

Effect of different concentrations of dopant titanium dioxide nanoparticles on electro-optic and dielectric properties of ferroelectric liquid crystal mixture

Pradeep Kumar, Avinash Kishore, Alok Sinha*

Department of Physics, Indian Institute of Technology Delhi, 110016, India

*Corresponding author. Tel: (+91) 11-26596003; E-mail: aloka@physics.iitd.ac.in

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ABSTRACT

In the present study, the insulating titanium dioxide (TiO_2) nanoparticles were dispersed in two different concentrations of 0.5 wt % and 1.0 wt % in pure ferroelectric liquid crystal (FLC) mixture, W206E. The effects of different concentrations of dopant TiO_2 in W206E for electro-optical and dielectric properties have been studied. The optical microscopy measurements clearly show the isotropic transition temperature of both the doped samples slightly increases by about $1 \sim 2^\circ\text{C}$ as compared to the pure sample. Further, with the increasing concentrations of dopant TiO_2 , the value of spontaneous polarization decreases. The value of dielectric permittivity also decreases for both the doped samples and this decrease in the value of permittivity is more prominent with increasing concentrations of TiO_2 at the lower frequencies. The dielectric measurements also confirm the presence of Goldstone mode in Smectic C^* phase in pure as well as in both the doped samples. The conductance measurements confirm the decrease in conductivity of doped samples as compared to the pure W206E FLC. This study may help in improving the transition temperature and reducing the impact of free ionic charge impurities on various physical properties of liquid crystal host by screening the free ions using TiO_2 nanoparticles as dopant. Copyright © 2016 VBRI Press.

Keywords: Ferroelectric liquid crystal; titanium dioxide nanoparticles; transition temperature; spontaneous polarization; dielectric behavior.

Introduction

Ferroelectric liquid crystals (FLCs), have attracted the significant attention of researchers since it has been discovered in 1975 by Meyer [1] due to their physical properties which can be used in various electro-optical applications such as displays, spatial light modulators for optical processing and optical storage [2-4]. The new and modern devices require liquid crystals (LCs) with improved properties. It is difficult to synthesize new liquid crystal materials with enhanced properties due to the complexity in the synthesis of new LC materials. It has been observed that the doping of nanoparticles in different FLC mixtures can enhance their physical properties like frequency modulation response, faster switching time, non-volatile memory effect and lower operating voltage [5-7] which may be useful in the designing and fabrication of modern and sophisticated devices. The doping of different types of nanoparticles i.e. metallic, semiconducting, dielectric, ferroelectric, ferromagnetic in liquid crystal has been studied by different research groups. Doping of small amount of silica nanoparticles in FLCs has improved the switching time and memory effect [8]. FLCs doped with metal nanoparticles such as gold, palladium, platinum enhances the switching

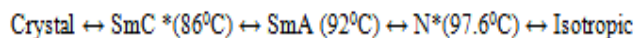
time and luminescent properties [9, 10]. Semiconducting nanoparticles such as zinc oxide when dispersed in FLCs, reduce the threshold voltage and improve the optical contrast of the display devices [11]. Doping of ferroelectric barium titanate (BaTiO_3) nanoparticles results in the enhancement of the switching time, transition temperature and decrement in the polarization of FLCs [12,13]. It has been observed that, the impurity ions present in LCs are responsible for the lower contrast, slower response time and high threshold voltage. The addition of insulating nanoparticles such as diamond powders in LCs reduces the impurity ion concentration and thereafter decreases the transient current and threshold voltage. A significant amount of research has been done on TiO_2 nanoparticles due to its existence in different phases and therefore exhibit different physical properties which may be utilized for the design and fabrication of novel photonic devices [14] and for other applications such as gas sensors [15]. Apart from this TiO_2 nanoparticles are the best candidates for the suppression of free ionic charges present in liquid crystal due to trapping of free ions by dopant TiO_2 nanoparticles. A very small amount of ionic impurities are always present in each liquid crystal, it may originate from different sources i.e. during the synthesis of liquid crystal, chemical decomposition of liquid crystal and aging etc. These ionic

impurities always degraded the performances of liquid crystal based devices. The decrement in contrast ratio, enhancement in operating voltage and slower response time are some of the common problems arise due to the presence of free ions in liquid crystals. The doping of TiO₂ nanoparticles in nematics liquid crystal has reduced the ion concentration and hence ion current and voltage holding ratio [16, 17]. While in case of FLC the doping of TiO₂ has reduced the conductivity [18]. It has been observed by Tang *et al.* [19], that the doping of anatase titania (TiO₂) nanoparticles in nematics liquid crystals lowers the ionic concentration, diffusion constant, activation energy and improves the voltage holding ratio. Recently, Gupta *et al.* [20] reported the decrease in response time, conductivity and increase in spontaneous polarization of TiO₂ doped FLC. A significant amount of research work has been devoted in nanodoping of liquid crystals but still there are many conflicting results and explanations and hence nanodoping of liquid crystals need further systematic investigations. In this paper, we report the effect of doping of two different concentrations of insulating TiO₂ nanoparticles in pure FLC mixture acronym W206E on the transition temperature, spontaneous polarization, dielectric behavior and conductivity of TiO₂ doped W206E samples.

Experimental

Materials

The FLC mixture W206E utilized for the present study has the following phase sequence;



The TiO₂ nanoparticles with particle size 18~24 nm, obtained from Sigma Aldrich USA, were used to prepare two different concentrations of TiO₂ nanocomposites W206E + 0.5 wt % TiO₂ and W206E + 1.0 wt % TiO₂.

Synthesis of liquid crystal nanocomposites and cell fabrication

To prepare each nanocomposite sample, TiO₂ nanoparticles in fixed proportion were first mixed in pure W206E, and then chloroform was added to the W206E + TiO₂. Thereafter the mixture was sonicated using ultrasonicator till the solution was visibly homogeneously mixed and after that the solution was left for 3~4 hours at temperature 40~ 45°C or till the chloroform evaporated completely in order to get nanocomposites of the respective concentrations.

The liquid crystal cells were prepared using indium tin oxide (ITO) coated glass plates. The square patterns on ITO- coated glass substrates were created using optical lithography. Thereafter, a thin layer of nylon solution was deposited and antiparallel rubbing was performed on the pairs of glass substrates to get homogeneous alignment and finally a Mylar spacer of uniform thickness of 3.5 micron was sandwiched between the glass plate pair to obtain a cell. The pure and both doped liquid crystal samples at their isotropic temperatures were introduced by capillary action in the self-designed liquid crystal cells.

Characterizations

The filled cells were studied at different temperatures for different liquid crystal phase's present, spontaneous polarization and dielectric behavior. The temperature controller INSTEC mK-1000 was used for all these observations. The optical polarizing microscopy measurements were carried out using OLYMPUS BX-51. The spontaneous polarizations were determined by current reversal technique using function generator Tektronix AFG 3021 and oscilloscope Tektronix TPS 2024. The molecular and collective dynamics were studied by dielectric spectroscopy using Agilent E4980A LCR meter in the frequency range of 20 Hz to 2MHz.

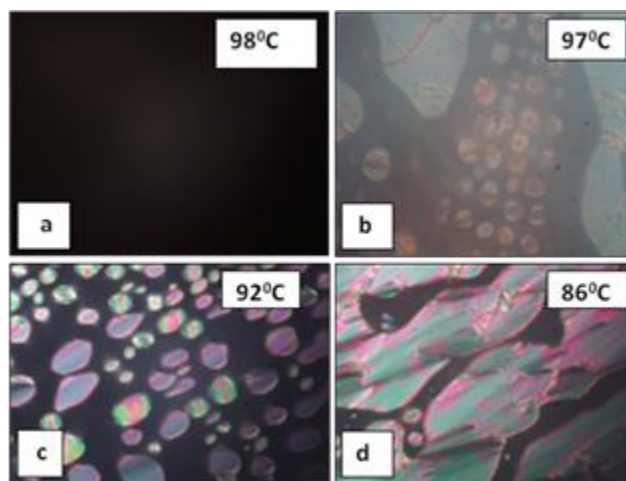


Fig. 1. Optical micrographs of pure W206E sample.

Results and discussion

Optical microscopy

Optical microscopy measurements were performed to investigate the transition temperature and different phases present in pure as well as both TiO₂ doped samples of W206E FLC. **Fig. 1** shows the different optical micrographs observed for pure W206E. It is clear from **Fig.1 (a)**, that the isotropic temperature of pure W206E sample is around 98 °C. The cholesteric droplets separating out from the isotropic melt along with the oily streaks texture shown in **Fig. 1(b)** confirms the presence of chiral nematic (N*) at 97 °C. The growth of focal conic textures in **Fig. 1(c)** shows the existence of Smectic A (SmA) phase at 92 °C. The fan shaped structure and break up of focal conic structure at transition temperature shown in **Fig. 1(d)**, confirms the presence of Smectic C (SmC*) at 86 °C [21].

Fig. 2 shows the optical micrographs for W206E + 0.5wt % TiO₂ sample. It has been observed that the isotropic temperature as shown in **Fig. 2(a)**, of this doped sample slightly increases to 99~100°C as compared to the pure W206E sample for which the isotropic temperature was 98 °C. The oily streaks present in **Fig. 2(b)**, confirms the presence of N* phase at 98 °C. The appearance of focal conic texture shown in **Fig. 2(c)** at 94 °C corresponds to the SmA phase while **Fig. 2(d)** corresponds to the SmC* phase at 87 °C [21]. **Fig. 3** represents the optical micrographs for W206E + 1.0 wt % TiO₂ sample. The isotropic temperature for this doped sample is also around 100 °C. The

cholesteric droplets present in **Fig. 3(b)** shows the presence of N* phase at 98 °C. The typical fan shaped structure shown in **Fig. 3(c)**, confirms the presence of SmA phase while in **Fig. 3(d)**, the focal conic texture with slight striation along fans represents the SmC* phase [21].

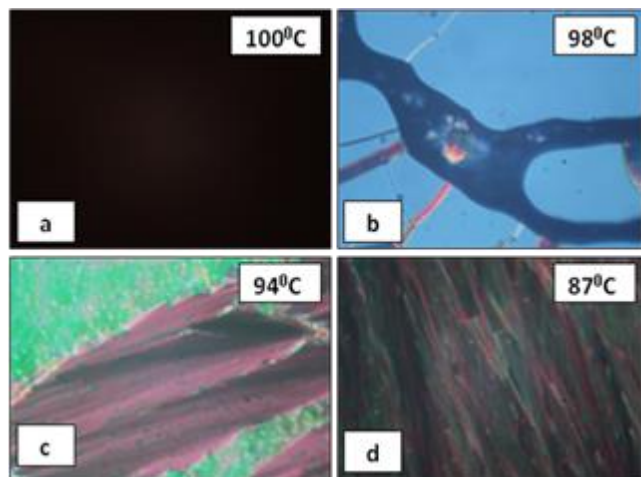


Fig. 2. Optical micrographs of W206E + 0.5wt %TiO₂ sample.

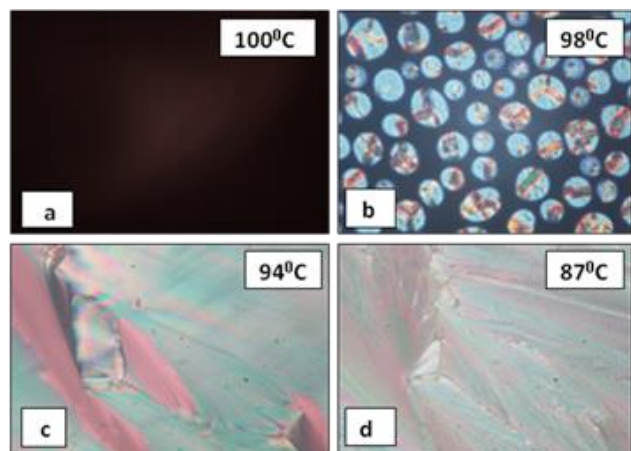


Fig. 3. Optical micrographs of W206E + 1.0 wt% TiO₂ sample at different temperatures.

The optical microscopy measurements confirms a slight increase of 1~2 °C in the transition temperature of both the doped samples. The increase in isotropic temperature of liquid crystal nanocomposites was also reported earlier by various research groups. Lee *et al.* [22], reported a small increase in isotropic temperature of a nematic liquid crystal when it was doped with silica nanoparticles and explained qualitatively this was because of the orienting influence of the non -uniformly distributed silica nanoparticles in the nematic liquid crystal and such orienting influence stabilizes and enhances the local orientational order of liquid crystal and hence results in the increase in transition temperature. Li *et al.* [23] reported the increase of isotropic temperature of the order of 38.9 °C, when the nematic liquid crystal was doped with BaTiO₃. But later on, they modified and reported that this increase in transition temperature of 38.9 °C was due to the presence of heptane in the liquid crystal nanocolloid. However, the actual

increase in transition temperature was 9 °C, which was attributed to the influence of the ferroelectric nanoparticles on the orientational coupling in LC [24]. The effect of doping of carbon nanotubes on the phase transition of nematic liquid crystal was studied by Duran *et al.* [25]. They have reported a positive shift in the isotropic temperature of doped nematic liquid crystal and attributed this as anisotropic alignment of liquid crystal molecules along the carbon nanotubes bundles.

In this present study we have also found a slight increase of 1~2 °C in the transition temperature as well as the temperatures corresponding to the different phases present in both the doped samples. To ensure that there is no solvent left in the liquid crystal nanocomposites, we have evaporated the ferroelectric nanocomposites at a temperature higher than the boiling point of chloroform repeatedly. We have also repeated the optical polarizing microscopy measurements several times and found a slight increase in transition temperature as well as in the temperature corresponding to the different phases present in both the doped samples. This may be attributed to the strong interaction between W206E molecules and the non-uniformly distributed TiO₂ nanoparticles which result in anisotropic alignment of W206E molecules induced by TiO₂ nanoparticles which improves the local interactions and orientational order in W206E molecules. This results in the increased isotropic temperature as well as the temperature corresponding to different phases present in both the liquid crystal nanocomposites.

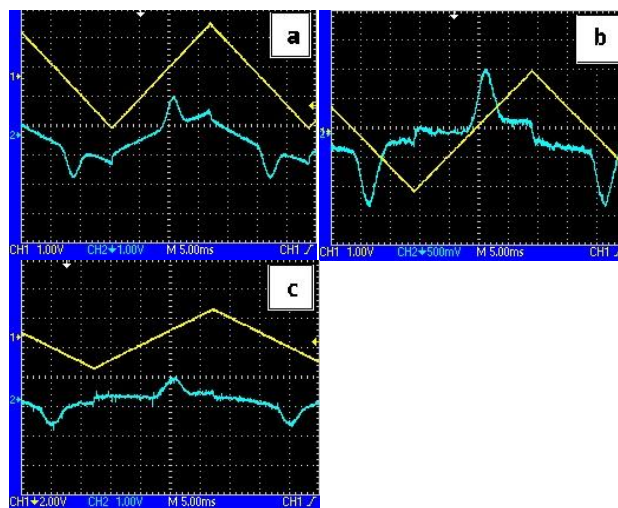


Fig. 4. Oscillogram images for a) Pure W206E, b) W206E + 0.5wt%TiO₂ and c) W206E + 1.0wt% TiO₂.

Spontaneous polarization

The spontaneous polarization P_s is the main characteristic of FLCs and it is one of the important physical parameters in FLC to investigate the transition from the lower SmC* phase to the high temperature SmA phase at a particular transition temperature T_c , where P_s disappears with increasing temperature. The value of spontaneous polarization strongly depends on the cell thickness, frequency and the concentration of ionic charges [26, 27]. The spontaneous polarization was calculated by using the following relation;

$$P_s = \frac{1}{2R_s A} \int \Delta V dt \quad (1)$$

where, R_s is the resistance connected in series with the sample, A is the area of the square pattern created on the cell and ΔV is the excess voltage because of the polarization reversal. The oscillogram images for pure and doped samples corresponding to the maximum value of spontaneous polarization are shown in **Fig. 4**.

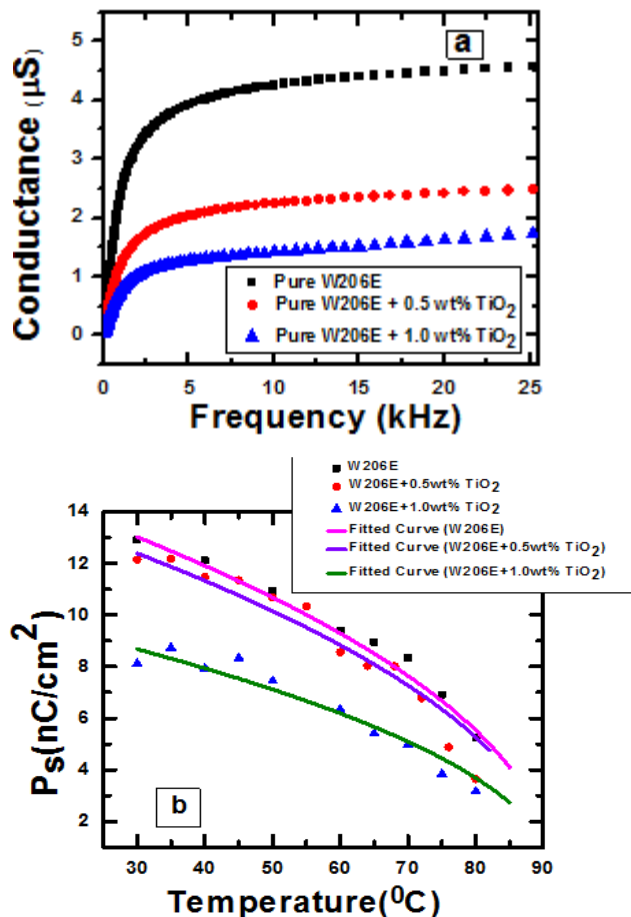


Fig. 5. (a) Conductance vs. Frequency for Pure W206E and both TiO₂ doped samples, (b) Variation of spontaneous polarization with temperature for Pure and doped samples.

The maximum value of P_s for pure W206E FLC at 30 °C is 13.00 nC/cm², while it is 12.20 nC/cm² for W206E + 0.5 wt % TiO₂ and 8.73 nC/cm² for W206E + 1.0 wt % TiO₂ at 35 °C. A similar type of decrement in the value of P_s with increased concentration of LiNbO₃ in FLC was reported by Shukla *et al.* [28] and explained the decrease in the value of P_s by two different mechanisms. First, the distortion of polarization vector by the dopant LiNbO₃ nanoparticles and also the antiparallel correlation between LiNbO₃ nanoparticles and FLC molecules reduces the value of P_s . Secondly, the captured ions screen the net dipole moment contribution and hence reduction in the value of P_s of the nanocomposites is observed.

Chaudhary *et al.* [29] have doped ZnO nanoparticles in FLC W206E in different proportions and found that there is an increase in P_s value upto 0.3 wt % of ZnO nanoparticles doped samples and reported this is due to the

dipole - dipole interactions between ZnO nanoparticles and FLC molecules. The ZnO nanoparticles add up the dipole moment into the FLC material and hence there is a corresponding increase in P_s value. Unlike Shukla *et al.* [28] results for P_s value, Gupta *et al.* [20] recently reported a small increase in P_s value, when they doped FLC with only one concentration of 1.0 wt % TiO₂ nanoparticles and explained, TiO₂ nanoparticles are suppressing the screening effect by trapping free ion charges and because of this the polarization component of FLC molecules align more preferably along the field direction which cause increment in the value of P_s .

In our case, we have found a decrease in the value of P_s when W206E sample was doped with insulating TiO₂ nanoparticles in different proportions. This decrease in P_s of both the doped samples as compared to pure W206E may be explained on the basis of trapping of free ions phenomenon as reported earlier by Shukla *et al.* [28]. The TiO₂ nanoparticles capture the free charge ions present in pure W206E. These captured ions enhance the local electric field in doped samples as compared to the local electric field present within pure W206E sample. Further, these trapped free charge ions also reduce the value of P_s of doped samples by shielding the net dipole moment effect and hence reduction in P_s value of both the doped samples has been observed [28]. With increase in concentration of TiO₂, the concentration of free charge ions is lesser and trapping of free charge ions is more which may further reduce the value of P_s to 8.73 nC/cm² in case of 1.0 wt % TiO₂ doped sample. The suppression of free ion charges can be explained on the basis of decrease in the conductivity of doped samples. To substantiate this, conductance measurements have been carried out. The conductance for pure W206E and both the doped samples is shown in **Fig. 5(a)**. It is clear from the graph that with increase in concentration of dopant TiO₂, the value of conductance decreases due to the free ionic charge trapping phenomenon and hence conductivity decreases for both the doped samples as compared to the pure FLC. The contribution of free charge ions is maximum at lower frequencies. These TiO₂ particles trap the free ion charges present in pure W206E and consequently their movements towards liquid crystal cell walls result in the decrease in conductivity of both the doped samples.

The variation of P_s with temperature for all the three samples is shown in **Fig. 5(b)**. It is clear that, with increase in temperature, the P_s is going down. The experimental data are fitted according to the following equation;

$$P_s = P_o (T_c - T)^\beta \quad (2)$$

where, T_c is the transition temperature, P_o and β are the fitting parameters, from the mean field theory the value of β is 0.50. The value of β obtained from equation (2) for pure W206E sample, W206E + 0.5 wt % TiO₂ and W206E + 1.0 wt % TiO₂ are 0.54, 0.52 and 0.51 respectively, which are greater than 0.50, indicates a second order transition corresponding to ferroelectric to paraelectric in all three samples [30]. The second order phase transition is corresponding to a symmetry breaking phenomenon. At T_c , the lowest- symmetry phase i.e. ferroelectric phase (SmC*) becomes high-symmetry paraelectric phase which

causes the value of P_s to go to zero at T_c . The value of P_s decreases with the increase in temperature and at T_c , it becomes zero. The fitting parameters P_o and β are given in **Table 1**.

Table 1. The fitting parameters P_o and β .

System	P_o (nC/cm ²)	β
Pure W206E	1.50	0.54
W206E + 0.5wt% TiO ₂	1.46	0.52
W206E + 1.0wt% TiO ₂	1.18	0.51

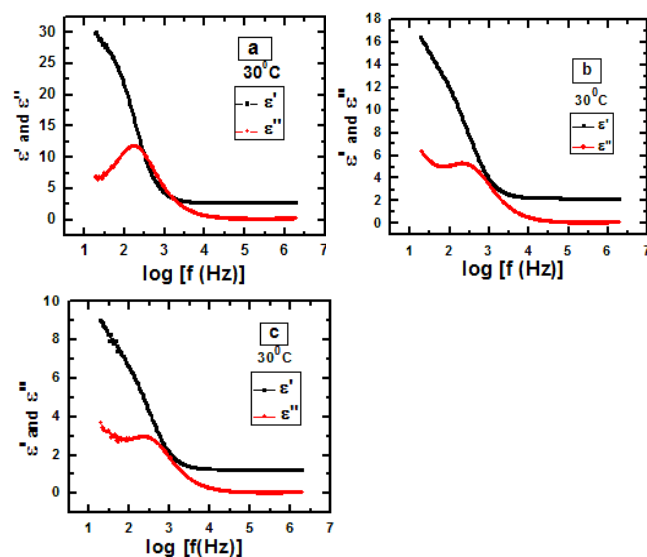


Fig. 6. ϵ' and ϵ'' vs. $\log f$ for a) pure W206E, b) W206E + 0.5 wt% TiO₂ and c) W206E + 1.0 wt% TiO₂ in SmC* phase.

Dielectric spectroscopy

Dielectric spectroscopy is directly related to the macroscopic polarization of the material. There are various polarization mechanisms or relaxation processes. Apart from these relaxation processes, the two low frequency collective modes, the Goldstone mode because of fluctuations in the azimuthal angle ϕ and the soft mode due to the change in the tilt angle Θ may be present. Therefore, to study the different molecular and collective modes present, the dielectric measurements were carried out in pure and both doped samples in the frequency range of 20Hz to 2MHz. **Fig. 6**, shows the variation of ϵ' and ϵ'' with frequency for pure as well as both doped samples at 30 °C, at which the sample is in SmC* phase. The dielectric loss i.e. the peak appearing in ϵ'' as shown in **Fig. 6(a-c)**, appears at frequency 178 Hz, 212 Hz and 238 Hz for pure W206E, W206E + 0.5 wt% TiO₂ and W206E + 1.0 wt% TiO₂ doped samples respectively corresponds to the Goldstone mode present in these samples. The Goldstone mode can be further confirmed by the application of DC field [31]. With the application of DC bias the peak will be suppressed because of the unwinding of helix in SmC* phase. The suppression of dielectric loss peak with increasing DC bias from 0V to 5V as shown in **Fig. 7** confirms the presence of Goldstone mode in SmC* phase in all the three samples.

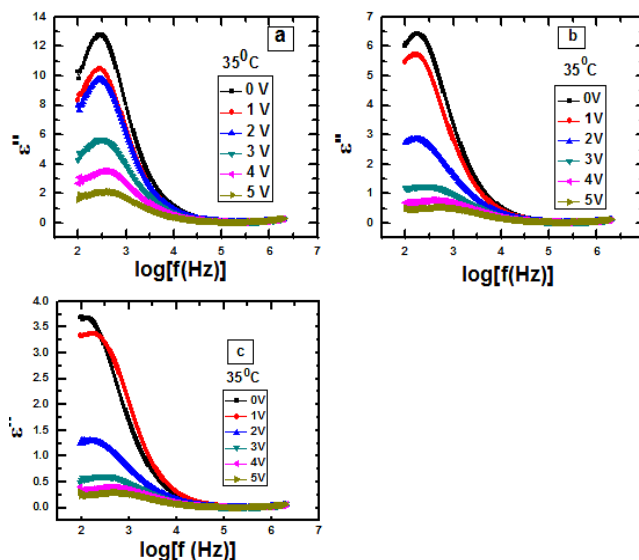


Fig. 7. Effect of DC bias on Gold stone mode of (a) pure W206E, (b) pure w206E+0.5wt % TiO₂ and (c) pure w206E+1.0wt % TiO₂.

The effect of the doping concentration of TiO₂ on the dielectric properties of pure sample with the variation in frequency has been studied. It is clear from **Fig. 6** that, with increasing doping concentration the value of dielectric permittivity ϵ' is decreased. **Fig. 6(a-c)**, clearly show that initially the value of ϵ' for pure and both doped samples decreases with the increasing value of frequency and further at higher frequencies it remains almost constant and is independent of the increase in frequency. The dielectric permittivity of pure W206E is high in lower frequency region, however the dielectric permittivity observed for TiO₂ doped W206E samples is low. In lower frequency region (<1.5 kHz) the dielectric permittivity of the liquid crystal mainly depends upon the space charge polarization and the orientational polarization contribution. Pure W206E also has free ions present in the liquid crystal matrix. With the application of electric field, non-uniform distributions of these free ions occur, which depends mainly on the frequency of the applied field and hence it has significant contributions in both the real and imaginary part of the dielectric permittivity. When pure W206E is doped with TiO₂ nanoparticles, the orientation of nanoparticles was such that the induced dipole moment of TiO₂ nanoparticle is screening the total inherent dipole moment of W206E molecule and hence the dielectric permittivity decreases for nanocomposites. With increasing concentration of TiO₂ this effect is more and hence the observed value of dielectric permittivity for 1.0 wt % TiO₂ doped sample is lower as compared to 0.5wt % TiO₂ doped W206E. The value of ϵ' is 29.73 for pure W206E sample, while it is 16.33 and 8.99 for W206E + 0.5 wt %TiO₂ and W206E + 1.0wt % TiO₂ samples respectively.

The variation in the value of ϵ'' due to change in the concentration of dopant TiO₂ with frequency is also depicted in **Fig. 6**. The maximum value of ϵ'' for pure W206E sample is 11.78 at 178 Hz, but with increasing concentration of TiO₂ the maximum value of ϵ'' decreases and it is 5.26 at 212 Hz and 2.94 at 238Hz for 0.5 wt % and 1.0 wt % TiO₂ doped W206E samples respectively. Since the free ionic charge contribution is maximum in the lower

frequency region and hence this decrease in the value of ϵ'' at the lower frequencies may be attributed to the decrement in the conductivity of respective samples. Further, with the increasing concentration of dopant TiO_2 , the free ion charge trapping phenomenon is more which further subsequently reduces the conductivity of doped samples. The observed decrease in the value of conductivity represented in Fig. 5(a) with increasing concentration of TiO_2 supports this fact.

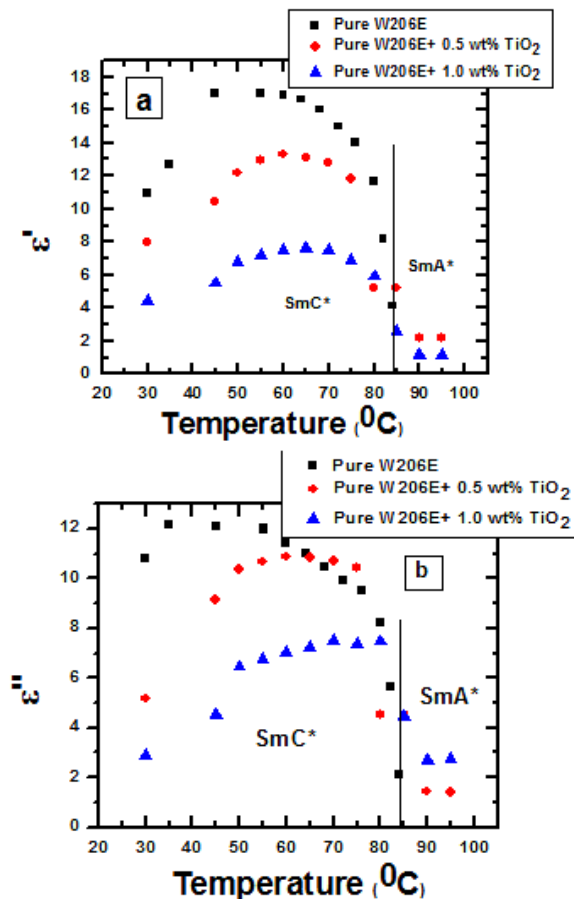


Fig. 8. Variation of (a) ϵ' with temperature and (b) ϵ'' with temperature at 300 Hz.

The variation of both real and imaginary part of dielectric constant with the temperature for pure and both doped samples at 300 Hz of frequency is shown in Fig. 8. The value of ϵ' and ϵ'' in the SmC^* region decreases with the increase in the doping concentration of TiO_2 . The sharp increase in the value of dielectric permittivity ϵ' at the transition temperature of SmC^* - SmA^* phase is because of the presence of the helical structure in the SmC^* phase. As soon as the temperature increases beyond this transition temperature, the value of dielectric permittivity decreases sharply because of the unwinding of the helix in all the three samples [29].

Conclusion

The effect of two different concentrations of insulator TiO_2 nanoparticles in pure W206E has been studied for the different phase's present, spontaneous polarization and the dielectric behavior of both the doped samples. From

polarizing microscopy, it has been observed that the transition temperature of both the doped samples is increased by $1\sim 2^{\circ}\text{C}$ as compared to the pure sample. This increase in transition temperature is attributed to the strong anisotropic interactions between W206E molecules and non-uniformly distributed TiO_2 nanoparticles, which enhances the local interactions and orientation order in W206E molecules and hence increases the isotropic temperature by $1\sim 2^{\circ}\text{C}$. The decrease in spontaneous polarization for both the doped samples as compared to the pure W206E sample is due to the ion capturing phenomenon which screens the net dipole moment and hence the P_s value. The conductivity measurements further show the decrease in conductivity with increased concentrations of TiO_2 . This is because of the trapping of free charge ions by TiO_2 nanoparticles at lower frequencies. Dielectric measurements also confirms the presence of Goldstone mode in the SmC^* phase. The decrease in the value of dielectric permittivity and dielectric loss with increasing concentration of dopant TiO_2 at lower frequencies as compared to pure W206E sample is also attributed to the free ionic charge capturing phenomenon. The present study may help to improve the transition temperature of different phases present in the sample, minimizing the free ionic charge impurities effects of existing FLC by the proper selection of insulating nanoparticles like TiO_2 .

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