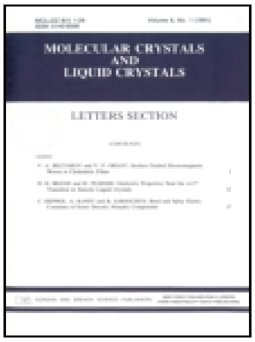

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Dielectric Study on the Alignment or Orientation of *N*(4-*n*-heptyloxy benzylidene)4′-*n*-butylaniline and the Anomalous Dielectric Behavior in the Smectic G Phase

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ABSTRACT

The present article reports on the alignment or orientation of a particular compound N(4-n-heptyloxy benzylidene)4′-n-butylaniline through dielectric study. The effect of using two different alignment layers (for homogenous alignment) and doping silver nanoparticles (0.25% by weight) on the behavior in dielectric property with varying temperature and biasing electric field was studied in terms of liquid crystal alignment or orientation using the mechanism proposed by earlier workers. The study includes the anomalous dielectric behavior in SmG phase by presenting a technique for the analysis of the absorption process, which exists only in SmG phase at fixed frequency while varying temperature, is also presented.

KEYWORDS

Dielectric permittivity; alignment; dielectric absorption; relaxation time

1. Introduction

In studying many physical properties, it is necessary to prepare highly oriented liquid crystals (LC) for the basic understanding of interfacial phenomena and also for the fabrication of electro-optical devices [1]. The alignment of the LC molecules are achieved through a number of ways such as rubbed polymer films [2], oblique evaporation technique [3,4], Langmuir-Blodgett films [3,5], photoaligned LC [6], ion beam [7], and other methods which produce a grooved surface [8,9]. Following these studies, several models were proposed on the mechanism of LC alignment. In the early models, the existence of microgroves at the surface of the alignment film [10,11] was assumed. It was suggested [12] that long-range anisotropic elastic effects induced by grooved surfaces were responsible for LC alignment. However, a different report [13] that the surface structures produced upon rubbing a polymer layer may not be responsible for the LC alignment was given. It may be noted that all these interpretations were based on measurements of surface morphology on a macroscopic length scale (micron). In another case, Crawford et al. [14] have examined the role of surface coupling agents on LC alignment and the ordering mechanisms at the LC-solid interface using deuterium nuclear magnetic resonance technique. They concluded that the interactions between the long alkyl chains and the LC molecules are predominantly steric, resulting in homeotropic ordering,

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while strong interactions between the alumina surface and the core of the LC molecules dominate in the case for short chain lengths, and thereby led to planar ordering [15]. Interestingly, it was reported that untreated structureless walls are known to align the nematic director in a planar axial manner at their surfaces [14] possibly as a result of entropic considerations; but dipolar LC molecules tend to align with their dipoles perpendicular to the surface even if the surface is structureless and free of charge [16]. A general phenomenological theory explaining LC alignment by a molecular interaction that links the orientational order of the LC system to the molecular order at the surface of the alignment substrate may also be found in [17]. However, it would be almost impossible to list all the reports on the LC alignment and mechanism.

It is surprising that except for an earlier work [18] where dielectric technique was used to check LC alignment, discussion or studies on the alignment mechanism were hardly pursued with this method. In the wake of recent interest [1] on the alignment of the LCs molecules, an attempt is made to investigate the alignment mechanism of the LC molecules using dielectric technique.

Composite materials comprised of nanosized particle dispersions in LC have drawn a lot of interest in recent years [19,20] because the presence of the nanoparticles introduces or modifies many properties of the host LCs. It is known that the interaction of particles in the bulk [21,22] and at the surfaces [23] of LCs provide a rich variety of new physical phenomena of interest to both fundamental and applied science of LCs. For example, a spherical particle immersed in LC with a uniform far-field director orientation has been reported to create distortions that are usually of dipolar or quadrupolar symmetry [24]. Anisotropy of molecular interactions at the particle surface leads to elastic distortions of the LC orientation. So, the extension of the present study to include the doping of silver nanoparticles (SNP) into the LC matrix is also made to understand how the orientations of LC molecules inside the bulk are affected.

The LC compound *N*(4-*n*-heptyloxy benzylidene)4′-*n*-butylaniline (also known as 7O.4) has the following phase sequence: Cr - (32.2°C) - SmG - (62°C) - SmC(64°C) - SmA - (72°C) - N - (74°C) - Iso. The static dielectric behavior following the change in temperature is studied minutely on the homogenously aligned LC molecules by giving two different treatments separately. In this study, the homogenous alignment of LC molecules in cells treated with low concentration of Cetyl Trimethyl Ammonium Bromide (CTAB) (which otherwise provide homeotropic alignment of LC molecules) is investigated by comparing it with the result of the same samples filled in rubbed Poly vinyl Alcohol (PVA)-treated cells. The analysis is based on the planar alignment mechanism [10,12] given in literature.

2. Experimental section

The compound was synthesized following a standard procedure given in literature [25]. SNPs capped with dodecanethiol procured from Sigma-Aldrich (Product No.667838, particle size 5–15 nm) was used to dope the compound in 0.25% (by weight). The mixture was sonicated for 1 hr to obtain a uniform dispersion and the hexane present in the suspension was allowed to evaporate completely. The quality of the doped sample was checked for the presence of any unwanted impurities like free thiols by taking the FTIR measurement (Model FTIR-8400S). The dispersed SNPs in the doped samples are characterized using TEM (Model JEOL JEM 2100). The phase transition temperatures and the LC phases exhibited by 7O.4 were confirmed by differential scanning calorimetry (DSC) and polarising thermal microscopy (PTM). Two kinds of cells with ITO coated glass plates – one treated with PVA (0.5% by weight) followed

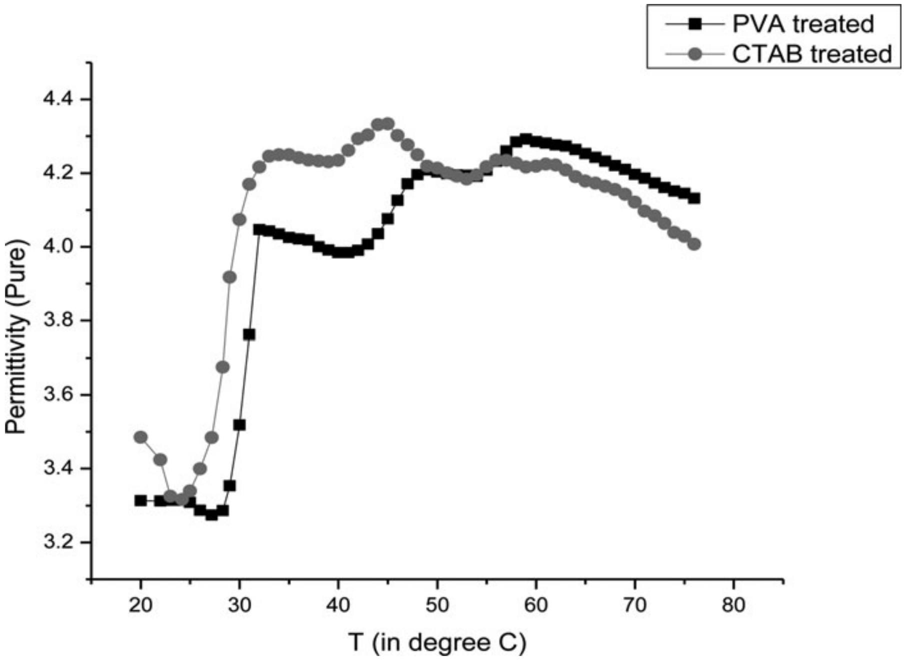

Figure 1. Temperature (*T*) dependence of static permittivity (ε') for the pure 7O.4 filled in the two types of cells (PVA treated and CTAB treated).

by rubbing and other with low concentration of CTAB (0.254 μM L) [26] – were made for homogenous alignment. Mylar spacers of 25 μm thickness were used to maintain the cell gap. The cells were calibrated using air and standard liquid like benzene for absolute values of permittivity. The samples were filled in the calibrated cells at temperature slightly above isotropic temperature. All the measurements were made with LCR meter (Agilent 4284 A) in the frequency range of 1 kHz–1 MHz.

3. Results and discussion

3.1. *Liquid crystal alignment and static permittivity behavior*

The variations of static permittivity (ε') with the variations of temperature for the pure 7O.4 sample filled in the two different types of cells - PVA treated cell (to be called P cell) and CTAB-treated cell (to be called C cell) – are shown in Figure 1. It is found that the static permittivity increases with the increase in temperature for the pure sample filled in P cell. The result indicates the homogenous alignment of the LC molecules as the compound is positive dielectric anisotropic material. This result tallies with the claims made by other workers [27, 28] that rubbing a substrate or polymer films produce unidirectional homogenous alignment though they had used different methods. The permittivity versus temperature curve for the pure sample filled in C cell is also found to increase with temperature and have a similar general form as that of the pure sample filled in P cell. This indicates the homogenous alignment of the LC molecules in cells treated with low concentration of CTAB and agrees with the report given elsewhere [26]. It may be recalled that dielectric technique of measuring the capacitance was used to check the LC alignment [18].

The similar general form of the two curves in Figure 1 reflects the dipole behavior and hence, the measured permittivity of the bulk LC samples. This may be seen from the following reasoning. We consider a LC cell as three capacitors in series – two due to the alignment layers (AL) and one due to the LC layer [29], so that the effective permittivity, $\varepsilon'_{\text{eff}}$ is

$$\varepsilon'_{\text{eff}} = \frac{\varepsilon'_{\text{AL}} \times \varepsilon'_{\text{LC}}}{\varepsilon'_{\text{AL}} \times \frac{\text{d}}{\text{d}_{\text{total}}} + 2\varepsilon'_{\text{LC}} X \frac{\text{d}'}{\text{d}_{\text{total}}}}, \tag{1}$$

where ε'_{LC} and ε'_{AL} are permittivities of the LC and alignment layers; d, d' and d_{total} are the thickness of LC layer, alignment layer and total thickness of the cell. If $d \sim d_{\text{total}}$ and $d' << d_{\text{total}}$, then $\varepsilon'_{\text{eff}} = \varepsilon'_{\text{LC}}$. In our experiment, $d \sim 20$ nm (using DEKTAK XT) while $\text{d}_{\text{total}} = 25$ micron, so our measured permittivity represents the contribution due to the bulk LC sample.

The effective voltage across LC layer is the same as applied voltage [30] for both types of cells as long as $d >> d'$. Hence a comparison of the measured permittivity values for the different types of cells may be made and their difference attributed to the effect of the alignment layer used.

A close look in the two curves of Figure 1 reveals the variation of permittivity with temperature resembling one another for temperature above 55°C. However, the permittivity corresponding to P cell is higher in values than the other. This may be due to a difference in orientation of LC molecules near the alignment layers (the LC molecules near the PVA surface bending more towards the normal in comparison to the LC alignment near the CTAB surface). The present explanation is based on the report [31,32] that rubbing process induces polar functional groups and repeating units to reorient out of the plane of the alignment film and non polar aliphatic side chain to partially orient towards the bulk of the alignment film. It was also demonstrated that the pretilt angle of LC molecules increases along with rubbing strength [30,31]. So, these factors must have accounted for the difference in permittivity values for the PVA-treated cells and CTAB-treated cells. However, for temperature below 55°C, we find an anomalous variation of permittivity with temperature. This may be due to an imperfect alignment in SmG phase or some processes occurring in this phase and will be addressed in later section.

The variation of permittivity as a function of temperature for the pure and the doped samples filled in the P and the C cells are shown in Figures 2 and 3. If the SNPs had segregated near the alignment layers, then one expects an absorption process at low frequency region as dielectric phenomenon near the electrode usually occurs at low frequency region [33]. However, such phenomenon was not observed in our case. This implies that segregation of nanoparticles near the alignment layers do not takes place in our prepared cells and the SNPs must have distributed in the bulk. In a similar work, it was reported [34] that the origin of SHF (subhertz frequency) process has not been changed when SNP was doped in the sample. It is found that the nature of variation of permittivity with temperature for the pure and doped samples in both type of cells – P and C – is similar except for the SmG phase in low temperature region. The difference in permittivity value between the pure and doped samples differs anomalously in the SmG phase (This aspect will be addressed in later section). At higher temperature above 55°C, the curve for doped sample is similar in behavior to that of the pure sample, thereby indicating the same LC phases as that of the pure sample. However, the value of the permittivity in doped sample is comparatively lower than that of the pure sample. And this could be due to an effective reduction in average dipole moment per unit volume. The mechanism of the reduction in permittivity could arise from the rearrangement of a few LC molecules

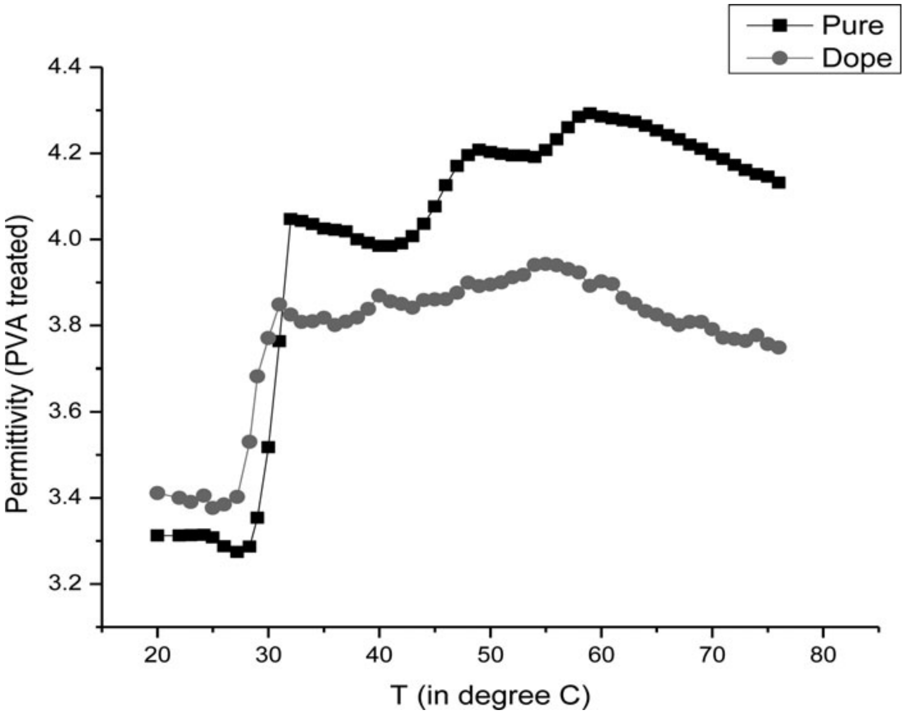

Figure 2. Permittivity versus temperature curves for pure and doped 7O.4 samples filled in PVA-treated cell.

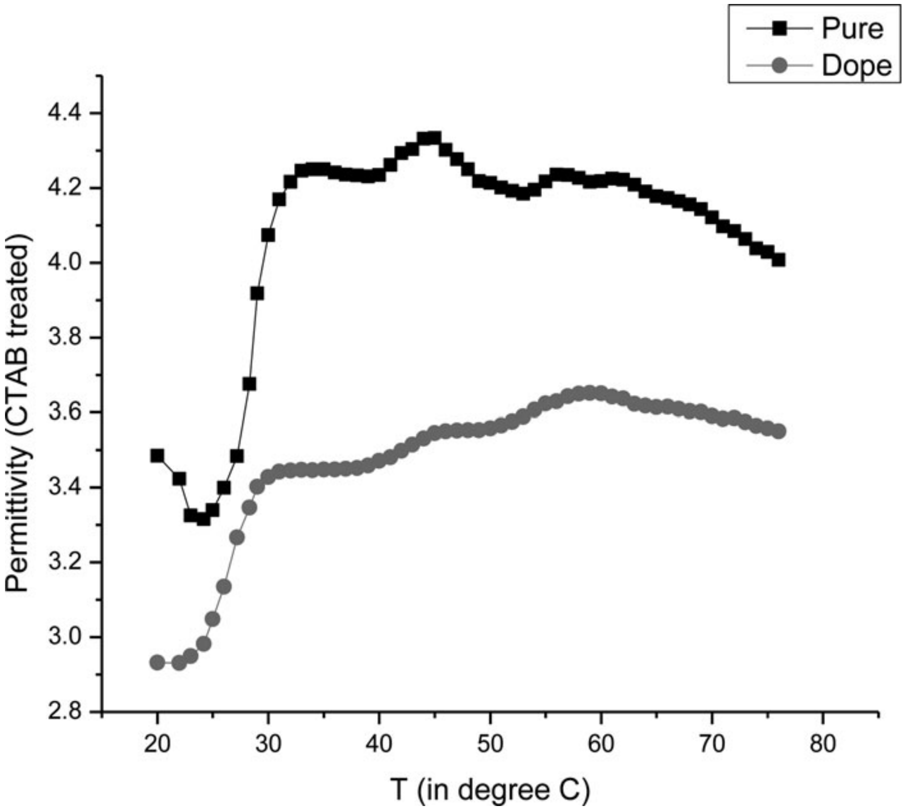

Figure 3. Permittivity versus temperature curves for pure and doped 7O.4 samples filled in CTAB-treated cell.

Table 1. Phase transition temperature (in°C) of 7O.4 (pure and doped) from dielectric studies.

Phase transition	SmG-SmC	SmC-SmA	SmA-N	N-Iso
Sample				
Pure 7O.4 (PVA treated)	62	64	72	74
Doped 7O.4 (PVA treated)	61.5	64.5	71	73.5
Pure 7O.4 (CTAB treated)	62.5	64	72	74
Doped 7O.4 (CTAB treated)	62	64	72	73.5

or dipoles surrounding the SNP over a small region without affecting the distribution of far away LC molecules in the bulk.

The phase transition temperatures for the pure and doped samples obtained from dielectric studies are given in Table 1. The transition temperature values obtained in dielectric studies are very close to DSC and PTM studies. It is found that the phase transition temperatures are slightly affected by the presence of SNPs. Similar report on the effect of doping SNPs in other LC compound was reported by Barmatov et al [35] that the phase behavior (type and temperature interval of mesophase) of polymer nanocomposites depends on the content of SNPs and the glass transition temperature increased with the increase in the concentration of metal particles. The behavior was explained by chemical adsorption of cyanobiphenyl and carboxylate groups of the polymer onto the surface of the SNPs.

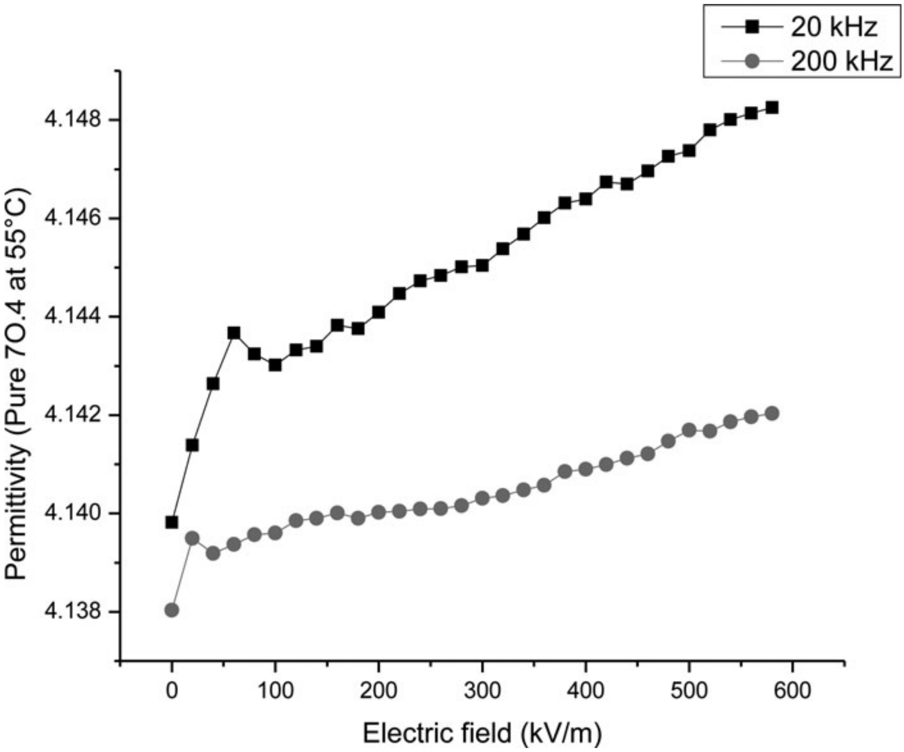

Figure 4. Permittivity (ε') variation with biasing dc electric field at a representative frequency of 20 and 200 kHz for pure 7O.4 sample filled in PVA-treated cell at 55°C.

3.2. Alignment and reorientation under an electric field

As a part of the systematic dielectric study on the planarly aligned LC molecules using low concentration of CTAB, the electric field dependence of permittivity for samples filled in CTAB-treated cell is also investigated in the SmG phase.

In Figures 4 and 5, the variations of permittivity with the biasing dc electric field (at constant frequency 20 and 200 kHz) at 55°C are shown for the pure and the doped samples. At constant frequency 20 kHz, permittivity increases up to a maximum value at electric field 60 kV m and then decreases to a minimum value at an electric field of 100 kV m, and finally increases linearly with increasing electric field. The behavior for permittivity versus field for the doped samples is also similar but the maximum and the minimum occur around at 40 and 100 kV m, respectively. This could mean a reduction in threshold voltage. A similar report on the reduction of threshold voltage was reported [36] elsewhere when the LC was doped with SNP. At higher frequency of 200 kHz, the maximum and the minimum are less pronounced. The mechanism may be explained by considering motion of ions and conductivity anisotropy. The dipole is along the molecular axis and the LC molecules are initially aligned along the surface due to the treatment given. As the biasing dc electric field increases, a few number of molecules start orienting along the field direction; it means the homeotropic contribution increases with increasing electric field. Consequently, permittivity increases till a maximum value with the electric field. After reaching the maximum value, the decrease in permittivity with the increase of field may be related to the effect of the ions in motion. As the conductivity anisotropy is usually negative in smectics [37], the torque due to the motion of ions opposes the electric torque and start disturbing the molecular alignment from the field

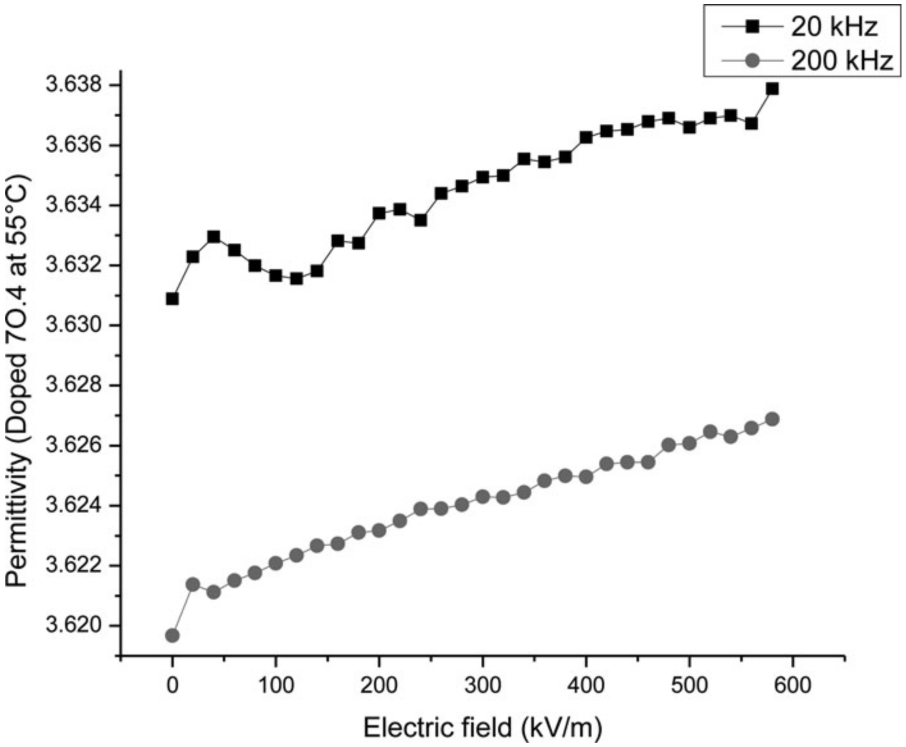

Figure 5. Permittivity (ε') variation with biasing electric field at a representative frequency of 20 and 200 kHz for doped 7O.4 sample filled in CTAB-treated cell at 55°C.

direction. This may result in the decrease of the permittivity. However, the increase in permittivity after attaining the minimum might indicate the onset of electric torque greater than that due to ionic motion. When the samples are doped with SNP, the modification in local rearrangement of LC molecules surrounding the nanoparticle could possibly affect the dipole correlation of the LC molecules [38] resulting in the early onset of ionic motion. However, the behavior for the pure and the doped samples at higher frequency 200 kHz are almost similar. This may be attributed to the lesser importance of ions at higher frequency. The present explanation may be analogous to nematic LC behavior like that of domain mode [39] at low electric field and scattering mode [40] at high field region.

3.3. *Dielectric absorption in SmG*

Figures 6 and 7 show the three-dimensional plot of dielectric loss factor with the variation of temperature and frequency for the sample filled in PVA-treated and CTAB-treated cells in planar alignment. It may be observed that there occurs an absorption process in the SmG phase with the variation of temperature by keeping the frequency fixed. This behavior was also observed in the SNP doped 7O.4 sample (Figures not shown). A similar report on the existence of this absorption process was reported in same homologues of 8O.m [41] compounds, which has not been analyzed in detail. A close observation on the said absorption reveals that the process is a kind of relaxation absorption process but existing at all the low frequency region studied. So it may be treated as a response to the variation of temperature by keeping the

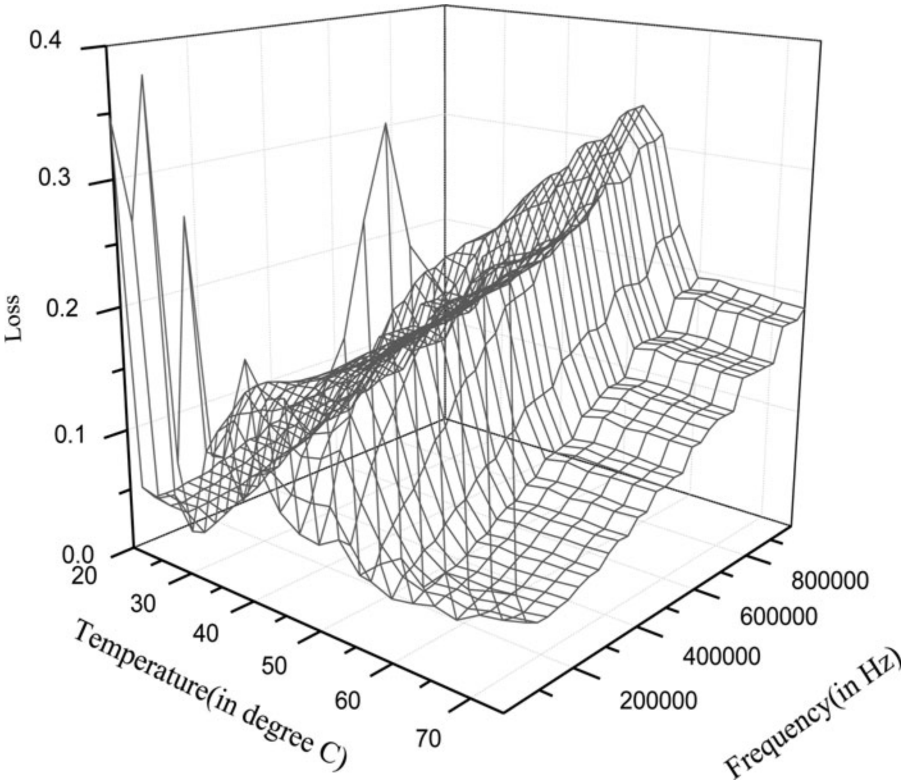

Figure 6. Three-dimensional plot of temperature and frequency dependence of dielectric loss of pure 7O.4 sample filled in PVA-treated cell.

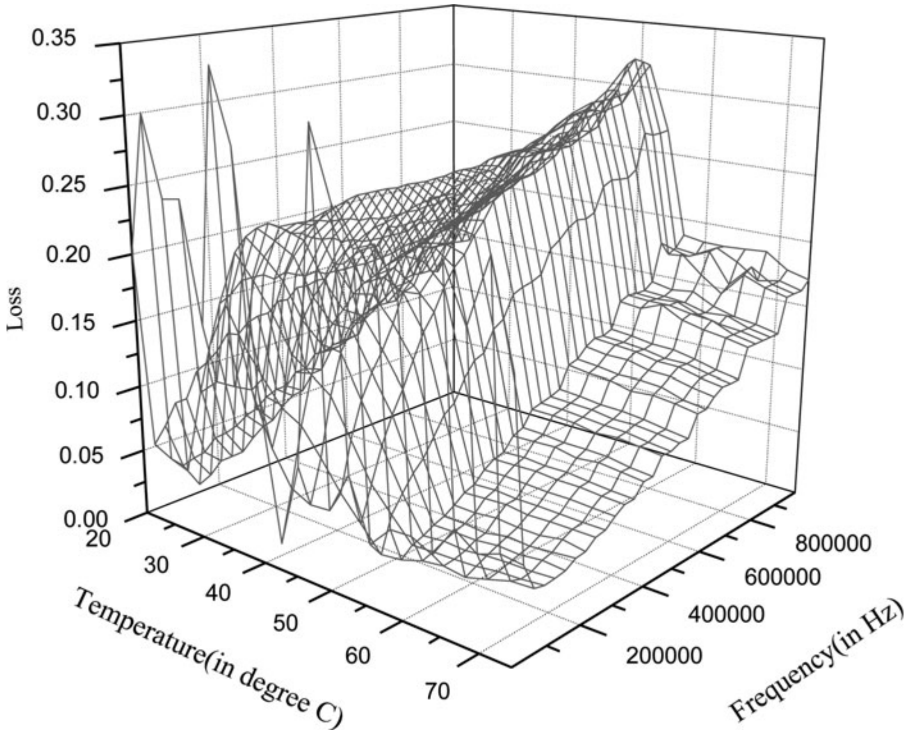

Figure 7. Three-dimensional plot of temperature and frequency dependence of dielectric loss of pure 7O.4 sample filled in CTAB-treated cell.

frequency fixed. Our analysis follows a method proposed by Chelskowki [42]. The relaxation time corresponding to this type of absorption process may be considered as a function of the temperature, that is τ s $\tau(T)$. If a Debye type for the observed absorption process is assumed, then

$$\partial\left(\frac{\varepsilon''}{\frac{\varepsilon'(s)-\varepsilon'(\infty)}{\partial x}=0}\right) \tag{2}$$

Solving this equation, we get

$$\omega\tau(T_{max})=1, \tag{3}$$

where $\tau(T_{\max})$ represent the relaxation time at the temperature ($T_{\max}$) corresponding to the location of the absorption peak. This $T_{\max}$ would be addressed here as peak relaxation temperature and the values of $T_{\max}$ found for each value of the corresponding relaxation frequency are plotted in Figure 8, as a function of relaxation frequency for the pure and the doped 7O.4 samples in planar alignment. It may be observed that the relaxation frequency of the doped 7O.4 sample has shifted towards the lower temperature side. The appearance of such type of absorption behavior both in the homeotropic alignment [43] of 7O.4 and 7O.6 and in the homogenous alignment (present work) seem to indicate that the process is linked to the molecular interaction of SmG phase of the two samples studied and the relaxation time could correspond to a parallel or perpendicular component [44]. This could be the reason for the observed anomalous behavior in dielectric permittivity in SmG phase.

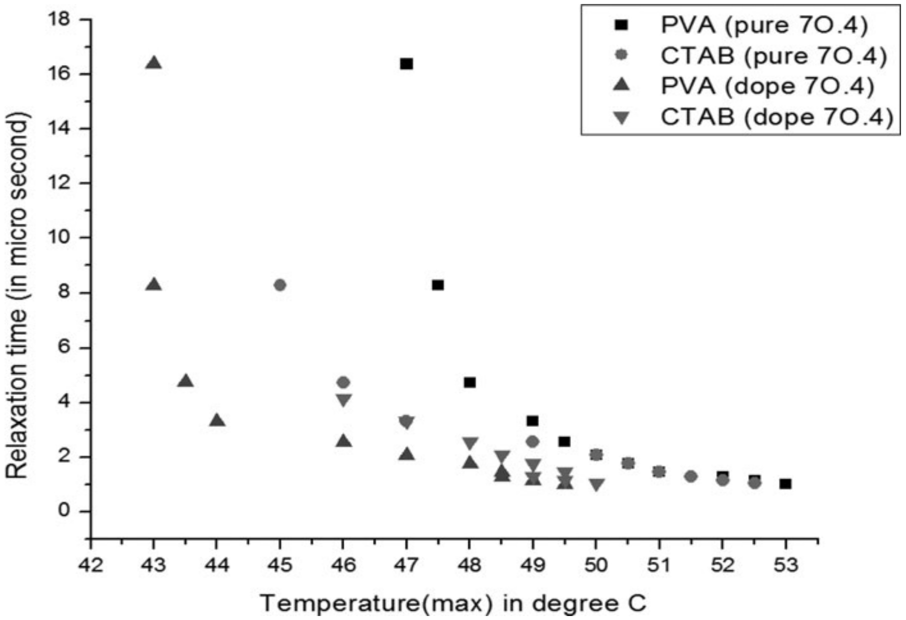

Figure 8. Relaxation time versus temperature (where absorption peak is maximum for fixed frequency) in SmG phase.

4. Conclusions

In summary, it can be seen that the alignment of the LC molecules could be studied through dielectric technique on the assumption of a known fixed orientation of the dipole relative to the LC molecule. The study reveals that the use of different alignment layers reflects on a difference in values of static dielectric permittivity, which could be attributed to a possible difference in relative orientation of the LC molecules very near to the alignment layers. However, the similarity in general shape of the permittivity versus temperature curves does indicate the achievement of homogenous alignment of the LC molecules in the bulk. In the case of SNPs doped sample, the local rearrangements of the LC molecules around the nanoparticles took place, but preserve the general LC orientation in the bulk and thus lead to a similar shape in the permittivity versus temperature curve. The dielectric properties of the dipolar LC are related more to the molecular arrangement and any modifications of it may be achieved by a local molecular rearrangement near the boundary (as in the case of using alignment layers) or in the interior (as in the case of doping SNPs). The same assumption on the known fixed relative orientation of the dipole with respect to the LC molecule was also used in the explanation for reorientation of the molecules under an applied electric field leading to the observed dc electric field dependence of permittivity. We believe that the existence of an absorption process in SmG phase at the entire low frequency region studied is the cause for anomalous behavior in this phase.

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