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Dielectric relaxation and electrical properties of $ZnO_{1-x}S_x$ nanoparticle dispersed ferroelectric mesophase

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ABSTRACT

The $ZnO_{1-x}S_x$, metal oxide nanoparticles (MNPs) have been dispersed in the ferroelectric mesophase (FLC). The electrical properties and dielectric relaxation processes have been studied for the MNPs dispersed FLC system with the variation of frequency and temperature. The dielectric measurements have been carried out in the frequency interval of 1Hz-10MHz to investigate different relaxation processes. Three different relaxation modes have been observed in the case of the pure FLC at frequency 2.5Hz, 20Hz and Goldstone relaxation mode at 200Hz. The addition of MNPs, suppressed the relaxation mode observed at 2.5 Hz for the pure FLC whereas the relaxation mode observed at 20 Hz is shifted to the higher frequency side. The conductivity and the relative permittivity of the pure FLC have also been enhanced by the dispersion of the MNPs. The present investigation establishes the MNPs as an intelligent material to tune the relaxation process and to enhance the conductivity of the materials. Copyright © 2013 VBRI press.

Keywords: Ferroelectric mesophase; nanoparticles; relaxation mode; conductivity.

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Introduction

Chiral smectic C (SmC*) mesophase of the liquid crystal (LC) materials **[1]** evinces some spectacular properties like fast response time of micro second order, wide viewing angle, and memory effect **[2-4]**. The SmC* phase is characterized by the chirality and tilting of the molecules with respect to the layer normal. The molecules presses around the layer normal along the smectic tilt curve with finite phase angle (Φ) from layer to layer resulting in the helical structure. By virtue of the helical pattern of the molecules, this phase has ferroelectricity. These LCs are generally termed as ferroelectric liquid crystals (FLCs) **[5]**. The helical superstructure arrangement of the molecules in FLC phase gives rise to the collective motions around the phase angle and tilt angle. This fluctuation in the tilt angle (θ) of the molecules gives rise to the soft mode in FLC materials whereas the phase angle fluctuations (Φ fluctuations) results the Goldstone mode of relaxation. The soft mode can be observed after suppressing the Goldstone mode by applying a bias field near the transition temperature **[6]**. Broadband dielectric spectroscopy is an ideal tool to analyze the collective relaxation phenomenon and to study the electrical properties of the materials **[7]**.

In addition to these dielectric relaxation processes, a number of other dielectric relaxation modes have also been investigated in the FLC materials. Uehara et al. **[8]** has observed a very low frequency mode associated with the space charge accumulation on the interface of the LC and polymer layer, while domain modes have been observed due to formation of surface and bulk ferroelectric domains in the FLCs having high spontaneous polarization value. Novotna et al. **[9]** has reported a thickness dependent low frequency mode near 50 Hz in SmC* phase of the FLC material. This mode was attributed to the fluctuations of the director field modified by a non-homogeneous ionic charge distribution across the sample. Havriliak et al. **[10]** has studied low frequency dielectric analysis of a FLC material in SmC* phase and observed two separate peaks in the loss polarisability. They have also examined the shifting of the relaxation frequency with the variation of bias field and temperature. Biradar et al. **[11]** has observed a Debye- type relaxation at a very low frequency for a non-chiral LC material. They have attributed it to the space charge accumulation in the surface alignment layer. Dielectric relaxation behaviour of a fluorinated anti-ferroelectric LC was performed by another group and they found five different modes of relaxations in different phases of the material **[12]**.

The field of nanotechnology has attracted the immense interest of the researchers because of its vital applications in all the fields of research and technology. A large number of researchers are well dedicated in synthesizing the different kind of nanomaterial like nanoparticles, nanotubes, quantum dots etc. Nanoparticles (NPs) are intelligent nanomaterial **[13]** which has shown their utility to alter the properties of any materials and the new properties of the material based on NPs are more useful. Therefore, nanotechnology based devices are the key factor for future technology **[14]**. Spherical shape $ZnO_{1-x}S_x$ NPs have been synthesized by typical co-precipitation method. The present NPs are spherical in shape and have large surface to volume ratio similar to other NPs. These NPs have ZnO and ZnS phase simultaneously which is clear from XRD spectra. Due to large surface to volume ratio, FLC molecules shows greater interaction with the NPs, therefore, the conductivity and other properties of the FLC material will enhanced with the addition of the NPs.

The doping of nanoscale materials in LCs has resulted in the improvement in the properties of the nanomaterial based LC composite systems **[15-18]**. Biradar et al. **[19]** has reported the study of relaxation processes in gold nanoparticle-FLC based composite system and has also shown that the presence of Alumina nanoparticles affect the relaxation mode and diminish the ionic effects at lower frequencies **[20]**. Recently Mandal et al. has also reported the sub hertz relaxation process in chiral smectic mixtures doped with Ag NPs **[21]**.

In the present paper, we report the dielectric relaxations of a FLC mixture in the frequency region of 1 Hz-10 MHz. The effect of the addition of metal oxide nanoparticles (MNPs) on the relaxation modes has also been discussed. The presence of MNPs causes the suppression of relaxation mode found near the 2.5 Hz frequency whereas the relaxation mode found near the 20 Hz frequency has been shifted to the higher frequency side. No noticeable change in case of the Goldstone relaxation mode has been observed. The value of conductivity has also been enhanced with the dispersion of MNPs for the studied frequency and temperature range. The effect of large surface to volume ratio of the NPs on the conductivity of the FLC material and the ability of the NPs to tune the relaxation processes of the FLC material is the main focus of the present investigation.

Experimental

FLC Materials

The investigated FLC material used in the present study is Felix 16/100 (commercial FLC mixture purchased from Clariant Chemicals Co. Ltd. Germany) and the phase sequence of the FLC mixture is Cr, SmC*, SmA, N*, Iso at -20^{0}C, 72^{0}C, 82^{0}C and 90-94^{0}C. The standard values of spontaneous polarization, cone angle and rotational viscosity of the pure FLC sample at 25^{0}C are -10.5 nC/cm^2, 54.30 ^{0}, 60 mPas respectively.

Metal Oxide nanoparticles (MNPs)

In a typical co-precipitation method nominal composition of zinc acetate dehydrate $\{Zn(CH_3COO)_2.\ 2H_2O\}$ and sodium sulphide (Na_2S) were used. Appropriate amount of zinc acetate dehydrate and 0.5 molar oxalic acid were dissolved in methanol with vigorous stirring for 30 minutes. In the clear solution obtained thus, aqueous solution of sodium sulphide was added very slowly drop by drop at 5° C with constant stirring for two hours, which was then subjected to 150 kPa pressure for two hours in an autoclave. The resulting white solid product was centrifuged, washed several times with double distilled water and absolute ethanol to remove the possible impurities left in the final product before drying it. The prepared spherical shape $ZnO_{1-x}S_x$ nanocrystals were characterized by X-ray diffraction (XRD), and transmission electron microscopy (TEM), in order to elaborate structural properties in a very precise manner. All the chemicals used

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to synthesize the nanomaterial were purchased from Sigma-Aldrich of purity 99.999%.

Spherical shape $ZnO_{1-x}S_x$ nanoparticles (NPs) were used to prepare the FLC-MNPs composite. **Fig. 1** shows the XRD spectra of the prepared $ZnO_{1-x}S_x$ nanoparticle whereas the inset of the **Fig. 1** shows the SEM image of the NPs. XRD Spectra show broad peaks at the positions of 31.63°, 34.50°, 36.25º, 47.50°, 56.60°, 62.80°, 66.36°, 67.92° and 68.91°. These peaks are in good agreement with the standard JCPDS file for ZnO (JCPDS number 36-1451, a = b = 3. 249 Å, c = 5. 206 Å) and can be indexed as the hexagonal wurtzite structure of ZnO having space group P63mc whereas peak at the position of 28.52°, is also in good agreement with the standard JCPDS file for ZnS (JCPDS number 39-1363, a = b = 3.82 Å, c = 24.96 Å) and can be indexed as the hexagonal wurtzite structure of ZnS having space group P63mc. This means $ZnO_{1-x}S_x$ alloys the XRD spectra showed the presence of all the peaks of ZnO as well as of ZnS, indicating the formation of intermediate species. The diameter of the MNPs was found to be 10-15 nm.

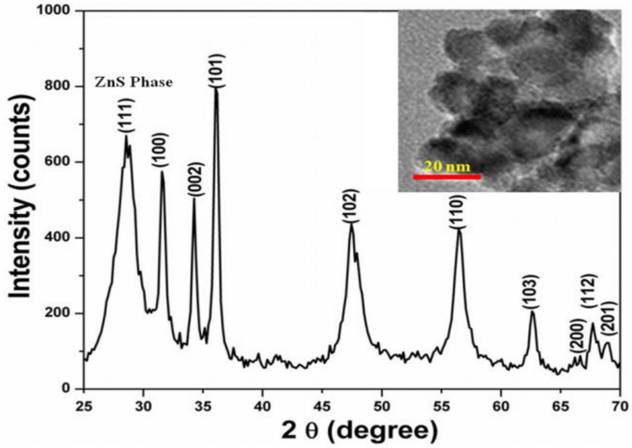

Fig. 1. X-Ray diffraction spectra of the spherical shape $ZnO_{1-x}S_x$ metal oxide nanoparticle. The inset of the figure shows the Scanning electron microscopic image (SEM image) of the nanoparticles.

Preparation of Sample Cell

The Dielectric study of the FLC sample was conducted on planar geometry. The sample cells for the present study were prepared using Indium tin oxide (ITO) coated glass plates. The thickness of the sample cells were maintained ≈ 6μm by using Mylar spacers. The empty sample cells were calibrated using analytical reagent (AR) grade carbon tetrachloride (CCl_4) and benzene (C_6H_6) as standard references for dielectric study. To prepare the FLC-MNPs composite, an appropriate amount (in the weight ratio i.e. 1%) of the spherical shape MNPs were mixed into the pure FLC and then homogenized with an ultrasonic mixer at 90^0C for one hour and uniform dispersion of MNPs was ensured. The pure and the MNPs dispersed FLC were filled in the assembled cells at a temperature higher than the isotropic temperature of the FLC sample by capillary method. It was cooled slowly under a.c. electric field after filling the sample in the cell and the alignment of the sample was confirmed under the crossed polarizing microscope. The detailed information about the preparation of the sample cell and the filling of the pure and the doped FLC sample has been reported earlier **[18]**.

Dielectric Measurement

The Dielectric measurements have been performed by a computer controlled Impedance/Gain Analyzer (Solartron SL1260) in the frequency range 1Hz to 10MHz. The dielectric measurements have been carried out as a function of frequency and temperature by placing the sample on computer controlled hot plate INSTEC (HCS-302) .The temperature stability was better than ±0.1^{0}C. The experimental data has been corrected for low frequency values due to the electrode polarization capacitance and ionic conductance and high frequency effect due to the ITO resistance and lead inductance. **[22-23].**

The low frequency dielectric relaxation phenomenon of the pure FLC and the MNPs doped FLC sample has been analyzed using Cole-Cole relation **[24]**.

$$\varepsilon^* = \varepsilon' - \varepsilon'' = \varepsilon(\infty) + \frac{\varepsilon_0 - \varepsilon_\infty}{1+(i2\pi f\tau_0)^{1-\alpha}} - i\frac{\sigma}{2\pi f\varepsilon_S} \qquad (1)$$

Where, f is the frequency, τ_0 is the relaxation time, α is the distribution parameter, ε' and ε'' are the real (dielectric permittivity) and imaginary (dielectric loss) parts of the ε^* (complex dielectric permittivity) respectively, ε_0 and ε_∞ are the low and high frequency limits of the electric permittivity, ε_S is the electric permittivity of the free space and σ is the conductivity.

Results and discussion

The dielectric spectroscopy is a useful tool to investigate the relaxation processes, which arises due to the rotational fluctuations of the molecular dipoles. In the SmC* phase (or ferroelectric phase), chiral FLC molecule are tilted by an angle θ to the director. Two types of fluctuations are possible in this situation i.e. along the phase angle (Φ) and tilt angle (θ).These two fluctuation result in the appearance of the Goldstone mode (GM) and the Soft mode (SM) respectively in the SmC* phase of the FLC sample. In the present investigation, we have observed the phase angle fluctuation near 200 Hz, which is analogous to Goldstone relaxation mode. The soft mode was absent in the entire SmC* phase and also near the SmC*-SmA phase transition temperature. The tanδ (i.e. the ratio of the imaginary and real part of the complex dielectric permittivity, ε′′/ε′) curves have been plotted against the log of the frequency scale (for frequency range 100 Hz-10MHz) at two different temperatures i.e. 35^0C (temperature in SmC* phase) and 72^0C (SmC*-SmA phase transition temperature) for the pure and the MNPs dispersed FLC which are shown in **Fig. 2(a)** and **2(b)** respectively. The inset of the **Fig. 2(a)** and **2(b)** shows the Cole-Cole plots to analyze the existence of the goldstone and soft mode.

From **Fig. 2**, the Goldstone mode of relaxation is well observed near 200 Hz at 35^0C and 72^0C temperature. However, the Soft mode (SM) has not been observed at both the temperature. The soft mode mainly appears at the SmC*-SmA phase transition temperature, therefore the existence of the soft mode was checked at 72^0C. The Cole-Cole plots (inset of **Fig. 2a** and **2b**) have also been investigated to analyze the existence of different relaxation mode. The Cole-Cole plots clearly indicates the existence of the single relaxation process due to phase angle fluctuation i.e. Goldstone mode at 200 Hz. The decreased value of tanδ indicates that the dispersion of the MNPs reduces the loss factor in the FLC-MNPs composite. This happens due to the adsorption of the free ions present in the FLC material on the spherical surface of the MNPs.

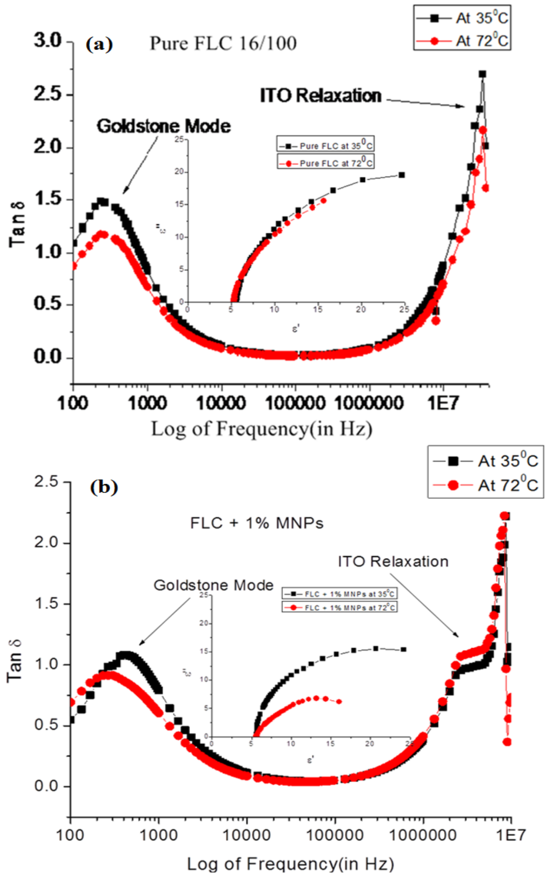

Fig. 2. The variation of tanδ for the (a) pure FLC and the (b) MNPs dispersed FLC at 35^{0}C and 72^{0}C. The inset figures show the Cole-Cole plots for the same samples.

The low frequency relaxation process (in the frequency interval of 1Hz-100Hz) has also been investigated for both the pure and the MNPs dispersed FLC. The tan δ against the log of the frequency scale at 35^{0}C for the pure and the MNPs dispersed FLC have been plotted in the **Fig. 3(a)** and **3(b)** respectively.

Two relaxation modes have been observed at 2.5 Hz and 20 Hz in the frequency interval of 1Hz-100Hz. The tan δ curve has also been fitted theoretically in the equation 1 to avoid the existence of the spurious relaxation process influenced by the impurity ions or additional free ions present in the low frequency region. The fitted tanδ curve clearly indicates the existence of two relaxation modes in the frequency interval of 1Hz-100Hz. The relaxation mode at 2.5 Hz frequency is related to fast ions in the ionization-recombination assisted diffusion **[25]** whereas the relaxation mode at 20 Hz frequency is related to the again fusion of the diffused ions. The influence of MNPs suppresses the ionization-recombination assisted diffusion in the low frequency region. The relaxation mode at 2.5 Hz frequency is suppressed due to the presence of the MNPs which indicates that the presence of MNPs reduces the rate of ionization-recombination assisted diffusion of the fast ions. In addition to this, the relaxation mode at 20 Hz frequency has also been shifted towards the higher frequency side due to influence of the MNPs. This shifting of the relaxation mode is due to the presence of guest MNPs in the pure FLC matrix. The shifting of the relaxation frequency with the addition of silica nanoparticle has been reported recently by Malik et al. **[26]**. However, in the low frequency region, most of the effect comes from the ionic contaminants and observation need a good repeatability, therefore, the experimental process was repeated five times. UV radiation was also exposed to the Pure FLC and the MNPs dispersed FLC sample cells to analyze the effect of impurity ions in the low frequency region. No noticeable change on the relaxation modes was observed due to the exposure of the UV light. Biradar et al. **[19]** has also been represented the low frequency mode attributed to ionic-recombination assisted diffusion of slow ions present in FLC material and also showed that the presence of the Al nanoparticle reduces the ionic effects in the low frequency region **[20]**. These low frequency relaxation processes are mostly thickness dependent **[27]**, therefore we have also observed the effect of the thickness on the relaxation modes. But in our case due to weak polar surface anchoring of the sample, thickness dependent nature of the relaxation mode has not been observed.

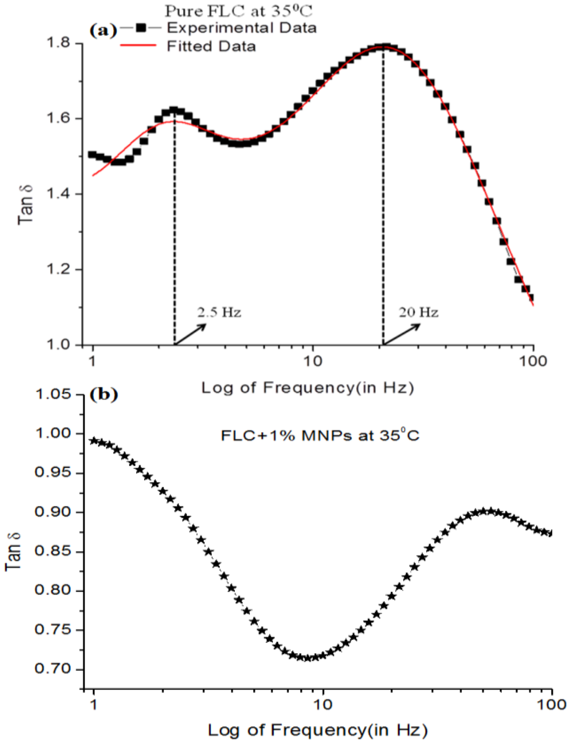

Fig. 3. The change in tanδ for the (a) pure FLC and the (b) MNPs dispersed FLC in the frequency interval of 1Hz-100Hz at 35^{0}C. Red solid line in tanδ curve denotes the theoretical fit of the experimental data.

The variation of the relative permittivity on the temperature scale for the entire SmC* phase has been observed at two different frequencies (i.e. 1Hz and 200 Hz) and results are depicted in the **Fig. 4(a)** and **4(b)** respectively. The non-linear trend of the relative permittivity on the temperature scale has been observed for the pure FLC at 1 Hz frequency. At 1 Hz (**Fig. 4a**), the

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relative permittivity of the pure FLC increases nonlinearly on the temperature scale whereas the relative permittivity of the MNPs dispersed FLC is almost temperature independent. At 200 Hz (**Fig 4b**), the relative permittivity of the pure and the MNPs dispersed FLC increases slightly with increase in the temperature and achieves its maximum values. As temperature approaches to the SmC*-SmA phase transition temperature, the relative permittivity decreases sharply. But the value of the relative permittivity for the MNPs dispersed FLC is higher than that of the pure FLC in both the cases. The value of relative permittivity for the MNPs dispersed FLC at 1 Hz is approximately 2.5 fold as compared with the pure FLC whereas at 200 Hz, it is only 1.28 fold. This enhancement in the value of relative permittivity for the MNPs dispersed FLC is due to the parallel correlation of the dipole moments between the MNPs and the FLC molecules **[28]**. In addition to this, the enhancement in the value of the relative permittivity for the MNPs dispersed FLC is frequency dependent. This enhancement in the relative permittivity is higher at lower frequency (i.e. 1Hz) as compared with the values at 200 Hz frequency. Additionally, we have not observed any noticeable change in the SmC*-SmA phase transition temperature for the MNPs dispersed FLC.

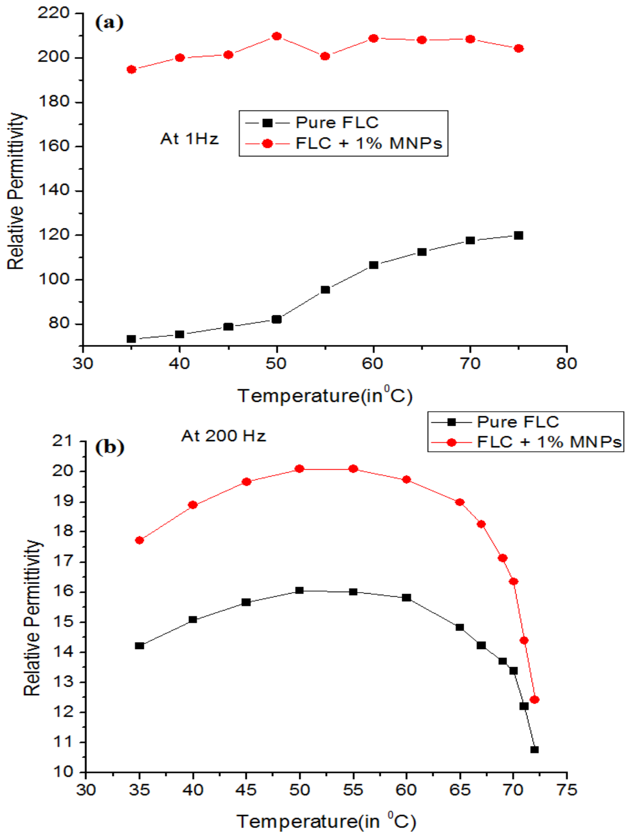

Fig. 4. The change in relative permittivity on the temperature scale for the pure and the MNPs dispersed FLC at (a) 1 Hz, and (b) 200 Hz.

The presence of MNPs also affects the conductivity of the pure FLC. The conductivity of the MNPs dispersed FLC system has also been evaluated at two different frequencies. The variation of the conductivity with the change in temperature at 1 Hz and 200 Hz has been shown in the **Fig. 5(a)** and **5(b)** respectively. The variation of conductivity of the MNPs dispersed FLC system is not similar for both the frequencies due to the symbioses of the MNPs and the free ions present in the FLC material. The presence of MNPs rouses the motion of the free ions near the 200 Hz frequency while it is less effective at lower frequency (i.e. near 1 Hz) but the value of conductivity for the MNPs dispersed FLC is greater than that of the pure FLC. The value of conductivity for the MNPs dispersed FLC system is almost two and four times as compared with pure FLC at 1Hz and 200 Hz respectively. The conductivity at 1 Hz for the pure FLC increases slightly with the increase in temperature whereas for the MNPs dispersed FLC, The conductivity increases nonlinearly with the increasing temperature. The conductivity at 200 Hz for the pure FLC is almost constant on the temperature scale, while it increases nonlinearly with increase in the temperature for the MNPs dispersed FLC. The behavior of the conductivity for the MNPs dispersed FLC is mainly driven by the semiconducting nature of the MNPs; therefore the conductivity of the MNPs dispersed FLC increases with increase in the temperature like semiconducting materials but the additional ZnS phase present in the MNPs affect the real trend of the conductivity. The overall conductivity of the MNPs dispersed FLC is greater than that of the pure FLC.

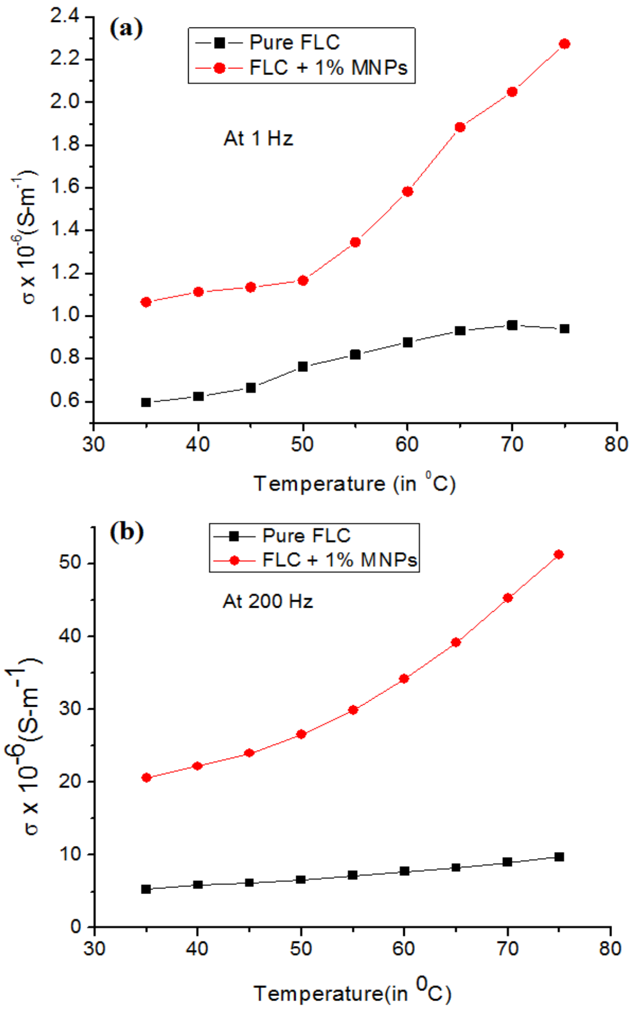

Fig. 5. The nature of electrical conductivity with the variation of temperature for the pure and the MNPs dispersed FLC at (a) 1 Hz, and (b) 200 Hz.

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At 1 Hz, conductivity of the MNPs dispersed FLC is almost two times as compared with the pure FLC and at 200 Hz, it is four times. For small electric field strength, the dielectric displacement (D) is directly related with the complex dielectric function ($D=\varepsilon^{*}\varepsilon_0 E$) whereas the complex electrical conductivity is also directly related with ε^{*} by ($\sigma^{*}=i\omega\varepsilon_0\varepsilon^{*}$). The current density ($j=\sigma^{*}E$) and the time derivative of the dielectric displacement are equivalent quantities, therefore, the greater value of relative permittivity is directly related with the electrical conductivity. The present observation supports this physical phenomenon in the presence of MNPs which is an interesting result of the present study. The present investigation indicates the importance of the synthesis of metal oxide NPs due to their capacity to alter the materials properties. In NPs based composite system, the alteration in the electrical or relaxation phenomenon occurs due to the modification in net dipole moment or easier charge transportation due to high surface to volume ratio of the NPs.

Conclusion

In summary, we investigated the behavior of electrical property and relaxation modes of a pure and the MNPs dispersed FLC for a definite frequency regime. The effect of the dispersion of MNPs on these relaxation modes and electrical property has also been discussed. Three different relaxation modes for the pure FLC systems have been found in the low frequency region. The relaxation modes at 2.5 Hz, 20 Hz and 200 Hz are related to the motion of fast ions in the ionization-recombination assisted diffusion, again fusion of the diffused ions and phase angle fluctuation of the FLC molecules respectively. The presence of MNPs reduces the contribution of free ions in the low frequency region. Therefore, the mode at 2.5 Hz has been suppressed in the presence of MNPs. Another low frequency mode at 20 Hz has been shifted towards the higher frequency side due to the dispersion of MNPs in the pure FLC. The relative permittivity of the MNPs dispersed FLC is also greater than that of the pure FLC which is analogous to the parallel correlation of the dipole moments of the MNPs with those of the FLC molecules. The conductivity of the pure FLC has also been enhanced with the addition of the MNPs and semiconducting nature of the MNPs is mainly responsible for the behavior of the dispersed system. This enhanced relative permittivity with enhanced conductivity is an interesting finding of the present study.

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