated the phase difference at different temperature using the following Eqs. (4) and (5) [39,40]:

$$\Delta\phi = m\pi + 2\sin^{-1} \sqrt{\frac{I - I_{\min}}{I_{\max} - I_{\min}}} \quad \text{for } m = 0, 2, 4, \quad (4)$$

$$\Delta\phi = (m+1)\pi - 2\sin^{-1} \sqrt{\frac{I - I_{\min}}{I_{\max} - I_{\min}}} \quad \text{for } m = 1, 3, 5, \quad (5)$$

Where m is the number of the maxima peak observed in Intensity Vs temperature graph in addition to peaks observed with temperature, I is the intensity at observed temperature, I_{\max} and I_{\min} is the maximum and minimum intensity of incident light coming through cell, respectively. Now we have calculated the birefringence (Δn) using Eq. (6) which is given below:

$$\Delta n = \frac{\lambda}{2\pi d} \cdot \Delta\phi \quad (6)$$

Where λ is the wavelength of the incident light, $\Delta\phi$ is the phase difference and d is thickness of the LC cell. Enhancement in the value of birefringence has many electro-optic applications [41,42].

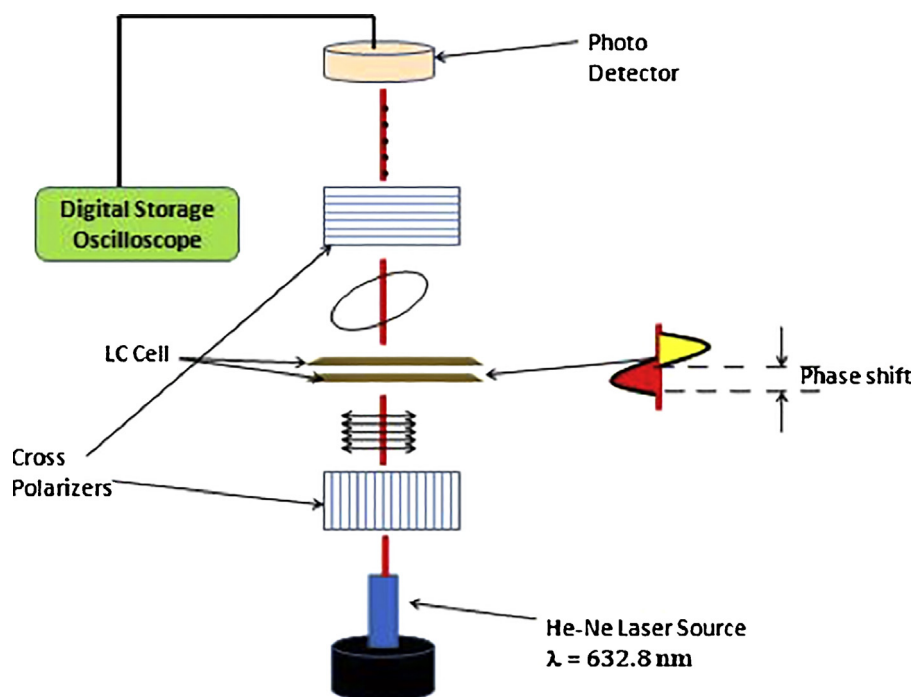


Fig. 1. Experimental set-up for the measurement of birefringence of pure NLC and NPs dispersed systems.

Results and discussion

Figure 2(a) and 2(b) show the variation in birefringence (Δn) for NLC 2020 and NP dispersed NLC system with variation of temperature. Here we can see that the birefringence has been decreasing for pure and dispersed system with increment in temperature. The birefringence is showing a higher value for dispersed system as compared to pure NLC as follows: mix 3 > mix 2 > mix 1 > pure NLC 2020 in all temperature range and for both solvents the changes almost the same. Figure 2(c) and 2(d) represent the variation of transmitted intensity with respect to change in temperature for pure NLC 2020 and dispersed system. As we can see that intensity goes through maxima and minima as we increase the temperature due to change in molecular orientation and so phase difference. Here the given model in Fig. 2(e) represents the presence of TiO_2 nanoparticles into the nematic liquid crystal. As we can see in the given model that the orientational order has been increased after dispersion of TiO_2 NPs into NLC, therefore anisotropy has been increased. Here TiO_2 nanoparticles are spherical in shape whereas NLC molecules are elongated in shape. The molecules of alkyl isothiocyanatolanes and alkylphenyl isothiocyanatolanes absorb strongly on the polar surface of TiO_2 NPs. For this NLC-NP composite system the intermolecular forces are very strong and therefore orientational order increases, as a result birefringence also enhanced.

Variation of birefringence with respect to different concentration of dopant for pure NLC 2020 and all three mixtures (mix 1- 0.1% concentration of TiO_2 NPs, mix 2- 0.25% concentration of TiO_2 NPs and mix 3- 0.5% concentration of TiO_2 NPs) has been shown in Fig. 3. Drastic change has been found in the value of birefringence after dispersion of TiO_2 NPs in amount of 0.1 wt. %. At 35 °C temperature the birefringence for pure NLC is found to be 0.44 while for mix 1, mix 2 and mix 3 the values of birefringence are 0.52, 0.57 and 0.62, respectively, the observed increment is 0.08, 0.13 and 0.18, respectively. We get almost 40% increment in the value of birefringence for mix 3 as compared to pure NLC which is a very large increment. It is necessary to mark that such big increase of birefringence values by chemical method has very much importance [3].

When we dispersed the TiO_2 NPs in pure NLC 2020 then the orientational order improves itself respectively for all the mixtures (mix 1, mix 2 and mix 3). Therefore, all concentrations of NPs show much more improvement in the orientation order and, therefore birefringence has also been increased. This is confirmed by shifting the isotropic point towards the higher temperature side due to strong anchoring of liquid crystal molecule on the aligning TiO_2 surface. Similar shifting of the isotropic point is observed after biased strong electric field [43]. We observe some birefringence value even at its isotropic point, see Figs. 2(a) and 2(b). Since the order in liquid crystal is directly related to the anisotropic nature of liquid crystal, therefore the behaviour of birefringence after dispersion of NPs in the NLC indicates the higher orientation order of NP dispersed NLC system as compared to pure NLC system. The enhancement in the optical birefringence occurs due to increased molecular alignment in the LC phase.

Variation in birefringence (Δn) for pure NLC 1823A (which is a low viscosity and mid birefringence material, $\Delta n = 0.14$) and NP doped NLC system with change in temperature is shown in Figs. 4(a) and 4(b). We can see from the figure that the mix 1 reaches near the saturation point and, therefore we are not getting any further increment in the values of birefringence for mix 2 and mix 3. So we can observe that birefringence is not increasing in the same fraction for higher concentration of TiO_2 NPs as compared to lower concentration, but it saturates. When the concentration of the TiO_2 NPs is increased in the low viscous NLC mixture 1823A, the aggregation of the nanoparticles occurs and, therefore effective number of the NPs decreases. Hence, no prominent change has been observed in the dispersed system. Figures 4(c) and 4(d) show the variation of transmitted intensity with respect to change in temperature for pure NLC 1823A and dispersed system. Here intensity varies through maxima and minima as we increase the temperature due to change in phase difference.

In this investigation Figure 5 shows the variation of birefringence with respect to different concentration of dopant for pure NLC 1823A and all three mixtures (mix 1, mix 2 and mix 3). At 35 °C temperature the value of birefringence for pure NLC 1823A is found to be 0.14 while for mix 1, mix 2 and mix 3 the values of

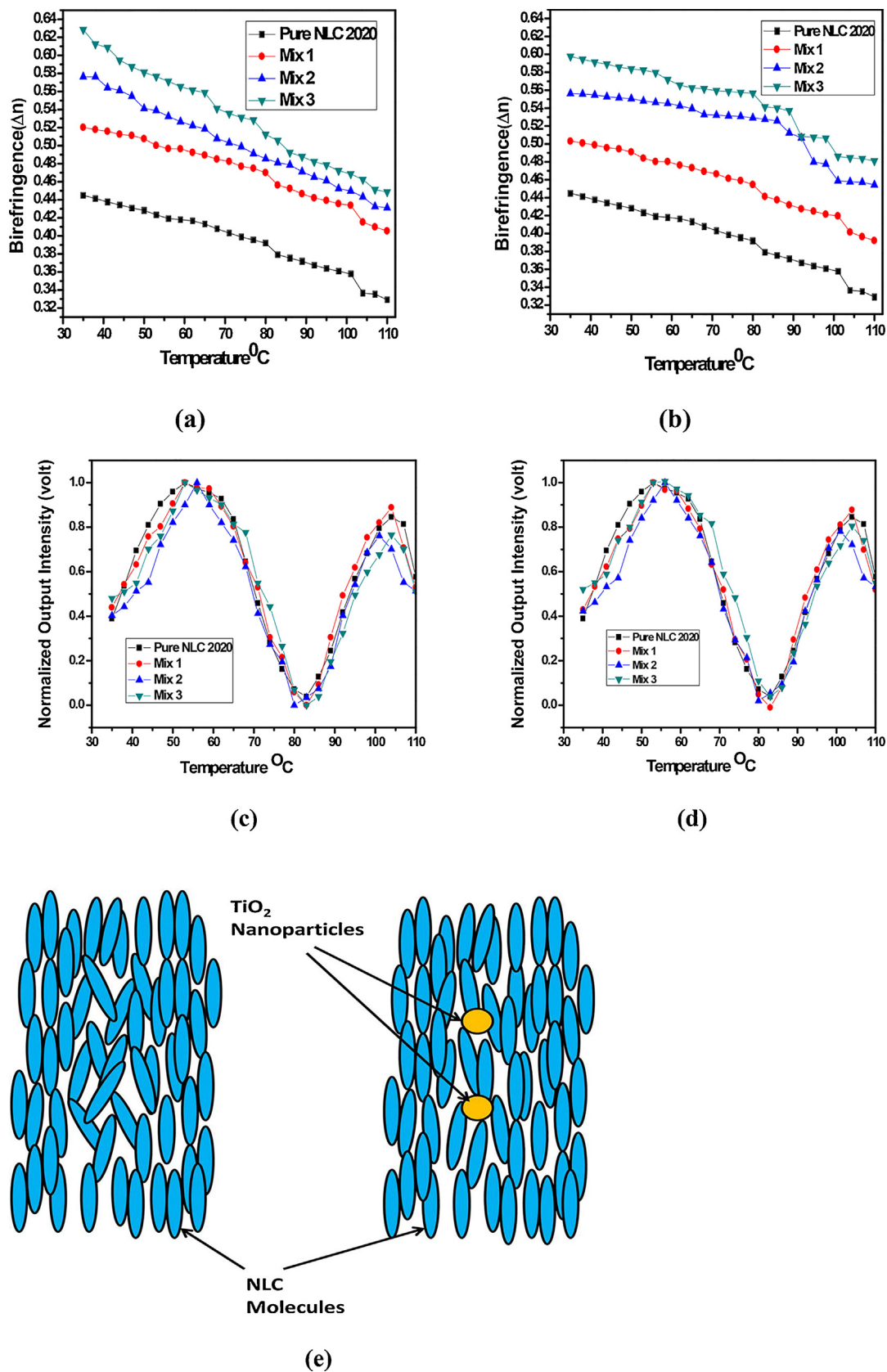


Fig. 2. Variation of birefringence (Δn) with respect to temperature for pristine NLC 2020 and NP doped NLC system in planar aligned cell, (a) Propanol-2 as solvent, (b) Acetone as solvent, (c) Variation of transmitted intensity with respect to temperature for pure NLC 2020 and dispersed system for propanol-2 as solvent, (d) Variation of transmitted intensity with respect to temperature for pure NLC 2020 and dispersed system for acetone as solvent, (e) Represents the interaction of TiO_2 nanoparticle with NLC molecules.

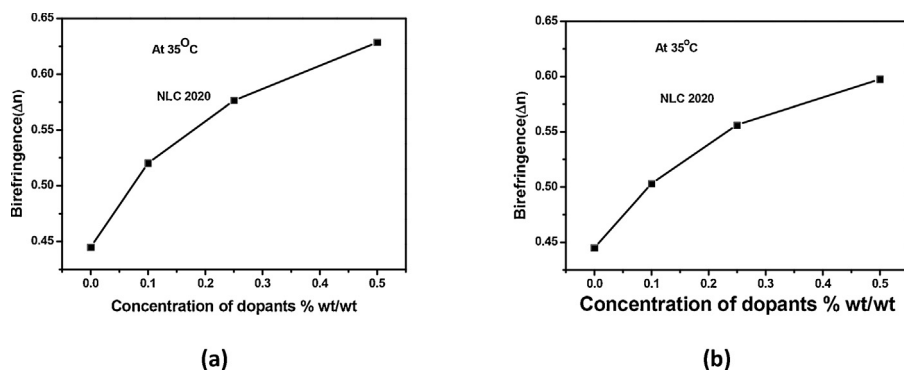


Fig. 3. Variation of birefringence (Δn) with respect to different concentration of dopant for pure NLC 2020 and all three mixtures (mix1- 0.1% concentration of TiO_2 NPs, mix2- 0.25% concentration of TiO_2 NPs and mix3- 0.5% concentration of TiO_2 NPs), (a) Propanol-2 as solvent, (b) Acetone as solvent.

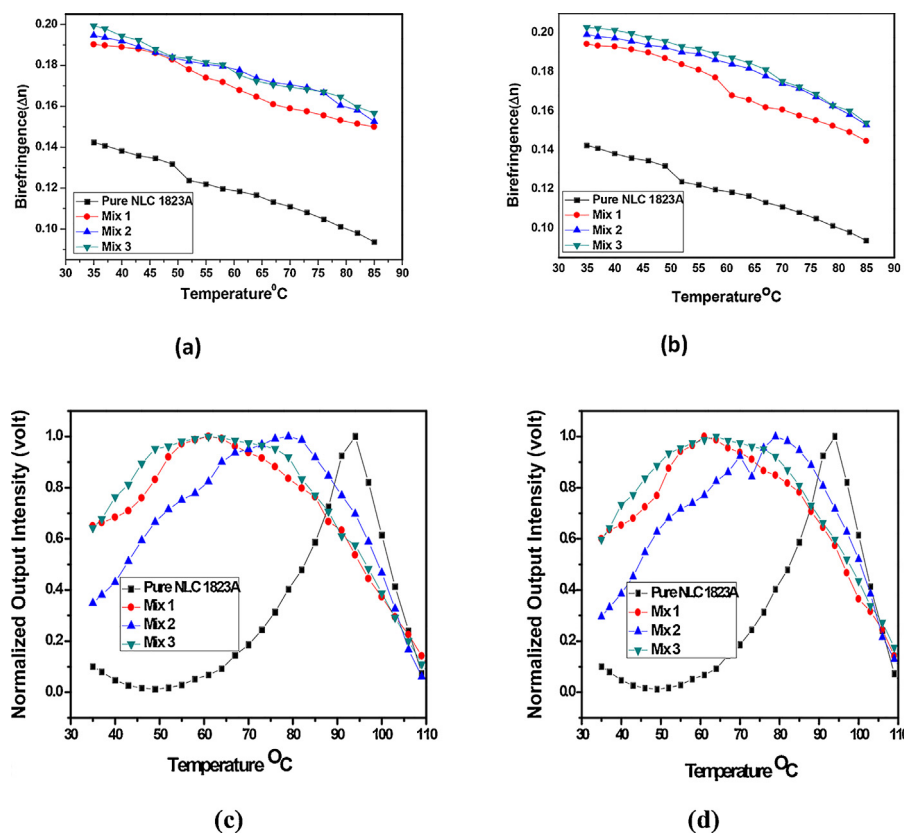


Fig. 4. Variation of birefringence (Δn) with respect to temperature for pristine NLC 1823A and NP doped NLC system in planar aligned cell, (a) Propanol-2 as solvent, (b) Acetone as solvent, (c) Variation of transmitted intensity with respect to temperature for pure NLC 1823A and dispersed system for propanol-2 as solvent, (d) Variation of transmitted intensity with respect to temperature for pure NLC 1823A and dispersed system for acetone as solvent.

birefringence are 0.190, 0.194 and 0.199, respectively. The observed concentration increment is only 0.05, 0.054 and 0.0059, respectively in the case of propanol-2 used as the solvent. We can see that there is a rapid increase in difference of birefringence value between pure NLC and mix 1; but mix 2 and mix 3 have almost same value of birefringence as mix 1. We found here that we get almost 29% increments in the value of birefringence for pure NLC to mix 3 which is also a very large increment. Probably in the case of low viscous mixture 1823A the aggregation of nanoparticles occurs and their effective number decreases with the increase of their concentration.

Variation of birefringence (Δn) for pure NLC 1550C and NP dispersed NLC system with change in temperature is shown in Fig. 6. Here in this case increment in the birefringence at higher temperature is not as prominent as at lower temperature. When we

dispersed TiO_2 NPs into pure NLC, ordering improves but only at low temperature. When we are approaching towards the isotropic point (above 50°C temperature), kinetic energy of all the liquid crystal molecules and TiO_2 nanoparticles will be enhanced. The random motion of the molecules of NLC and TiO_2 NPs will decrease the orientational order and, hence birefringence. Therefore after 50°C temperature we are not getting any prominent change in birefringence for dispersed system. Also, at temperature above 50°C interaction of NLC-NP with the molecules is broken, and the transition temperature is not changing in the presence of NPs. The components of mixture 1550C (dialkylbicyclohexanes and alkylbicyclohexylcarbonates) are unpolar molecules (their dipole moment $\mu \sim 0$) and they adsorb badly on the strongly polar surface of TiO_2 nanoparticles. Intermolecular forces between TiO_2 solid surface and LC molecules are very weak here and they have only Van

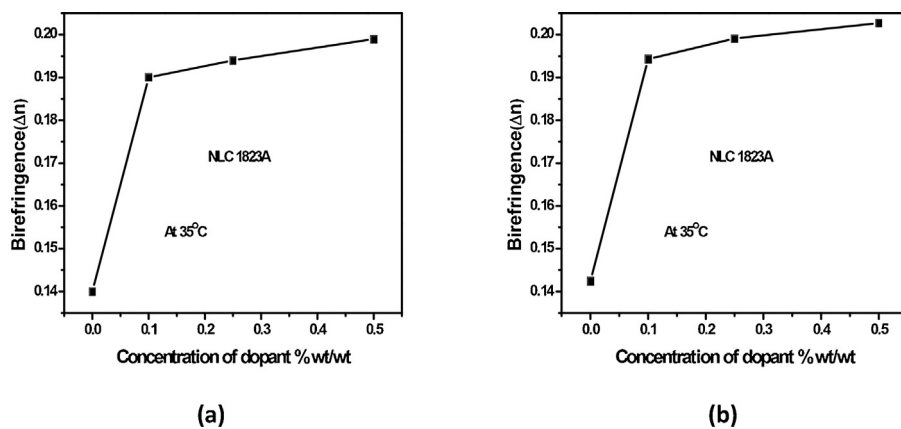


Fig. 5. Variation of birefringence (Δn) with respect to different concentration of dopant for pure NLC 1823A and all three mixtures (mix1- 0.1% concentration of TiO_2 NPs, mix2- 0.25% concentration of TiO_2 NPs and mix3- 0.5% concentration of TiO_2 NPs), (a) Propanol-2 as solvent, (b) Acetone as solvent.

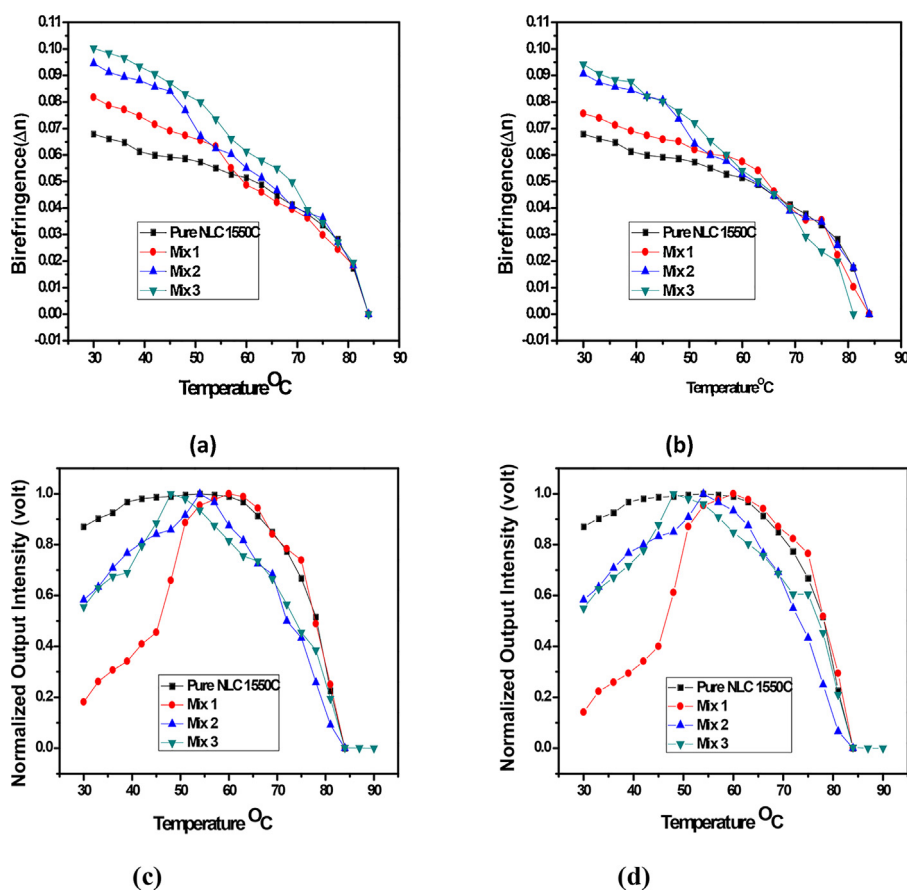


Fig. 6. Variation of birefringence (Δn) with respect to temperature for pristine NLC 1550C and NP doped NLC system in planar aligned cell, (a) Propanol-2 as solvent, (b) Acetone as solvent, (c) Variation of transmitted intensity with respect to temperature for pure NLC 1550C and dispersed system for propanol-2 as solvent, (d) Variation of transmitted intensity with respect to temperature for pure NLC 1550C and dispersed system for acetone as solvent.

der Waals character which are easily broken at high temperature region. Therefore, this also confirms that birefringence does not only depend upon TiO_2 concentration. We can also see from Figs. 6(a) and 6(b) that there is a discontinuous change in the refractive indices at the nematic-liquid transition point and the birefringence gradually falls to zero at the same temperature. Also Figs. 6(c) and 6(d) show the variation of transmitted intensity with respect to change in temperature for pure NLC 1550C and dispersed system. Intensity varies through maxima and minima as we increase the temperature due to change in molecular orientation and therefore phase difference also changes.

Figure 7 shows the variation of birefringence with respect to different concentration of dopant for pure NLC 1550C (which is a low birefringent material, $\Delta n = 0.067$) and all three mixtures (mix 1, mix 2 and mix 3). At 35°C temperature the value of birefringence is found to be 0.067 for pure NLC while for mix 1, mix 2 and mix 3 the values of birefringence are 0.081, 0.094 and 0.1 respectively and observed concentration increment for Δn is 0.014, 0.027 and 0.033. We can see that birefringence is increasing continuously for dispersed system. The absolute increase of the Δn is not high, but relative increase at low temperature is big. We found here that we

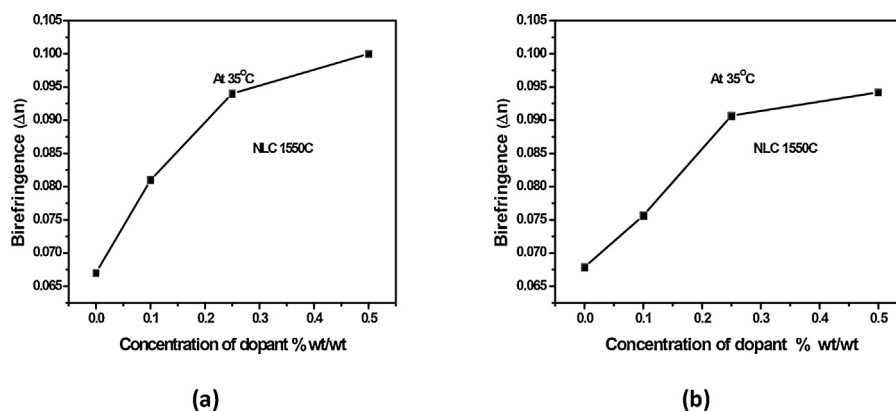


Fig. 7. Variation of birefringence (Δn) with respect to different concentration of dopant for pure NLC 1550C and all three mixtures (mix1- 0.1% concentration of TiO_2 NPs, mix2- 0.25% concentration of TiO_2 NPs and mix3- 0.5% concentration of TiO_2 NPs), (a) Propanol-2 as solvent, (b) Acetone as solvent.

Table 1

Birefringence data of all nematic liquid crystals along with their all mixtures of TiO_2 nanoparticles.

NLC Materials	Birefringence (Δn) values			
	Pure NLC	mix 1	mix 2	mix 3
NLC 2020	0.44	0.52	0.57	0.62
NLC 1823A	0.14	0.190	0.194	0.199
NLC 1550C	0.067	0.081	0.094	0.100

get almost 33% increments in the value of birefringence for mix 3 as compared to pure NLC 1550C.

The birefringence of NLCs is the very important parameter for liquid crystal electro-optical devices. Δn varies when the liquid crystal is subjected to electric fields. In the present study we observed that the birefringence is continuously increasing for all the three NLCs samples (2020, 1823A and 1550C) in high fraction after dispersion of TiO_2 nanoparticles and increment depends upon the concentration of NPs. The percentage of increment is highest in NLC 2020 (almost 40%) while lowest in NLC 1823 (almost 29%). For low birefringent NLC (1550C) increment in the birefringence at higher temperature is not as prominent as at lower temperature whereas for high polar systems (2020 and 1823A) the interaction of NLC-NP does not decrease towards high temperatures, therefore increase of birefringence is observed at high temperature, too. The detailed birefringence data of all nematic liquid crystals along with their all mixtures of TiO_2 has been given in Table 1.

Conclusions

In the present investigation we have dispersed anatase TiO_2 NPs into three different nematic liquid crystals (2020, 1823A and 1550C having different chemical character, which leads to different birefringence, polarity and viscosity values) in three different concentrations. Here we have observed that the birefringence has been increased in large fraction for TiO_2 NPs dispersed system (concentration of NPs – 0.1%, 0.25% and 0.5%) as compared to the all pure NLCs but in different way for concentration and temperature in the investigated mixtures. In the high polar and higher viscous nematic mixture LC 2020 the increase of Δn is observed for all temperatures, wherein the nematic phase exists and for all dopant concentrations. Birefringence has been found to be increased with the increment of dopant concentration accordingly. The maximum increase in the birefringence value has been found to be 0.62 for mix 3 as compared to pure NLC 2020 (0.44). This will enable to use a 1.4 times thinner electro-optical cell. Such doped high birefringence LC mixtures may be especially interesting for GHz and THz application. In the medium polar and very low viscous mixture 1823A the increase of

Δn is observed in all temperature ranges of nematic phase and for all three investigated concentration, but optimum dopant concentration is in the region of 0.1 wt. %. At higher concentration probably aggregation process of nanoparticles occurs. At 35 °C temperature the increase of Δn is observed from 0.14 to 0.199 and its ratio is also 1.4. In the medium viscous and low polar mixture 1550C the increase Δn after doping TiO_2 nanoparticles is observed only below 50 °C but for all the three the investigated concentration and clearing points have not shifted towards higher temperature region. For 0.5 wt. % of doping Δn increases from 0.067 to 0.1 but that above 1.4 times. The improvement in the alignment in NLC matrix with the dispersion of NPs has led in the enhanced birefringence of NLC matrix, because the interaction of LC molecules with nanoparticles increases order parameters in all volume. Since we know that Δn is inversely proportional to cell gap (d), therefore by large Δn we can reduce the cell gap d and, thus shortening the response time in a quadratic way. Also thinner cell gap will serve to weaken the fringing fields, thereby more power being transmitted in the desired first order and less power being lost in the side lobes. High birefringence after dispersion of NPs into the NLC has many applications such as in phase shifters and in many more photonic devices. It can also be useful in flat panel displays, photonic devices and projectors if the stability of dispersed system will be long in time.

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