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RESEARCH

Effect of ion trapping behavior of TiO_2 nanoparticles on different parameters of weakly polar nematic liquid crystal

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Abstract

In the present investigation, TiO_2-doped weakly polar nematic liquid crystal (NLC) has been studied. This study mainly focuses on dielectric, electro-optical and optical properties of pure NLC and doped systems. Variation in different parameters like dielectric permittivity, dielectric loss, dielectric anisotropy and conductivity has been noticed. Permittivity of the doped system is almost the same as undoped system. With doping concentration, the ionic contribution to the dielectric loss as observed in low-frequency region has been suppressed and the shift in relaxation frequency toward higher frequency side has also been observed. The electrical conductivity and threshold voltage have been decreased with increasing concentration of nanoparticles (NPs). The continuous increase in dielectric anisotropy with increasing the concentration of NPs has also been observed. These results have been attributed to the trapping capability of free ions by TiO_2 NPs. Further, we focused on the study of photoluminescence (PL), UV absorbance and Fourier transformed infrared spectroscopy (FTIR) of pure and doped systems. The continuous increase in PL intensity without any shift in emission peak has been observed for doped systems. Enhancement in UV absorbance with increasing concentration of NPs has also been observed. The effect of NPs doping on molecular dynamics of NLC can be clearly seen by FTIR study. The results suggest that the TiO_2 NP-doped weakly polar NLC can have significant improved dielectric, electro-optical and optical properties. This makes the weakly polar NLC to be a potential candidate for many applications.

Keywords Nematic liquid crystal · TiO_2 nanoparticles · Dielectric anisotropy · Photoluminescence · UV absorbance

Introduction

NLCs have been widely studied over last few years, and it has variety of applications in our daily life. These applications include liquid crystal displays (LCD) [1], tunable lenses [2], retarders [3], filters [3], wave plates [3], diffractive optical elements [4], optical shutters [5] and smart windows [6]. Extreme work has been done by many researchers to improve the dielectric, electro-optical and optical properties of NLCs [7, 8]. With the advancement of technology, the enhancement in the basic properties of available NLCs is highly required. Two basic approaches have been adopted in order to modify these properties. One is to synthesize the new LC material according to required parameters and the other one is to alter the basic properties of LC materials by doping it with different nanomaterials like dye [9, 10], metallic NP [11, 12], semiconducting NP [13, 14], carbon nanotube [15] and some others [16–18]. Doping nanomaterials in the LCs to optimize the properties is potentially cost-effective, non-complicated and do not require great deal of efforts in comparison with synthesizing new one. Effect of doping nanomaterials on the properties of LCs depends on the type of nanomaterials used. Each nanomaterial has its own effect on the modification of properties of LCs [19]. Almost, all the liquid crystal-based devices have one thing in common that they are driven by electric field. Thus, the ions normally present in the liquid crystal in small quantity can alter the performance of LC devices [20]. Due to having an optical property of uniaxial crystal, NLCs have become dominating materials in the field of liquid crystal displays. Nowadays, the LCDs are expected to achieve high electro-optical properties with low power consumption as well. It

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has been observed that even a small fraction of impurity ions originating from LC material, sealing glue and alignment layer are strongly affecting the device performance. These impurity ions are deteriorating the quality of LCDs by reducing the voltage holding ratio, increasing threshold voltage, image sticking, gray-level shift and slowing down of response. There are also electro-optical devices like optical shutters and smart windows which rely on ions in liquid crystals [5, 6]. Despite, the negative effect of ionic contamination in display techniques, LCs with high ionic conductivity may be used in non-display applications [20, 21]. In recent years, doping nanoparticles in LCs has played an effective role in affecting the ionic impurities of LCs. Therefore, it becomes very important to understand how nanodopants can affect the ions in liquid crystals. Garbovskiy [22] discussed about the physical factors determining the type of the nanoparticle behavior and their effects on the concentration of ions in liquid crystals. For detailed quantitative analysis of the temperature effect in liquid crystals doped with NPs, reference can be made of [23].

The ionic species in the thermotropic liquid crystals are fully ionized, so the concentration of mobile ions does not depend on the temperature. But the LCs doped with nanoparticles exhibit different behavior. The concentration of ions in the NP-doped NLC systems becomes temperature dependence [24, 25]. The 100% pure NPs can only decrease the concentration of ions in the LC by means of adsorption or absorption process. The incorporation of contaminated NPs in the host LC can affect the concentration of ions in three different regimes: the purification, contamination and no change in the concentration of ions [26]. Generally for NP-doped LCs, the increase in the concentration of mobile ions with temperature is observed but under certain conditions, the concentration of ions in LCs decreases with increasing temperature. This effect has been modeled for pure and contaminated NPs systems [27].

TiO_2 NP is one of the most prominent NPs, which is highly employed to suppress the ionic effect. TiO_2 is an insulating NP that occurs in three different crystalline phases: anatase, brookite and rutile [28]. Among these three phases, anatase is chemically and optically active and therefore it is suitable to use as a doping material. The anatase structure is also preferred because of its high electron mobility, low dielectric constant and low density as compare to metallic or semiconductor NPs. The insulating NPs interact weakly or do not actually interact with NLC molecules [29]. Shcherbinin et al. studied the impact of TiO_2 NPs on nematic LC with different initial ionic contaminations. They explained that these types of NPs can be used to prevent uncontrolled ionic contamination that occurs during LC device production and utilization [30]. TiO_2 nanoparticles dispersion can actually change the relaxation parameters and dielectric anisotropy. Concentration and size of NPs play a crucial role in the alteration of LCs properties. Tang et al. [31] studied the electrical properties of NLC and observed that both the ionic concentration and diffusion constant have been reduced in TiO_2-doped NLC. Low-frequency dielectric spectroscopy can be used to explain the ionic transport behavior. Chen et al. [32] reported that insulating TiO_2 NPs, used as dopant in nematic LC, can reduce the ionic impurity contamination, thereby lowering the threshold voltage. Enhancement in electro-optical performance of NLC has been reported by Lee et al. [33]. Yadav et al. [34] explained the effect of TiO_2 NPs toward the suppression of screening effect in NLC. The photoluminescence study of nanoparticle-doped LC system promotes the better understanding of the interaction between nanoparticles and LC molecules. Photoluminescence behavior of NLC doped with TiO_2 NPs has been studied by Roy et al. [35], and the enhanced PL intensity for the doped system has been reported. Pathak et al. [36] observed the induced photoluminescence behavior in TiO_2 NP-doped NLC system.

Most of the studies on TiO_2 NP-doped LCs have been carried out on strongly polar NLCs by different research groups. Here is an attempt to explain the effect of Titania NPs on the dielectric, electro-optical and optical properties of weakly polar nematic LC. Weakly polar LCs is those which have weak dipolar strength; however, the dipole moment has a very little influence on the liquid crystalline properties [37]. In the present work, the dielectric permittivity and dielectric loss have been carried out to understand the charge storage and charge transportation phenomenon in doped NLC systems. To verify the ion trapping capability of TiO_2 NPs, the conductivity and dielectric anisotropy have also been studied. Threshold voltage of doped systems is decreasing with the increase in doping concentration which is also the consequence of ion trapping phenomenon. Photoluminescence behavior of TiO_2 NP-doped weakly polar NLC has also been obtained, and it is being absorbed that intensity of PL is continuously increasing with the concentration while its peak value is constant. UV–visible absorbance of pure and doped systems has been analyzed, and it is observed that the intensity of absorbance has been decreased in doped systems. Through the study of FTIR spectra, it is demonstrated that some physical and chemical changes are occurring in the doped systems.

Experimental details

The nematic liquid crystal used in the present study is D5AOB (4-4′dipentylazoxybenzene). The dipole moment of D5AOB is 1.7D, and the angle between dipole moment and the molecular axis is 64.9° [37]. This sample has been obtained from Flinton laboratory. D5AOB has the following phase sequence:

$$\mathrm{Cr}\xleftrightarrow{22\,^\circ\mathrm{C}}\mathrm{N}\xleftrightarrow{65\,^\circ\mathrm{C}}\mathrm{Iso}$$

Anatase TiO_2 NPs used as a doped material in this study have spherical shape and particle size 20–24 nm [38, 39]. These NPs have been purchased from Aldrich UK. The resistivity of anatase TiO_2 is 10^{15} Ω cm [40]. NPs have been used without any further purification. The planar aligned sample cells with ITO-coated glass plate are used to prepare sandwiched type cells. Firstly, the conducting layers are treated with adhesive promoter and then coated with nylon (6/6) to obtain a planar alignment. Thickness of the cell was fixed by placing a Mylar spacer (6 μm in our case) between the glass plates and then sealed with a UV sealant. The obtained empty cell is calibrated by using analytical reagent (AR) grade benzene and CCl_4 as standard reference for dielectric study. The composite has been prepared by mixing the Titania NPs with NLC D5AOB. The concentration of the dopant dispersed in the NLC was 0.05 wt%/wt (mix1), 0.1 wt%/wt (mix2) and 0.2 wt%/wt (mix3). For preparing the composites, the proper amount of TiO_2 NPs was first dispersed in propanol-2 and then the solution was ultrasonicated for 24 h to assure the proper dissolving of NPs. After that the appropriate volume concentration is mixed with fixed amount of NLC. This mixture is then repeatedly passed through the heating and cooling process until it is properly mixed. The empty planar cell is then filled with pure and doped LC above their isotropic temperature by capillary action.

The dielectric measurements of pure and Titania-doped NLCs were carried out by using an impedance gain/analyzer HP4194A. Instec hotplate (HCS-302) with an accuracy of ± .001 °C has been used to control the temperature of sample cell. Dielectric response is studied in the frequency range 100 Hz–40 MHz. The POM (Progress CT-3 Radical) has been used to record the optical texture of pure and composite systems. Carry eclipse fluorescence spectrophotometer (Agilent technology) is used to record the photoluminescence spectra of all the samples. A xenon lamp is used as an excitation source within the fluorescence spectrophotometer. All measurements are taken at room temperature, and the slit width of source is kept 5 nm. UV–visible spectrophotometer (ELICO, SL 210) has been used for UV–visible absorption study of pure and composite systems for wavelength range 190–500 nm. FTIR study in the wavelength range 400–4000 cm^{-1} was performed by Fourier transform of infrared spectrophotometer (IR affinity-1 Shimsdzu).

Results and discussion

To observe the effect of TiO_2 NPs on NLC, the POM images of planar aligned sample have been recorded. Figure 1 shows the textures of pure and three composite systems under cross-polarized condition. There is no visible aggregation in any texture, which confirms the homogeneous dispersion of TiO_2 nanoparticles in the nematic LC matrix. Homogeneous alignment of the doped systems can also be confirmed by seeing uniform color throughout the textures.

The dielectric study mainly emphasizes on the effluence of TiO_2 NPs on dielectric permittivity, dielectric loss and dielectric anisotropy of weakly polar NLC. The dielectric permittivity of the planar aligned cell has been depicted with the variation of frequency in Fig. 2 for the pure NLC and TiO_2-doped NLC at a constant temperature of 35 °C. It is clear from the figure that the dielectric permittivity is not showing any effective change in its value with the variation of concentration of TiO_2 NPs. This may be attributed to the non-conductive behavior of TiO_2 NPs. The NLC D5AOB has weaker dipole moment, and the contribution of TiO_2 NPs toward the dipole moment or polarization is very small. So due to addition of TiO_2 NPs in the NLC systems there has been no any such reinforcement in the net dipole moment. Figure 3 shows the variation of dielectric loss with frequency for pure and doped systems. It can be seen that the value of dielectric loss has been decreased for doped systems. The decrease in the value of dielectric loss is more prominent at lower frequency region where the free ionic charge plays an important role in the dielectric medium. The dielectric loss suffers two relaxation phenomenons: at high-frequency region, the observed relaxation is attributed to the well-known molecular reorientation process about short axis, while at lower frequency region the relaxation phenomenon is due to the ionic contribution [34]. In general, the dipole moment of the molecules makes an angle β with the long molecular axis. Therefore, longitudinal component (μ_l) and the transverse components (μ_t) (transverse to long molecular axis) have different projections in cases where $E\,||\,\hat{n}$ and $E\perp\hat{n}$. In both cases, two relaxation processes are observed. For $E\,||\,\hat{n}$, $\mu_{\mathrm{l}||}$ (rotation about short axis) and $\mu_{\mathrm{t}||}$ (rotation about long axis) contribute to the relaxation processes and for $E\perp\hat{n}$, $\mu_{\mathrm{l}\perp}$ (rotation about short axis) and $\mu_{\mathrm{t}\perp}$ (rotation about long axis) contribute to the relaxation processes. In NLC, the contribution from $\mu_{\mathrm{l}||}$ and $\mu_{\mathrm{l}\perp}$ is observed in MHz region, whereas the contribution from $\mu_{\mathrm{t}||}$ and $\mu_{\mathrm{t}\perp}$ is observed in 10^8–10^9 Hz region. For detail, the references can be made of [41, 42].

To verify this phenomenon, we have calculated the dc conductivity of pure and doped systems. DC conductivity

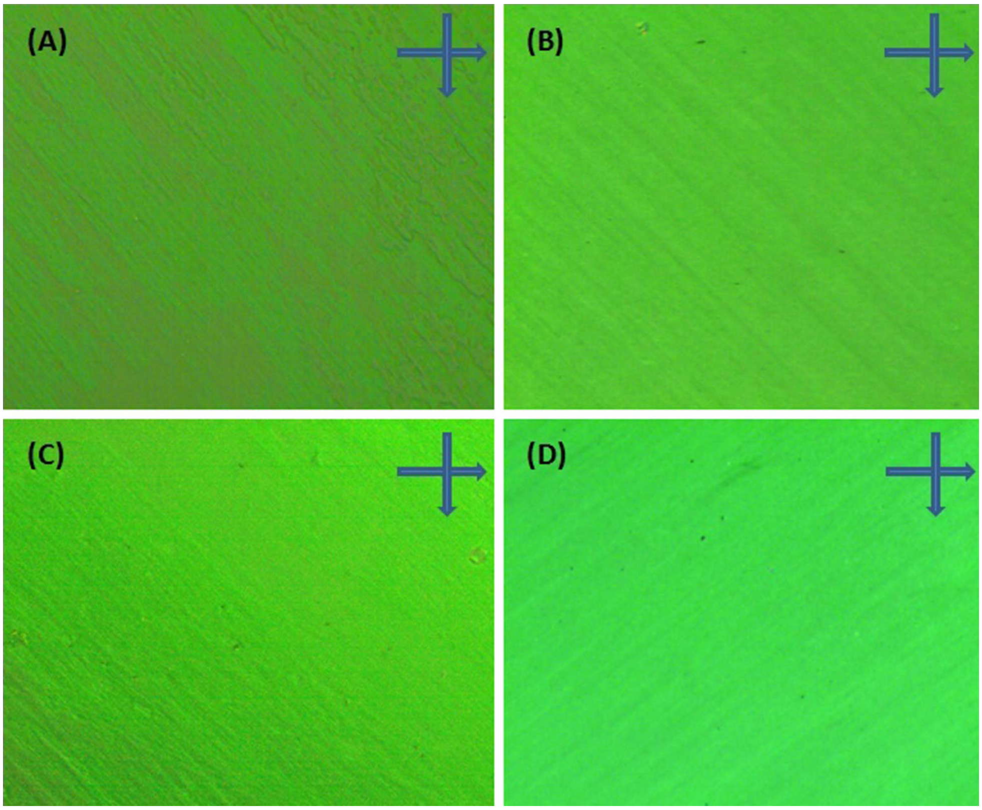

Fig. 1 Texture of pure (**a**), Mix1 (**b**), Mix2 (**c**) and Mix3 (**d**) under cross-polarized condition

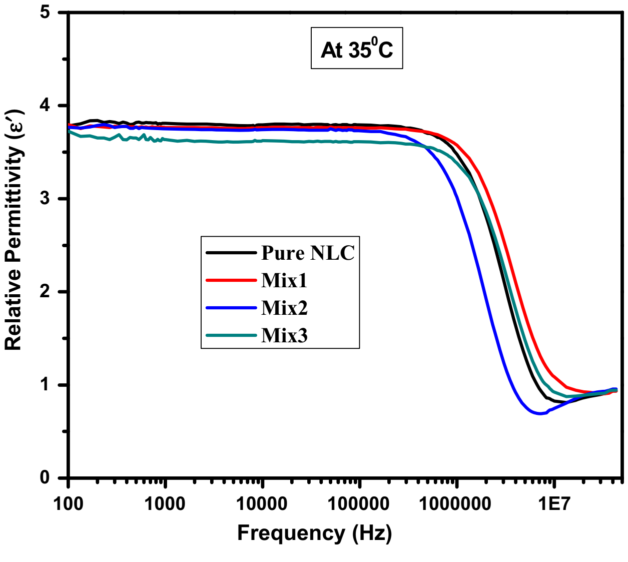

Fig. 2 Variation of relative permittivity with frequency for pure and three doped systems

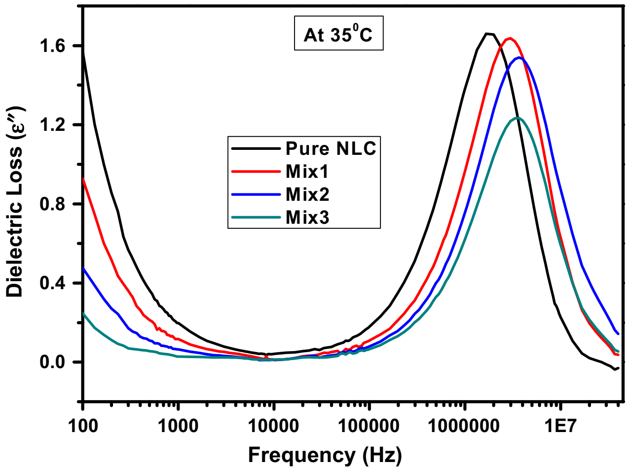

Fig. 3 Variation of imaginary part of dielectric permittivity with frequency for pure and doped systems

is caused by the movement of free ionic charges present in the LC medium, under the applied electric field. Figure 4 shows the variation of conductivity with temperature at 5 kHz frequency. The continuous decrease in the value of dc conductivity with the increase in concentration of Titania NPs has been observed, which clearly indicates that the numbers of mobile ions are decreasing as the concentration of NPs is increasing. The cause of such decrease is the adsorption of free ions on the surface of NP and this adsorption increases with the concentration of NP. However, the value of conductivity is increasing continuously with the increase in temperature. This may be attributed to the temperature dependence of ions concentration [23]. The variation in temperature is also influencing the mobility of

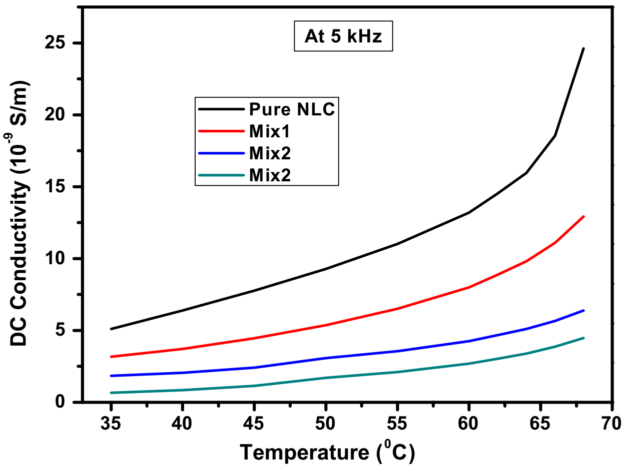

Fig. 4 Variation of dc conductivity with temperature for pure and doped systems

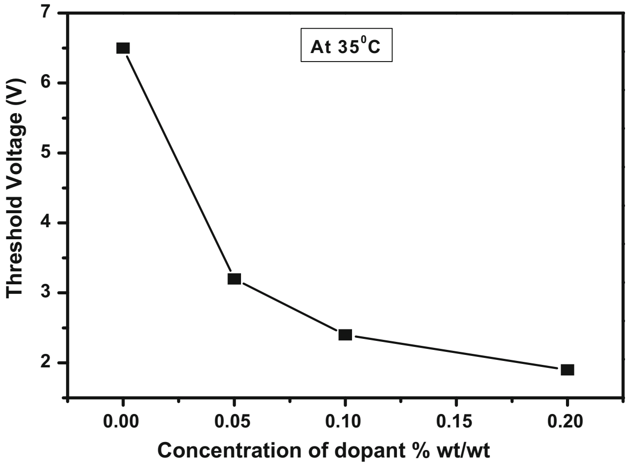

Fig. 5 Variation of threshold voltage with different concentrations for pure and doped systems

free ionic charges in liquid crystal. These two phenomenons simultaneously play an important role in deciding the behavior of conductivity of the LC system. At lower temperature, greater number of ions remain adsorbed at the NPs surface while as the temperature increases the ions trapped by NPs dispersed in LCs get released from the surface and the mobility of released ions continually increases with temperature. Thus, the decrease in ion trapping capability is caused by temperature-induced release of trapped ions by NPs in LCs [27].

The threshold voltage measurement of pure NLC and its composites directly supports the behavior of conductivity measurement. It also confirms the ion trapping behavior of TiO_2 NPs in NLC. The threshold voltage was determined by measuring the intensity of transmitted light by a given cell as a function of applied voltage. By changing the voltage stepwise and then detecting the change in the transmitted light intensity, we have determined the threshold voltage (the voltage, under which the transmitted intensity changed by the 10%) [43]. Figure 5 shows the variation of threshold voltage with concentration of NPs. It has been noticed that the value of threshold voltage is decreasing by increasing the concentration of NPs. The threshold voltage of pure NLC was found to be 6.5 V, which has been reduced to 1.9 V for mix3. During the experiment, when the field is applied across the cell the impurity ions present in LC begins to move toward the opposite side of the electrode and adsorbed by alignment layer (called the electric double layer). This phenomenon generates an electric field between the alignment layers which opposes the external applied electric field and reduces the effective electric field across the cell. This effect is known as screening effect which allows the liquid crystal molecules to experience relatively low voltage within the cell as compared to applied voltage. When the field is applied across the TiO_2 NP-doped systems, the free ion carriers transporting between the electrodes get trapped by TiO_2 NPs. As the numbers of NPs are increasing the free moving ions are decreasing, respectively, and hence the screening effect is also decreasing, this causes the molecule of NLC to experience less opposing field generated by free ions. Thus, when an external field is applied, the overall force on the NLC molecule increases and hence the threshold voltage decreases.

Figure 6 shows the variation of parallel and perpendicular components of dielectric permittivity with temperature, plotted at frequency of 5 kHz for pure and doped systems. The variation of dielectric anisotropy with concentration is shown in the inset of Fig. 6. Dielectric anisotropy is defined as the difference between the relative permittivity of planar and homeotropic alignments (i.e., $\Delta\varepsilon = \varepsilon_{||} - \varepsilon_{\perp}$). Since the NLC used here is weakly polar, so

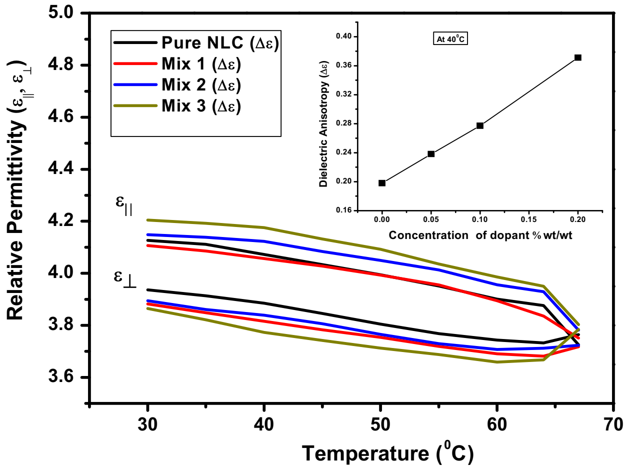

Fig. 6 Variation of parallel and perpendicular components of relative permittivity with temperature. Inset shows variation of dielectric anisotropy with different concentrations

the values of $\varepsilon_{||}$ and $\varepsilon_{\perp}$ are much smaller than the LCs having strong dipole moment. The NLC D5AOB has positive dielectric anisotropy; hence, the parallel component of permittivity is always greater than perpendicular component. This study shows that the value of $\varepsilon_{||}$ is decreasing and the value of $\varepsilon_{\perp}$ is increasing gradually with increasing temperature for all the systems. With increasing temperature, the disorder in the system increases and hence the difference between $\varepsilon_{||}$ and $\varepsilon_{\perp}$ decreases, that is, dielectric anisotropy decreases near transition temperature. It is also seen that the doping of TiO_2 NPs results in an increase in the parallel component of the permittivity and a decrease in the value of perpendicular component of the permittivity, leading to an increase in the dielectric anisotropy. The dielectric anisotropy effectively depends upon the angle β of the NLC system. The value of β is defined as the ratio of two components of the dipole moment given by $\beta = \tan^{-1}(\mu_t/\mu_l)$. Relative permittivity depends directly on the charge storage capacity of the system. The NPs effectively reduce the free ion density at the boundaries and increase the charge storage capacity which improves the orientation order of LC molecules. This increase in orientational order causes change in the effective polarization of the doped system and disrupts both the components of the dipole moment. In the present system, the contribution of the electronic polarizability to dielectric permittivity is greater in the direction along the molecular long axis than perpendicular to it. The dielectric anisotropy is inversely proportional to the threshold voltage, i.e., $\Delta\varepsilon = K_{11}\pi^2/\varepsilon_0 V_{th}^2$. The increased value of $\Delta\varepsilon$ can also be attributed to the decrease in the value of the threshold voltage with increasing concentration of NPs.

Figure 7 shows the UV–visible absorbance spectra of pure NLC and doped systems. From the graph, it is found that the absorbance is increasing continuously with the concentration of NPs. Maximum absorption occurs when electrons are excited from the valence band to the conduction band. In TiO_2, the valence band is composed of the 2*p* orbital of oxygen, while the conduction band is composed of 3*d* orbital of titanium [44]. The absorption peaks are due to electronic transition from 2*p* oxygen orbital to titanium 3*d* orbital. The enhancement in the absorbance is probably due to the coupling between the electromagnetic waves associated with the light source and low-energy phonons associated with the TiO_2 NPs. A slight shift in absorbance peak for doped systems has been noticed.

Figure 8 shows the normalized graph of photoluminescence spectra of the pure and TiO_2-doped systems. In order to know the effect of NPs on the PL spectra of D5AOB liquid crystal, the PL of LC doped with different NP concentrations was recorded on the almost same wavelengths range. The inset of Fig. 8 displays the PL spectrum of TiO_2 NP, which has been recorded before dispersing it in the NLC. The initial parameters of the instrument like slit width, excitation wavelength were kept constant for all measurements. All emission spectra are recorded at the room temperature. The undoped (D5AOB) and doped systems have different single band absorption peak (as mentioned above in UV absorbance graph). The luminescence study of pure and doped systems is performed by fixing the excitement wavelength at their maxima of absorbance. The PL intensity of NLC has been enhanced in the presence of TiO_2 NPs, and the enhancement strongly depends on the concentrations of NPs. The PL spectrum of pure system is showing very small PL intensity. It is clear from the spectra that as the concentration of NP increases from 0.05 to 0.2%, the increased intensity of PL has been observed. The emission band of TiO_2 NPs is much closer to the emission band of D5AOB molecules, so the increased

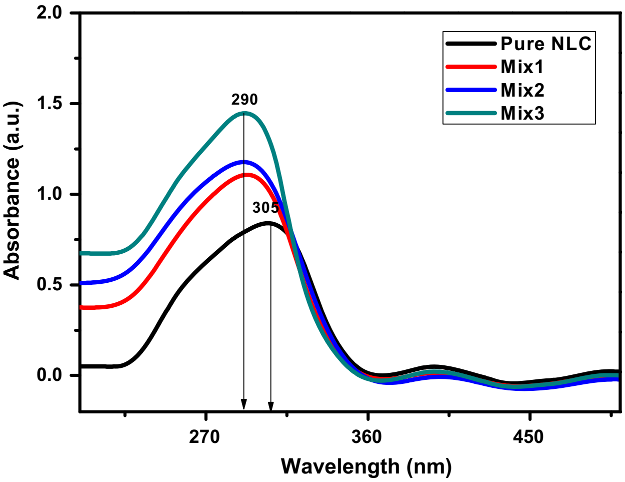

Fig. 7 UV absorbance graph of pure D5AOB and three doped systems

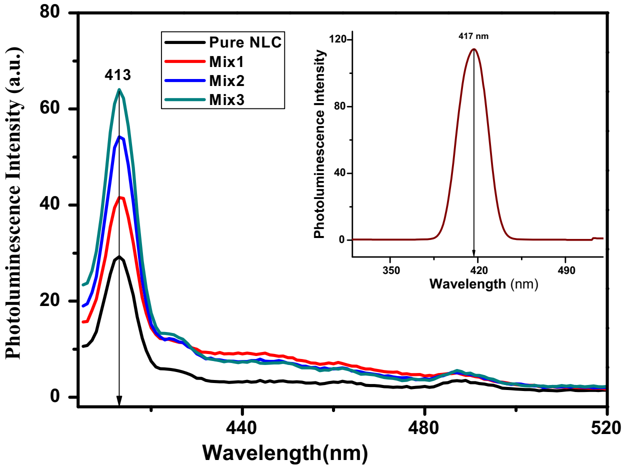

Fig. 8 Photoluminescence behavior of pure D5AOB and three doped systems

value of PL intensity of doped system may be due to constructive combination of emission of TiO_2 NPs and NLC molecules. In the spectral region, a secondary peak is also observed in all doped systems except pure one and is due to self-activated PL center [36].

The FTIR spectra of pure NLC and its composite systems have been shown in Fig. 9. The pure and the TiO_2-doped NLC were scanned for the wave number range of 1000–4000 cm^{-1}. It is clear from the figure that the transmittance for the doped systems is changing with the change in concentration of TiO_2 NPs. The change in FTIR spectra confirms the effect of doping the TiO_2 NPs on the molecular dynamics of NLC. The LC molecules consist of an aromatic central core and aliphatic side chains. The investigated sample D5AOB consists of N=N and N–O stretching vibrations. The most prominent bands in an aromatic ring are due to C–H and C=C in the region 1000–2000 cm^{-1}. The vibration band between IR spectral regions 2800 and 3000 cm^{-1} is probably due to N=N and N–O stretching vibrations.

Conclusions

The obtained results demonstrate the effect of doping TiO_2 NPs on the dielectric, electro-optical and optical properties of weakly polar nematic liquid crystal. Consistency in the variation of dielectric permittivity with frequency for both pure and doped systems reveals the negligible effect of TiO_2 NPs on the net dipole moment of NLC molecules. The value of dielectric loss is decreasing with concentration at lower part of frequency region which may be attributed to the trapping of free ions by TiO_2 NPs at its surface. The study also shows that the conductivity of doped NLC system has been decreased due to the reduction in the effective transported ion concentration. The decreased value of threshold voltage further confirms the suppression of screening effect in TiO_2 NP-doped systems. The dielectric anisotropy has been increased for all doped system. Increased value of $\Delta\varepsilon$ can be beneficial in the enhancement of electro-optical properties. For optical study, the photoluminescence, UV–visible absorbance and FTIR study has also been performed. The presence of TiO_2 NPs enhances the intensity of photoluminescence. This is due to constructive combination of emissions from NPs and NLC molecules. The UV absorbance is increasing continuously with the increase in concentration of NPs. This enhancement in the UV absorbance is attributed to coupling between electromagnetic wave and phonons. FTIR study confirms the effect of TiO_2 NPs on the molecular dynamics of NLC system. On the basis of these results, we conclude that the TiO_2 NPs are promising materials for enhancing the characteristic properties of D5AOB NLC. The most engrossing result of this study is the reduction in ionic contaminations present in NLC material, which can be highly beneficial for display applications.

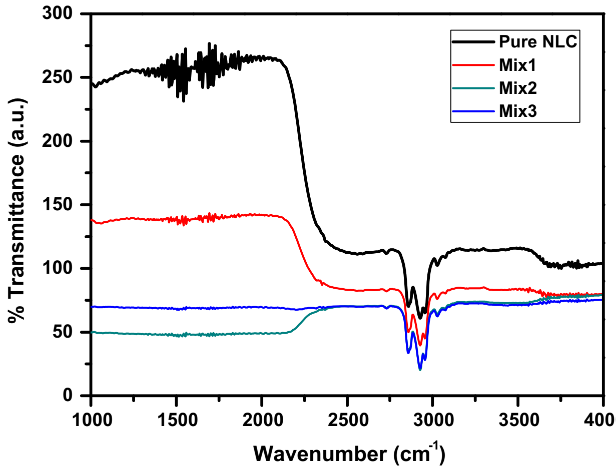

Fig. 9 FTIR spectra of pure D5AOB and three doped systems

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