



## Dielectric Relaxation Studies of Silver Nanoparticles dispersed Liquid Crystal

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### ABSTRACT

This article reports on the Dielectric Relaxation Studies of two Liquid Crystalline compounds - 7O.4 and 7O.6 - doped with dodecanethiol capped Silver Nanoparticles. The liquid crystal molecules are aligned homeotropically using CTAB. The low frequency relaxation process occurring above 1 MHz is fitted to Cole-Cole formula using the software Dielectric Spectra fit. The effect of the Silver Nanoparticles on the molecular dipole dynamics are discussed in terms of the fitted relaxation times, Cole-Cole distribution parameter and activation energy. The study indicate a local molecular rearrangement of the liquid crystal molecules without affecting the order of the bulk liquid crystal molecules but these local molecules surrounding the Silver Nanoparticles do not contribute to the relaxation process in the studied frequency range. The observed effect on activation energy suggests a change in interaction between the nanoparticles/liquid crystal molecules.

### Indexing terms/Keywords

liquid crystal, dielectric permittivity, dielectric loss factor, relaxation time, Cole-Cole distribution parameter, activation energy

### Academic Discipline And Sub-Disciplines

Physics; Liquid Crystals

### SUBJECT CLASSIFICATION

Physics

### TYPE (METHOD/APPROACH)

Experimental

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## INTRODUCTION

Dielectric Relaxation Studies of liquid crystals (LCs) provide important information on molecular arrangement and molecular reorientations. This technique enables one to investigate some aspects of molecular dynamics in LC materials with permanent molecular dipoles by using suitably aligned samples. As it is well-known that dielectric spectra show absorption domains with an origin linked to the rotational motions of the molecule around its long molecular axis or its transversal short axis [1-3], information on the relaxation of the dipoles associated with the LC molecules and the rotational motion of the LC molecules in the anisotropic phases may be deduced.

Most of the popular models / theories on dielectric relaxation have successfully been applied to LCs. The Martin et al. theory [1] has been applied to nematogens (such as alkyl-cyanobiphenyls) whose resultant dipole moments lie parallel to the long molecular axis. However, it is not applicable to LC with a significant dipole component perpendicular to the long molecular axis (eg. p-methoxy-benzylidene-p-n-butylaniline). Later, Nordio et al. [4] developed a theory (NRS) which can be applied to molecules with resultant dipole lying at an angle to the long molecular axis. There are reports [5-7] supporting the inference that the presence of the low frequency relaxation region for the LC molecules in parallel alignment is connected with hindering of molecular rotation around the short axis in the presence of the nematic potential. However the origin of the higher frequency relaxation is difficult to establish. Buka and Leyvraz [8] offer an explanation for this high frequency process by a combination of the rotational diffusion and flip-flop model. A transverse dipole moment (different from zero) can be connected with the flexibility of the alkyl chain and might influence the high frequency relaxation process. Generally the high frequency relaxation process is considered due to molecular rotation around the long axis. The absorption spectra are affected by certain factors. Multiple relaxations and local intermolecular perturbations may also occur. Flexibility of the molecules can lead to broadening of the relaxation spectra or appearance of additional absorption spectra at different frequencies. Such additional relaxations can sometimes contribute to the dielectric spectra of the isotropic phase [9]. In the case of more ordered liquid crystal phases such as smectic and columnar phases, dielectric absorption due to collective modes has been detected. Such modes are regarded as characteristics of the phase structure rather than the structural features of the constituent molecules [10].

As knowledge of the dielectric properties of liquid crystals is important for fundamental and technical reasons, a number of research activities on LC like - mixing different types of LC[11], adding dyes to LC[12] or doping non-LC material to the LC material[13] and so on - were reported.

In recent years, there is a growing surge in research activities on the doping of nanoparticles in LC material. This is so because the integration of nanoparticles in liquid crystal (LC) technology opens new perspectives for developing new devices. Our present dielectric relaxation study is a continuation of our systematic studies on the effect of doping silver nanoparticles (SNPs) on few alkyloxy benzylidene alkyylaniline (or nO.m) compounds. The integration of the SNPs in the chosen nO.m compounds is a good candidate for studying some fundamental aspects of the dielectric relaxation theory which has been used in these LC system because the compounds are very rich in showing smectic polymorphism while the nanoparticles impart additional or modify the properties of the bulk LC materials yet retaining the mesomorphic character of the LCs[14-16]. It is to be noted that the LCs act as a solvent for the suspending nanoparticles providing their long-range and self-alignment [17-20]. The addition of nanoparticles in LCs is likely to affect the local molecular arrangement and the environment. This idea is an extension of the report given in literature [21] that the dielectric response seem to be susceptible to molecular shape, partly because changes of shape are often accompanied by changes of molecular dipole, and also because dielectric properties probe the environment of relaxing dipoles, which is shape-dependent as well.

In the present paper, the measured dielectric data of the nO.m compounds for the low frequency relaxation process (order few MHz) corresponding to homeotropically aligned LC molecules are best fitted to the empirical Cole-Cole model. The fitting of the said Cole-Cole type of relaxation process to some members of nO.m compounds have also been reported elsewhere [22]. The software DIELECTRIC SPECTRA FIT is used. In the process of analysis, it is found that the fitted data correspond well to the said type of relaxation process for both the pure and the SNPs doped LC compounds. The SNPs increase the distribution parameter of the relaxation process.

## MATERIALS AND METHODS

The liquid crystalline compounds 7O.4 and 7O.6 were prepared following a standard procedure given in the literature [23]. The two compounds were separately doped with SNPs (purchased from Sigma-Aldrich (Product No. 667838, particle size 5 - 15 nm) in 0.25% (by weight). The mixture was sonicated for one hour to obtain a uniform dispersion and the hexane present in the mixture sample was allowed to evaporate completely. TEM images of the nanoparticles are shown in Figure1. Cells for homeotropic alignment were prepared by using highly conducting ITO coated (which were treated with CTAB solution (6.8 mM/L)) optically flat glass substrates as electrodes. Mylar spacer of 25 micron was used for maintaining cell gap. Next, the empty cells were calibrated using air and benzene. The calibrated empty cells are filled with the samples at temperature slightly above the isotropic temperature. And finally, all the dielectric measurements were made with LCR meter (Agilent 4284 A) in the frequency range of 1kHz - 1MHz.

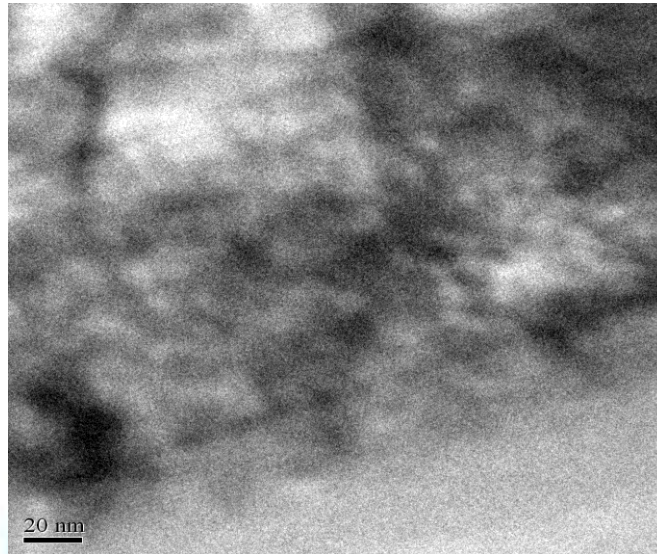


Figure 1: TEM images of silver nanoparticles

## RESULTS AND DISCUSSION

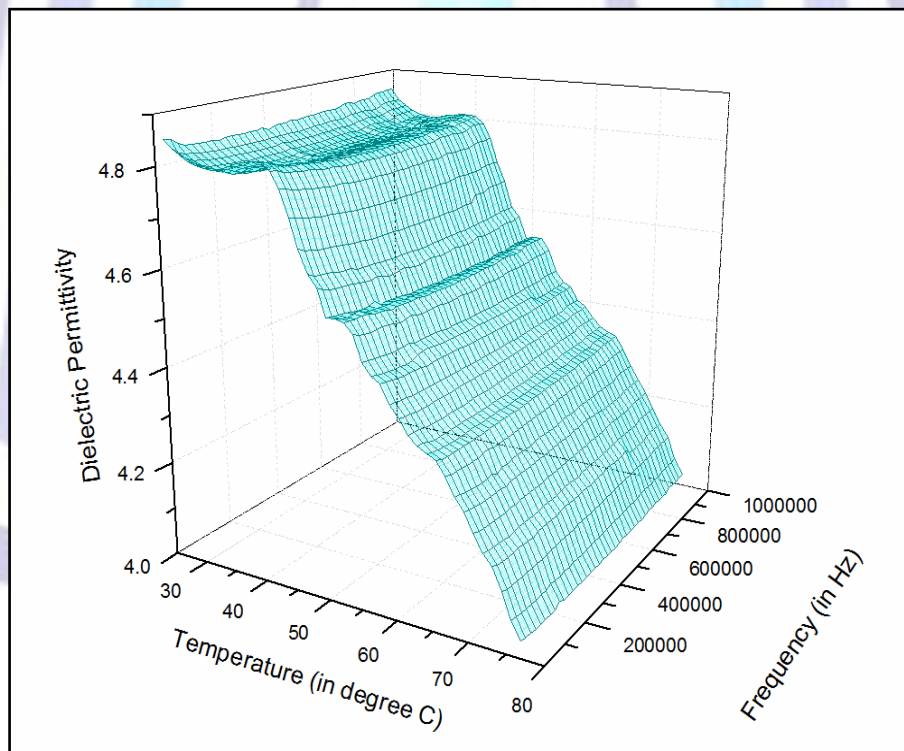
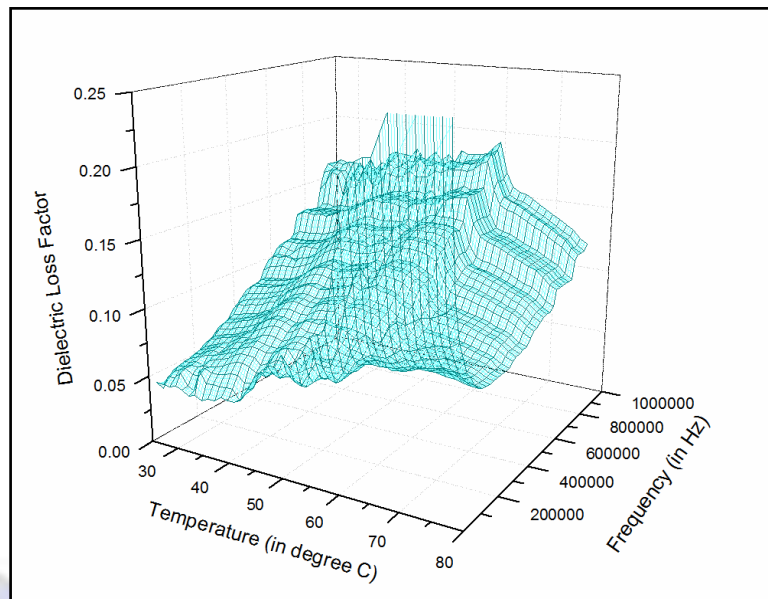


Figure 2a: 3 dimensional plot of dielectric permittivity with variation in temperature and frequency for pure 70.4 in homeotropic alignment

In above Figure 2a, the 3 dimensional plot of dielectric permittivity with the variation of temperature and frequency for the pure 70.4 in homeotropic alignment is shown. It is seen that the dielectric permittivity decrease with the increase of temperature showing the characteristic of a positive dielectric anisotropic material. In Figure 2b, the temperature and the frequency dependence of the dielectric loss factor for the pure 70.4 sample is given. It is observe that there is an absorption process with the variation of temperature at all the low frequency studied. This type of process was also reported earlier [22]. In the same Figure 2b, one may observe a trailing end of a relaxation process (at fixed temperature) occurring in the higher frequency region outside the experimental window. The nature of the variation of the dielectric permittivity and the dielectric loss factor with temperature and frequency for the pure 70.6 sample in homeotropic alignment are also found to be similar (not shown) to that for pure 70.4.

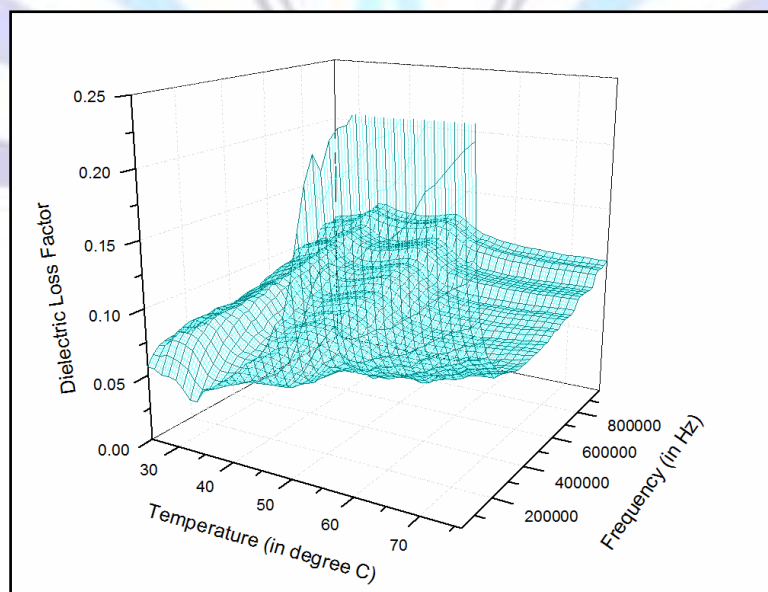


**Figure 2b: 3 dimensional plot of the dielectric loss factor with the variation in temperature and frequency for pure 70.4 in homeotropic alignment**

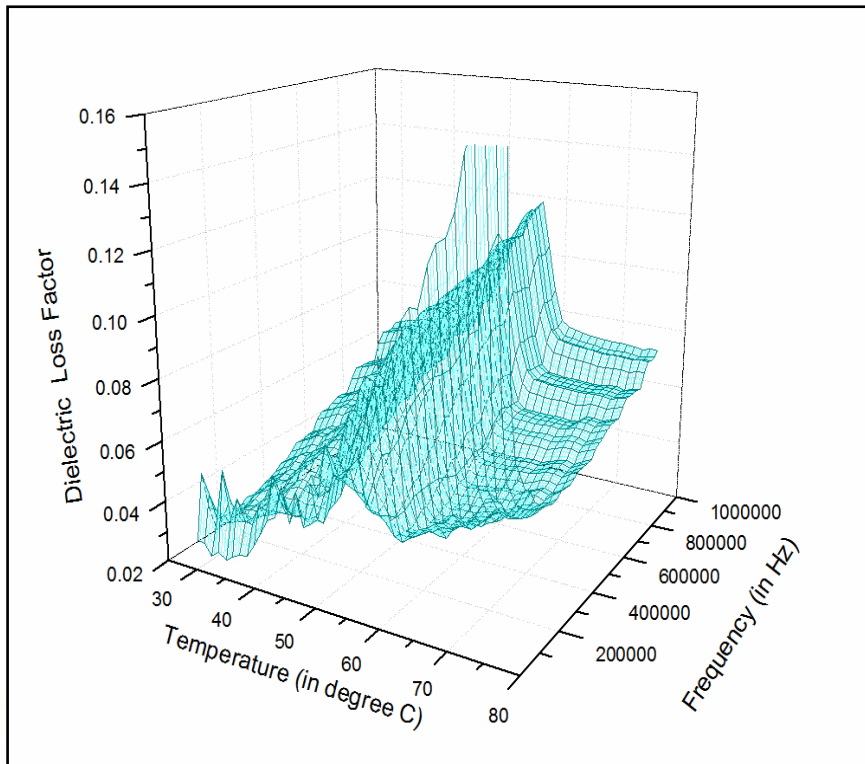
When the samples are doped with silver nanoparticles in small amount (about 0.25% by weight), the dielectric properties of the LC host are affected significantly. Both the dielectric permittivity and the dielectric loss factor values decrease in the doped samples. The 3d plot of dielectric loss factor as a function of temperature and frequency for the doped 70.4 and the doped 70.6 are shown in Figures 3a and 3b. The trailing end of the relaxation process occurring in the higher frequency region (> 1 MHz) can also be seen in the doped samples. A comparative analysis of this relaxation process is made for the pure and the doped samples by fitting the measured dielectric quantities with the modified Cole - Cole equation:

$$\epsilon^* = \epsilon_\infty + \frac{\epsilon_0 - \epsilon_\infty}{1 + (i\omega\tau)^{1-\alpha}} + Af^m \tag{1}$$

where  $\epsilon_\infty$  is the dielectric permittivity at very high frequency;  $\epsilon_0$  is the static dielectric permittivity;  $\omega$  is the angular frequency;  $f$  is the frequency;  $\tau$  is the relaxation time;  $\alpha$  is the Cole-Cole distribution parameter. The last term in the above equation takes into account of the effect of the non LC contribution[24, 25] at high frequency due to resonance effects [26]. The parameters  $A$  and  $m$  are to be fitted.

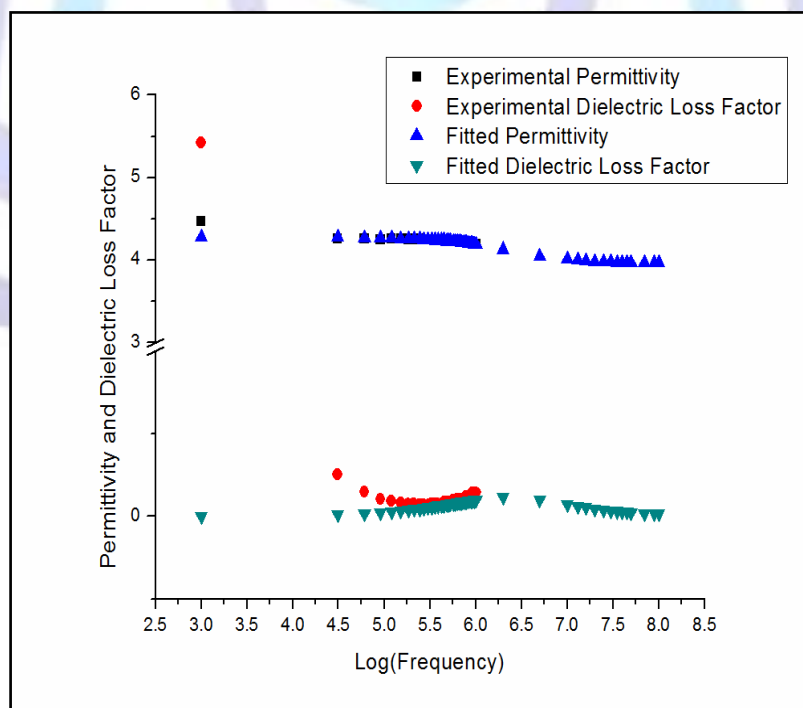


**Figure 3a: 3 dimensional plot of dielectric loss factor with variation in temperature and frequency for doped 70.4 in homeotropic alignment**

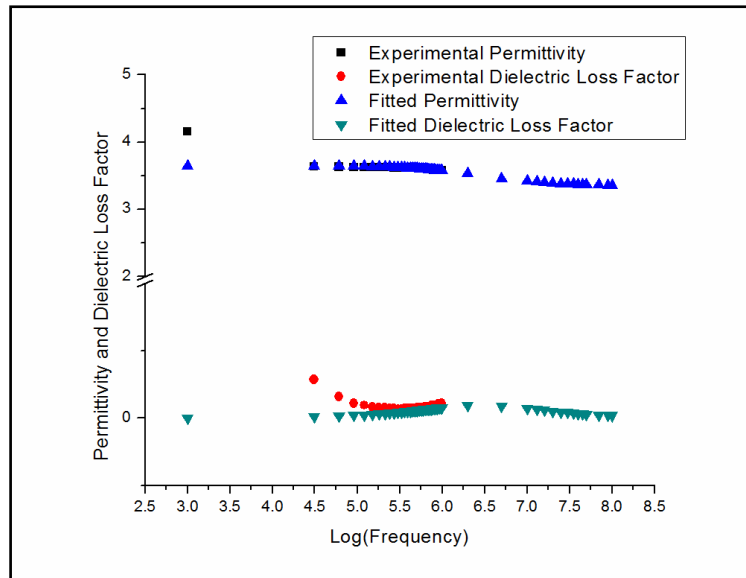


**Figure 3b: 3 dimensional plot of dielectric loss factor with variation in temperature and frequency for doped 7O.6 in homeotropic alignment**

We have used the software DIELECTRIC SPECTRA FIT for fitting the measured data. It is found that the non LC contribution exist at frequency above 700 kHz. Typical values of fitted parameters  $A$  and  $m$  for the pure 7O.4 at 67°C gives  $2.43 \times 10^{-5}$  and 0.29999785. Figure 4a shows the variation of the dielectric permittivity and the dielectric loss factor for the pure 7O.4 sample as a function of frequency at a representative temperature of 67°C. In the Figure, both the experimentally measured and the fitted dielectric data are indicated.



**Figure 4a: Frequency dependence of dielectric permittivity (experimental and fitted) and loss factor (experimental and fitted) for pure 7O.4 in homeotropic alignment for representative temperature at 67°C**



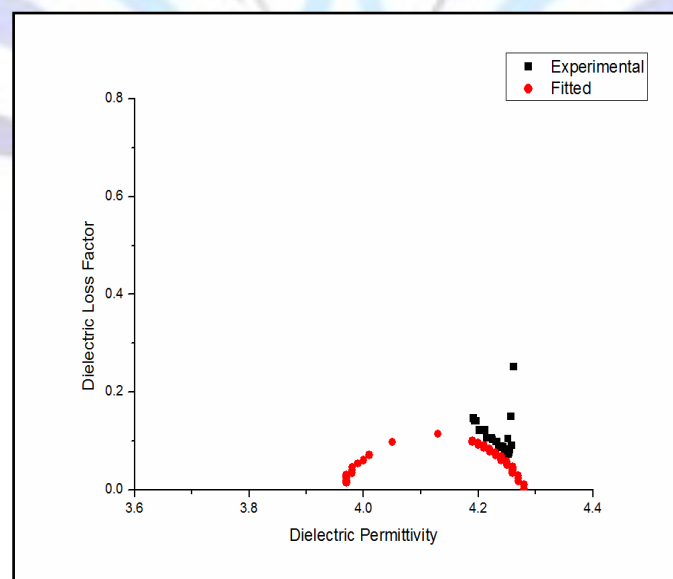
**Figure 4b: Frequency dependence of dielectric permittivity (experimental and fitted) and loss factor (experimental and fitted) for doped 70.4 in homeotropic alignment for representative temperature at 67°C**

The fitted data shows the contribution only due to the relaxation process of the LC sample in homeotropic alignment (after separation of non LC contribution in high frequency region and ionic conductivity in lower frequency region). Representative plots of the measured dielectric data and the fitted data for the doped 70.4 is shown in Figure 4b.

The fitted parameters *A* and *m* for all the pure and the doped samples at representative temperature are given in Table 1.

**Table 1: Fitted parameters *A* and *m* for the samples at few representative temperature (as indicated)**

Samples	<i>A</i>	<i>m</i>
Pure 70.4 at 67°C	$2.43 \times 10^{-5}$	0.29999785
Doped 70.4 at 67°C	$2.51 \times 10^{-5}$	0.300001021
Pure 70.6 at 68°C	$3.01 \times 10^{-5}$	0.301237555
Doped 70.6 at 68°C	$3.22 \times 10^{-5}$	0.311002464



**Figure 5a: Cole-Cole plot of the experimental and the fitted dielectric permittivity and loss factor for pure 70.4 at 67°C**

Figure 5a and 5b show the Cole-Cole plot of the measured dielectric data and the fitted dielectric data for the pure 7O.4 and the pure 7O.6 samples. It is seen that the fitted dielectric data lie on a semicircle with the centre lying slightly below the axis. This indicates that there is a distribution of the relaxation times for the said type of relaxation process. In the two Figures (5a and 5b) shown for the representative temperatures 67°C and 68°C, the values of  $\alpha$  for pure 7O.4 and pure 7O.6 are 0.21 and 0.18 respectively. It means the mechanism of the relaxation process is not as simple as that of a Debye type. It was reported in literature [27] that dipoles of large dipole moment give rise to large absorption peak. So, the small value of the absorption peak, which is less than 0.20 in both the two pure samples, could be due to the moderate strength of the dipoles associated with the LC molecules. A comparative Cole-Cole plot (using the fitted dielectric data) for the relaxation process corresponding to the pure and the doped sample of 7O.4 are shown in Figure 6a while that of the pure and the doped 7O.6 sample are shown in Figure 6b. The dielectric absorption peaks of the doped samples (7O.4 doped with SNPs and 7O.6 doped with SNPs) are found to be slightly decreased when compared to that of the pure LCs. This

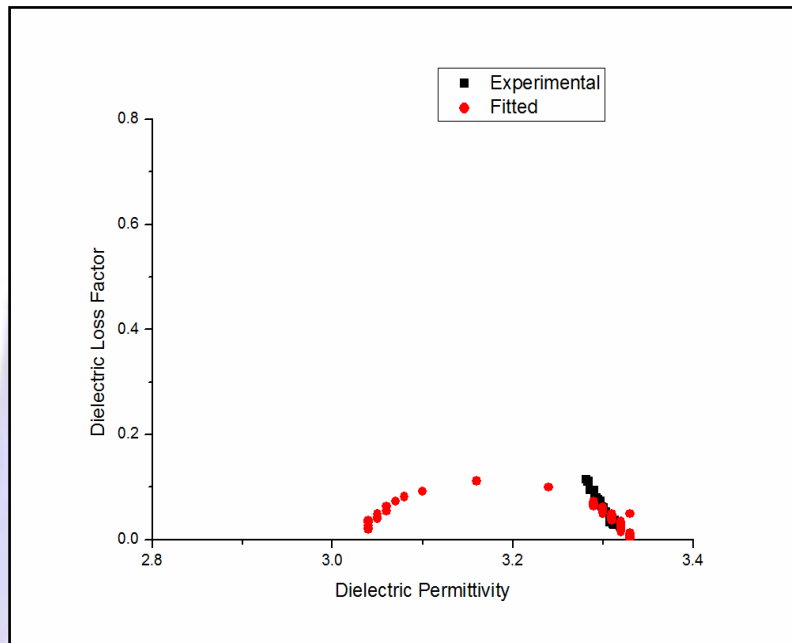


Figure 5b: Cole-Cole plot of the experimental and the fitted dielectric permittivity and loss factor for pure 7O.6 at 68°C

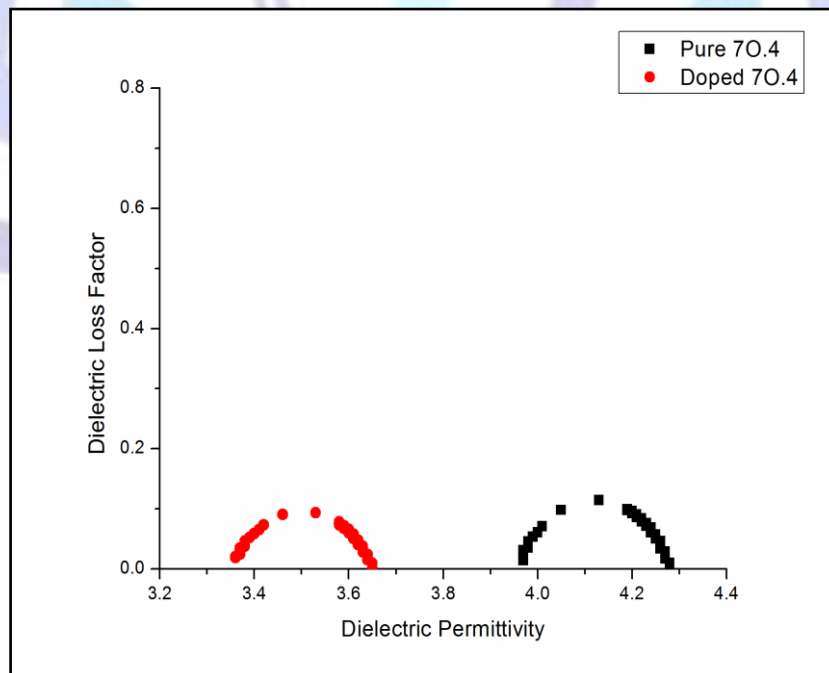
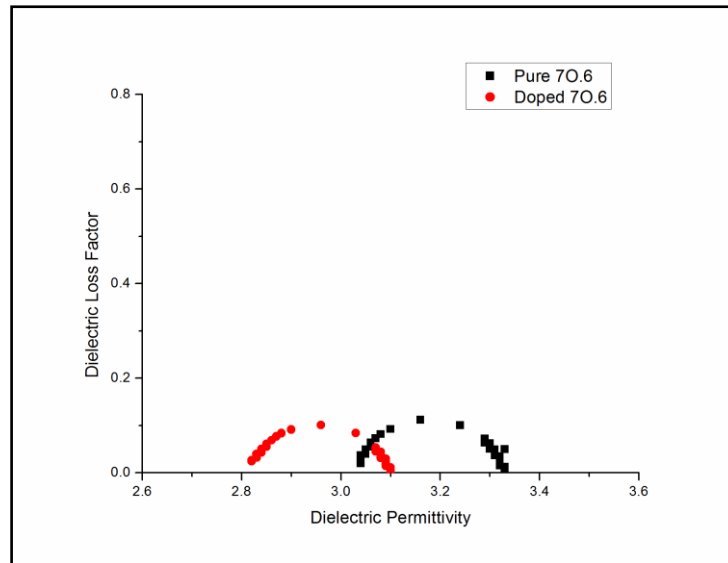


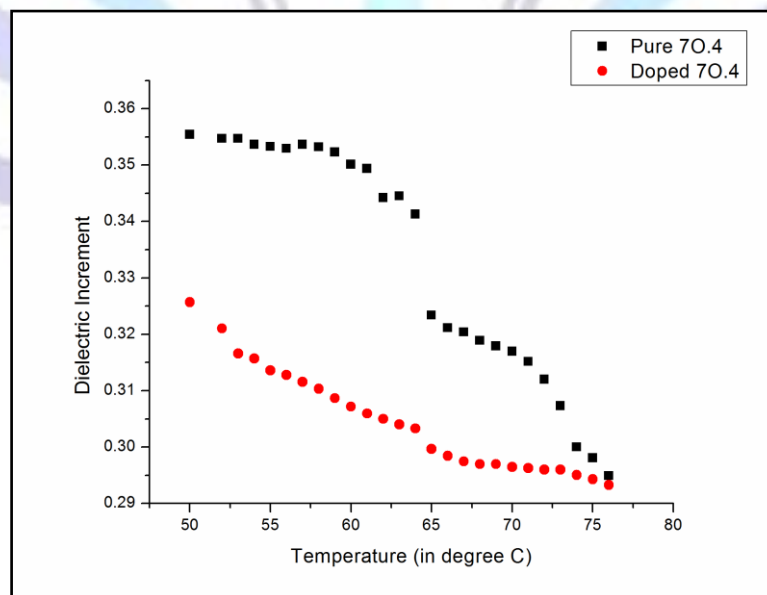
Figure 6a: Cole-Cole plot of the fitted dielectric permittivity and loss factor for pure and doped 7O.4 at 67°C



**Figure 6b: Cole-Cole plot of the fitted dielectric permittivity and loss factor for pure and doped 70.6 at 68°C**

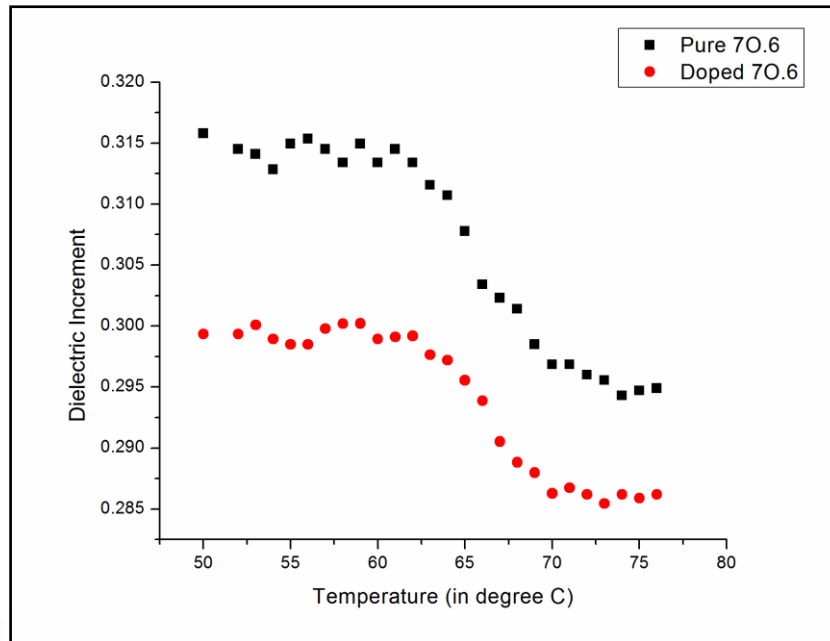
may be seen in the two Figures 6a and 6b that the fitted data of the Dielectric loss peaks are slightly lowered in comparison to that of the pure LC samples. Similar report on the decrease in dielectric loss when titania nanoparticles are doped in the ferroelectric LC may be found in literature [28]. It was also reported by Pankaj Kumar Tripathi et al.[29] that the doping of ZnO nanoparticles to a nematic LC lead to a decrease in the amplitude of loss factor.

Figures 7 and 8 represent the temperature dependence of the dielectric increment, given by  $\Delta\epsilon = \epsilon_0 - \epsilon_\infty$  for the samples - 70.4 (both pure and doped) and 70.6 (both pure and doped). It is seen that the values of the dielectric increment for the doped samples are smaller than the corresponding pure samples. This could mean a reduction in the absorption intensity as it is given in literature that the absorption intensity is measured by the dielectric increment,  $\Delta\epsilon = \epsilon_0 - \epsilon_\infty$  [5,30]. Since the intensity of the low frequency absorption in the parallel alignment is dependent on  $\mu_l^2(1+2S)$  [30], (where  $\mu_l$  is the longitudinal dipole moment component parallel to the long molecular axis), we believed that the doping of SNPs lead to a possible reduction in the order parameter  $S$  and the value of  $\mu_l$ . Thus, the observed decreased for the doped samples may be explained on the reasoning that the nearby LC molecules surrounding the SNP have a net reduced dipole moment and are strongly bound to it that they do not contribute to the absorption process. It may, however, be noted that the relative decrease in dielectric increment is not the same at all the liquid crystalline phases. For the pure 70.4 and 70.6 samples, there is a gradual decrease in dielectric increment when the temperature is increased. This trend also exists in the doped 70.4 and 70.6 samples.



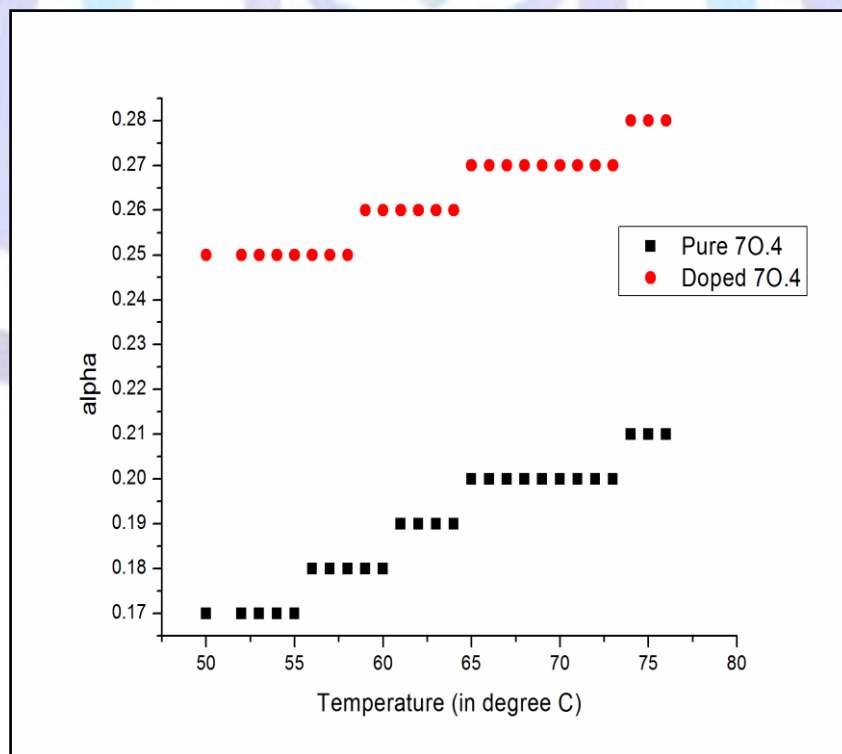
**Figure 7: Dielectric increment versus temperature for the pure and the doped 70.4 in homeotropic alignment**



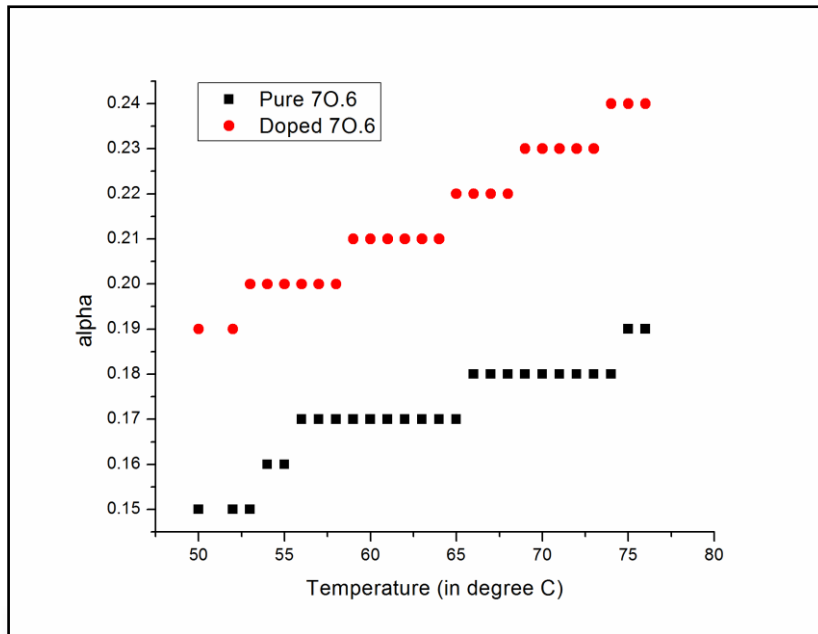


**Figure 8: Dielectric increment versus temperature for the pure and the doped 70.6 in homeotropic alignment**

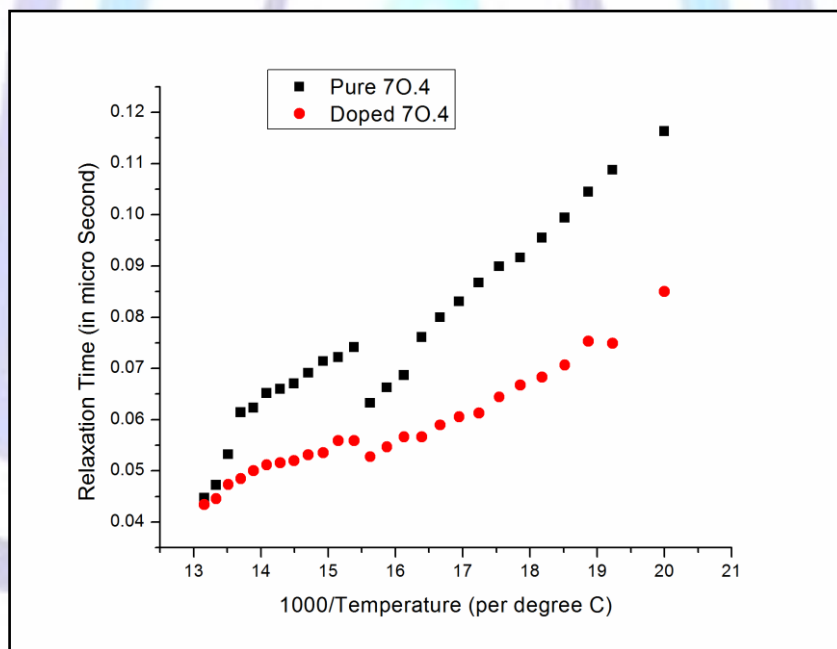
Figure 9 shows the temperature variation of the distribution parameter  $\alpha$  for the pure and the doped 70.4 samples. Likewise, Figure 10 shows the variation of  $\alpha$  as a function of temperature for the pure and the doped 70.6 sample. It is seen that the distribution parameter increase with the increase of temperature for both the pure and the doped samples. When SNPs are doped into the host LC, an increase in the value of the distribution parameter  $\alpha$  occurs. This could mean the complexity of the mechanism of the dielectric relaxation process. The SNPs might have change/ affect the local environment that a broadening of relaxation times occur and consequently in the increase of  $\alpha$ . The present reasoning is based on the explanation given in literature [21].



**Figure 9: Fitted distribution parameter ( $\alpha$ ) versus temperature for the pure and the doped 70.4 in homeotropic alignment**

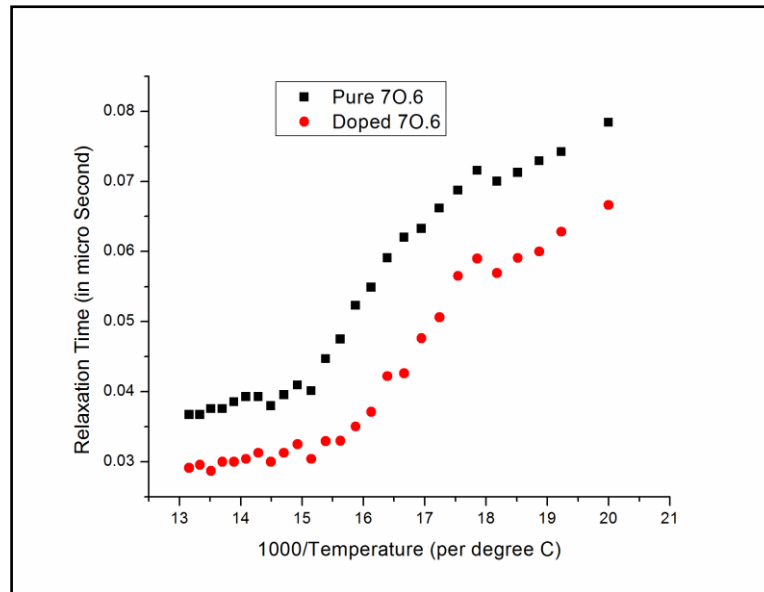


**Figure 10: Fitted distribution parameter (alpha) versus temperature for the pure and the doped 70.6 in homeotropic alignment**



**Figure 11: Relaxation time (in microsecond) versus inverse of temperature (1000/T) for the pure and the doped 70.4**

Figure 11 and 12 shows the plot of relaxation times versus the inverse of temperature for the samples -70.4 (both pure and doped) and 70.6 (both pure and doped). It is observed that all the studied samples show the general characteristic [31] where the relaxation frequency increase with temperature. It is found that the relaxation frequency for the doped samples shift to higher frequency (and hence lowering of the relaxation times as shown in Figure 11 and 12) when compared to that of the pure sample. Following the argument given in literature [32], our result suggest of a change in interaction between the LC molecules/ SNPs. In other words, it may be interpreted that there occur acceleration in dielectric relaxation of the SNP doped LC samples (Figure 11 and 12). As reported in literature [33] the acceleration of the short axis relaxation times in both the nematic and isotropic phase was caused by the disordering effect of the aerosil nanoparticles, we may, likewise, conclude that the SNPs produce some sort of disorder in LC samples - 70.4 and 70.6 possibly leading to a decrease in observed dielectric loss.



**Figure 12: Relaxation time (in microsecond) versus inverse of temperature (1000/T) for the pure and the doped 7O.6**

The temperature dependence of the relaxation time can be described by the Arrhenius formula:

$$\tau = \tau_o \exp(E_a / kT) \tag{2},$$

where  $\tau$  is the relaxation time;  $E_a$  is the activation energy;  $k$  is the Boltzmann constant and  $T$  is the temperature. The values of activation energy in the different phases of pure 7O.4 and dope 7O.4 sample are given in Table 2. The calculated activation energies of our present samples seems to be of correct order when compared to the reported value for activation energy for lower homologue 6O.6, where the activation energy is 3.5 eV in SmA and 1.4 eV and N phase[34]. The values of activation energy for pure and the doped 7O.6 are also calculated assuming an Arrhenius type and are given in Table 2. It is observe that the activation energy for the pure or the doped samples decrease in the smectic G phase with the lowering of temperature. This may be due to the appearance of the new type of ordering in this phase[35]. However in the above ref [36] it was also reported that in smectic G phase even the macroscopic orientation of the sample was destroyed, so the relaxation time could correspond to a parallel or perpendicular component. This must be the reason for the observation of dielectric absorption phenomena in the SmG phase of our sample in both the homeotropic (present work) and the planar alignment [34]. In the present study, the activation energy in SmA phase is smaller than nematic phase of 7O.4 (both in pure and doped samples). This type of behavior was also reported elsewhere [36-38]. In the report [36], it was also noted that the plot of  $\log(\text{Relaxation frequency})$  versus inverse of temperature did not fit to a straight line, but a curved one. Such behaviour is seen in our study. This could be related to the observance of the increase in activation energy in SmC but decreasing again in SmG phase. However, the change in activation energy was reasoned in different way. It can come from a change of orientator potential or from a change of diffusion coefficient[39]. The first one is linked to interaction effects between molecules while the second is linked to steric effects. It can also be due to both. The observed change in activation energy for the doped samples (Table 2) as compared to that of pure samples must definitely be related to a change in interaction between the SNP/LC molecules.

**Table 2. Activation energy for the pure and the doped samples-7O.4 and 7O.6**

Samples	Pure 7O.4 (in eV)	Doped 7O.4 (in eV)	Pure 7O.6 (in eV)	Doped 7O.6 (in eV)
LC phases				
SmG	4.363817	4.028603	2.326680	3.230638
SmF			6.937814	6.910239
SmC	6.111412	5.255711	6.468170	6.060570
SmA	4.155278	2.797186	2.559348	2.236198
N	7.066213	5.575414		



## CONCLUSION

In summary, it is found that the dielectric permittivity and the dielectric loss factor values decrease when the LC samples are doped with SNPs. Dielectric loss peaks are slightly lowered in comparison to the pure LC samples. The presence of the SNPs in the LC samples lead to an increase in distribution parameter  $\alpha$  and also shifts the relaxation frequency to higher frequency region. The SNPs produce some sort of disorder in LC samples - 70.4 and 70.6 possibly leading to a decrease in observed dielectric loss. We believe that the observed changes in the activation energy for the doped samples in comparison to those of pure samples are caused by the possible change in the local environment (local field) of the LC host due the presence of nanoparticles.

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