Dielectric and electro-optical studies of glycerol/ferroelectric liquid crystal mixture at room temperature

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The optical memory effect based on glycerol mixed deformed helix ferroelectric liquid crystal (DHFLC) material has been investigated. The observed memory effect has also been compared with the glycerol mixed ferroelectric liquid crystals (FLCs). The observance of memory effect has been verified by experimental data using dielectric spectroscopy, electro-optical, and textural studies under polarizing optical microscope. The glycerol not only helps to improve the memory effect in DHFLC material but also alters the physical parameters such as rotational viscosity, spontaneous polarization, response time, etc. too in both glycerol/DHFLC and glycerol/FLC mixtures. © *2009 American Institute of Physics*. [DOI: 10.1063/1.3149781]

I. INTRODUCTION

The studies of alcohols and their mixing/doping in other inorganic/organic materials have drawn a great deal of interest in fundamental and applied research because of their versatility and hydrogen bonded network since the past few decades. The dielectric properties of alcohols, alcohol-alcohol mixture, and of alcohol water mixture have received much attention, particularly due to potentiality of these systems to elucidate the mechanism of hydrogen network fluctuations of associating liquids.[1–8] Glycerol has been found the most versatile solvent because it possesses hydrogen bonded network, high boiling point, and high viscosity. Moreover, it supercools easily below 291 K and forms a glass at 185 K.[9] The antiplasticizing effect of glycerol on trehalose has been studied in the concern of increment preservation time of proteins.[10] The effect has been demonstrated on the basis of dielectric spectroscopy in a broad frequency range between 40 Hz and 18 GHz. Few studies have been carried out on the dielectric behavior of lysozyme and ferricytochrome-c using glycerol and water/ethylene glycol solution at radio frequencies.[11] The dielectric properties of glycol/water mixture at temperature below 10 and 50 °C have been carried out by Behrends *et al.*[8] and the authors demonstrated the formation of microphases. Lunkenheimer *et al.*[12] performed dielectric spectroscopy on supercooled glycerol for temperatures between 250 and 330 K. The doping/mixing of glycerol in liquid crystals (LCs) may bring drastic change in physical parameters of these materials which were hidden in these materials.

Most of reported work on such studies is based on nematic LCs (NLCs). Various studies have been carried out on crystal structure formed by glycerol droplets suspended in NLCs and explained by elastic capillary interactions.[13,14] Subsequently, it has also been observed that the glycerol droplets form the different lattices-hexagonal and dense quasihexagonal which can coexist and are separated by the energy barrier.[15] Yamamato *et al.*[16] demonstrated photomechanical manipulation of glycerol droplets in NLCs on the basis of photoisomerization of azo-benzene derivative. But the mixing of glycerol in ferroelectric LC (FLC), which are well known for their good optical contrast, low threshold voltage, memory effect, fast response, etc., is rather far from investigations. However, our group has been investigated the enormous change in various parameters of electroclinic LCs (a special class of FLCs) such as tilt angle, transition temperature, etc. by dielectric[17] and electro-optical[18] studies. The deformed helix FLCs (DHFLCs), which are a special kind of FLC, are very useful and have much applications in display devices because of their low driving voltage, gray scale generation capability, easily achievable alignment, fast response, etc.[19,20] The bistability or memory effect has also been demonstrated by us[21–23] in DHFLCs.

In the present paper, the optical memory effect based on glycerol/DHFLC mixture has been observed. The observed memory effect has also been compared with the memory effect in glycerol/FLC mixture. The material parameters such as response time, rotational viscosity, and spontaneous polarization have also been studied in glycerol/DHFLC and glycerol/FLC mixtures. The experimental results have been analyzed using dielectric relaxation spectroscopy, polarizing optical microscopy and other electro-optical studies.

II. EXPERIMENTAL

For this study, homogeneously aligned cells (thickness of 3 μm) of the glycerol mixed DHFLC material (FLC 6304, Rolic, Switzerland) and glycerol mixed FLC material (CS 1026, Chisso, Japan) have been prepared. The homogeneous alignment was obtained by coating the indium tin oxide coated optically flat (flatness of $\lambda/2$) glass plates with nylon 6/6 and then rubbing by using a buffing machine (LCBM, U.S.A.). The phase sequence of the materials used is as follows:

$$\text{cryst} \overset{-14\ ^\circ\text{C}}{\leftrightarrow} \text{SmC}^* \overset{60.5\ ^\circ\text{C}}{\leftrightarrow} \text{SmA} \overset{64\ ^\circ\text{C}}{\leftrightarrow} \text{iso} \quad \text{(FLC 6304)},$$

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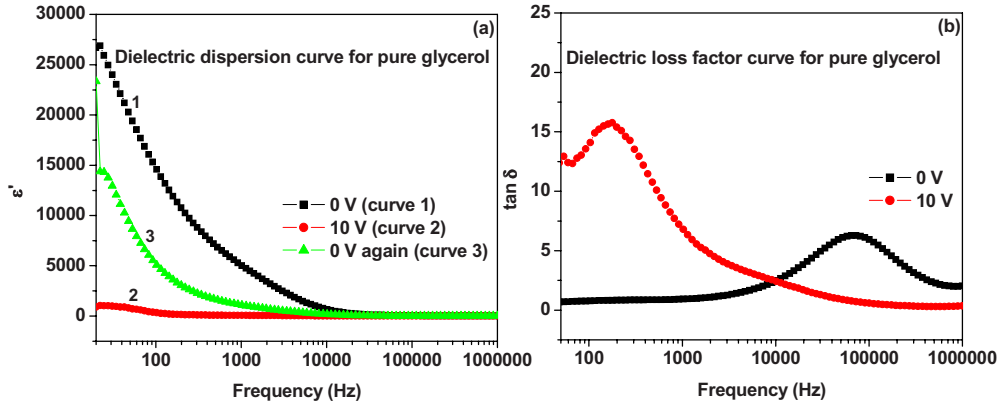

FIG. 1. (Color online) (a) Dielectric permittivity at 0 V (curve 1), 10 V (curve 2), and again at 0 V (curve 3) and (b) dielectric loss factor as a function of log of frequency (log ν) of pure glycerol at room temperature.

$$\text{cryst} \overset{-7\ ^\circ\text{C}}{\leftrightarrow} \text{SmC}^* \overset{64\ ^\circ\text{C}}{\leftrightarrow} \text{SmA} \overset{82\ ^\circ\text{C}}{\leftrightarrow} \text{N} \overset{91\ ^\circ\text{C}}{\leftrightarrow} \text{iso} \quad \text{(CS 1026)}.$$

The glycerol was mixed in LC at 75 °C far below its boiling point. The concentration of glycerol in LCs (either in DHFLC or in FLC) was taken at 0.5 μl/2 mg. The mixture (glycerol/DHFLC or glycerol/FLC) was introduced into the cells by means of capillary action at elevated temperature to ensure that filling takes place in the isotropic phase. The smectic layers were arranged with layer planes perpendicular to the cell surfaces. The dielectric measurements of the sample cells have been taken using impedance analyzer 6540A (Wayne Kerr, U.K.) on the application of low ac measuring voltage of 0.5 V. The electrical response has been observed by applying pulses of triangular shape generated from a pulse generator to the sample. For optical response, the sample was mounted on a polarizing microscope and the transmission of normally incident polarized light through the sample and analyzer was monitored with a photodiode. The time delayed square pulse generated from the pulse generator was applied to the sample and studied by using a storage oscilloscope (HM 1507–3, HAMEG, Germany) interfaced with the computer via SP-107 software. The determination of material constants such as response time, spontaneous polarization, and rotational viscosity has been carried out by using automatic LC tester (ALCT-P Instec, U.S.A.). The optical micrographs of the cells have been taken using polarizing optical microscope (Ax-40, Carl-Zeiss, Germany).

III. RESULTS AND DISCUSSIONS

A. Dielectric measurements

The dielectric properties (studies) provide the information on the structure and mechanism of molecular processes.[24] The dielectric studies of glycerol have been performed by several groups earlier.[24–26] The temperature dependence of the quasistatic dielectric permittivity of highly viscous glycerol has also been discussed.[25] Figure 1 shows the dielectric permittivity and dielectric loss factor of glycerol as a function of frequency at different applied voltages. One can see clearly from the figure [Fig. 1(a)] that the dielectric permittivity of glycerol is quite high, particularly at lower frequencies. The high dielectric permittivity may be due to the hydrogen bonded network of glycerol. In the dielectric loss factor spectrum, glycerol shows a nice peak, as shown in Fig. 1(b). Moreover, the peak shifts to lower frequency side as one applies bias to the sample cell of glycerol. At present, it is very difficult to interpret the dielectric permittivity in highly viscous liquids and in the system whose structure is determined by hydrogen bonded network, glycerol being a typical example, because the satisfactory theory of dielectric systems is absent. The very fascinating thing with glycerol which attracts us to mix it in DHFLC/FLC material is the memory effect shown by glycerol [Fig. 1(a)]. As can be seen from Fig. 1(a) the change in bias voltage of measuring field from 0 to 10 V, the dielectric permittivity decreases to minimum. Again on applying 0 V bias the permittivity appears the same as in the presence of bias, meaning thereby, the glycerol remains in memory state up to some extent.

Now we take the effect of mixing glycerol in DHFLC material on the dielectric measurements of glycerol/DHFLC mixture. Figures 2(a) and 2(b) show the dielectric permittivity and dielectric loss factor of glycerol/DHFLC mixture as a function of frequency. First, the dielectric permittivity of glycerol/DHFLC mixture has been compared with pure DHFLC material [Fig. 2(a)]. As can be seen from the figure the glycerol/DHFLC mixture possesses higher permittivity value (almost 2.5 times higher) than the pure one. The high dielectric permittivity in the glycerol/DHFLC mixture is due to high dipole moment of glycerol than the DHFLC material. Second, the dielectric loss factor spectra of glycerol/DHFLC mixture and pure DHFLC materials have been compared, which has been shown in Fig. 2(b). Two peaks can be seen clearly in the dielectric loss factor spectrum of glycerol/DHFLC mixture. The first peak clearly shows the presence of glycerol because the second peak appears almost at the same frequency at which the peak of pure DHFLC material appears. Figures 2(c) and 2(d) show the Cole-Cole plots of pure DHFLC and glycerol/DHFLC mixture, respectively, to confirm whether glycerol molecules introduce a distribution of the dielectric relaxation times or not. The analysis of Cole-Cole plots [Figs. 2(c) and 2(d)] at room temperature reveal that single collective process is observed in deep SmC* phase in pure DHFLC material, whereas two processes, Goldstone mode and other due to the presence of glycerol, are observed in glycerol/DHFLC mixture at room

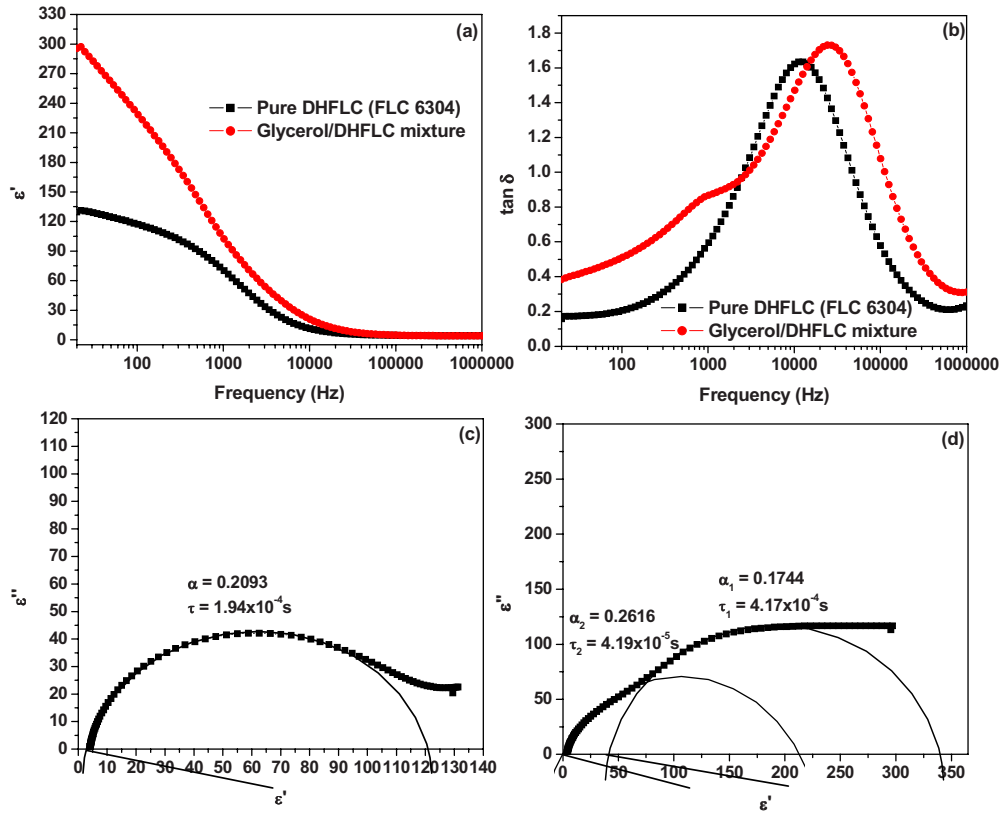

FIG. 2. (Color online) (a) Dielectric permittivity and (b) dielectric loss factor as a function of log of frequency (log ν) of pure DHFLC and glycerol/DHFLC at room temperature. Cole-Cole plots of (c) pure DHFLC and (d) glycerol/DHFLC mixture at room temperature. The value of distribution parameters (α) and dielectric relaxation times (τ) has been indicated in the figure. The subscripts 1 and 2 in part (d) indicate the Goldstone mode and mode due to glycerol, respectively.

temperature. The low frequency values in Figs. 2(c) and 2(d) are due to the conductivity effect. It is worth mentioning here that glycerol molecules introduce a distribution of the dielectric relaxation times in glycerol/DHFLC mixture (as observed in dielectric relaxation and Cole-Cole plots).

The dielectric measurements have also been performed on glycerol/FLC mixture to see whether the glycerol affected only the properties of DHFLC or it affects also the conventional FLCs. Figure 3 shows the dielectric permittivity and dielectric loss factor of glycerol/FLC mixture as a function of frequency. First, the dielectric permittivity of glycerol/FLC mixture has been compared with pure FLC material [Fig. 3(a)]. It has been clearly reflected in the figure [Fig. 3(a)] the glycerol/FLC mixture possesses higher permittivity value (almost 4.5 times higher) than the pure FLC material. The high dielectric permittivity in the glycerol/FLC mixture is again due to the fact that glycerol has high dipole moment than the FLC material. Second, the dielectric loss factor spectra of glycerol/FLC mixture and pure FLC material have been compared which has been shown in Fig. 3(b). Two peaks, as observed in the case of the glycerol/FLC mixture, can be seen clearly in the dielectric loss factor spectrum of glycerol/FLC mixture. The first peak clearly shows the presence of glycerol because the second peak appears almost at

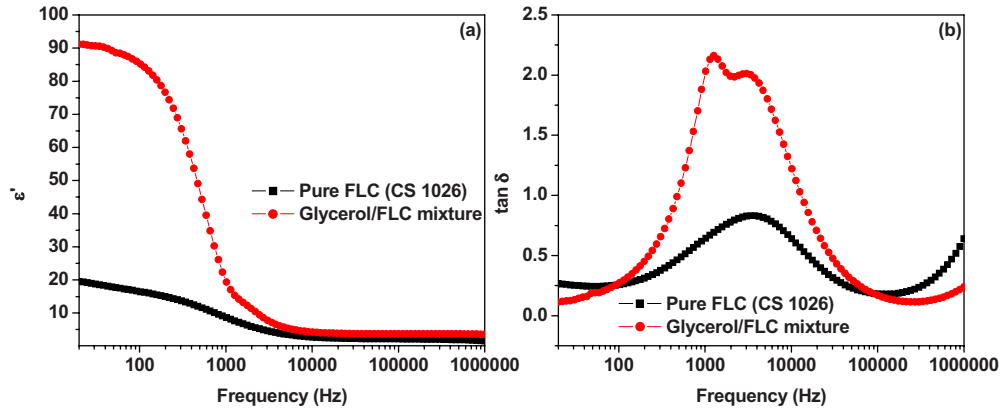

FIG. 3. (Color online) (a) Dielectric permittivity and (b) dielectric loss factor as a function of log of frequency (log ν) of pure FLC and glycerol/FLC at room temperature.

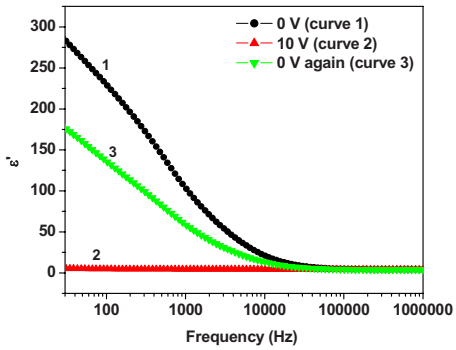

FIG. 4. (Color online) Dielectric permittivity as a function of log of frequency (log ν) at 0 V (curve 1), 10 V (curve 2), and again at 0 V (curve 3) of glycerol/DHFLC mixture at room temperature.

the same frequency at which the peak of pure FLC material appears.

B. Memory effect in glycerol/DHFLC mixture

The memory effect in glycerol/DHFLC mixture has been observed by dielectric, electro-optical, and textural methods. Figure 4 shows the memory effect in glycerol/DHFLC mixture by observing dielectric permittivity as a function of frequency at three different voltages (0, 10, and again at 0 V). As can be seen from the figure, with the change in bias voltage of measuring field from 0 to 10 V, the permittivity value decreases to minimum due to the suppression of phason (Goldstone) mode, which occurs due to phase fluctuation of the molecules.[27] Again on applying 0 V bias, the dielectric permittivity appears the same as in the presence of bias, meaning thereby, the glycerol/DHFLC mixture remains in memory state up to some extent.

Figure 5 shows the electrical response of glycerol/DHFLC mixture. The electrical response of the sample was observed by applying triangular wave pulse of different frequencies and a fixed voltage. We observed a single symmetric polarization peak in entire frequency range. The electrical response of pure DHFLC material has been reported elsewhere,[23] where the criticality of bistability was taken into account. In that report, we found two peaks in low (100–500 mHz) frequency and high frequency (40–70 Hz) regimes while as single peak was observed in middle frequency region (600 mHz–40 Hz). It was suggested there the first peak was due to helix unwinding-winding (deformation) process, whereas the second peak was due to molecular orientation process. When we observed single peak, we reported that there is a complete resonance in between two mentioned processes. Moreover, it has been mentioned there that wherever the single peak was observed the perfect memory effect was there. In the case of glycerol/DHFLC mixture, we exactly observed the same which is necessary for perfect memory effect.

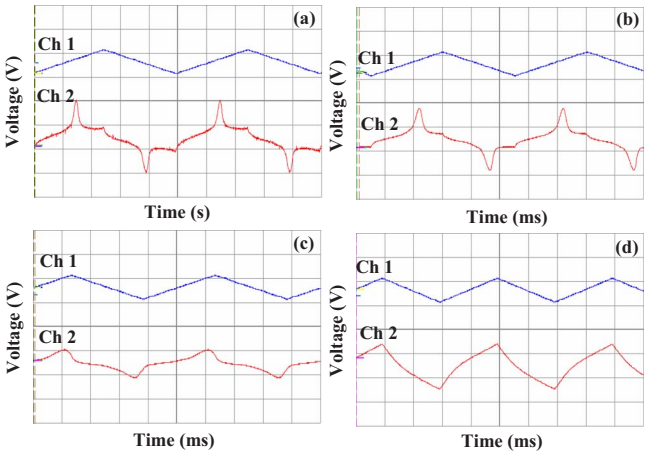

FIG. 5. (Color online) Electrical response of glycerol/DHFLC mixture at room temperature in 3 μm memory cell at 20 V at (a) 200 mHz (Ch 1: 10 V/div, Ch 2: 0.10 V/div, time: 1 s), (b) 1 Hz (Ch 1: 10 V/div, Ch 2: 0.20 V/div, time: 200 ms), (c) 10 Hz (Ch 1: 10 V/div, Ch 2: 1 V/div, time: 20 ms), and (d) 50 Hz (Ch 1: 10 V/div, Ch 2: 2 V/div, time: 5 ms). Ch 1 shows driving triangular voltage and Ch 2 shows its electrical response. The time scale per division is on *x*-axis.

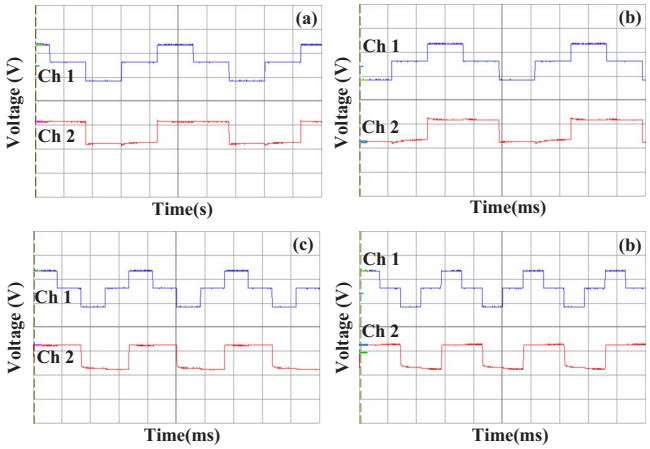

FIG. 6. (Color online) Optical response of glycerol/DHFLC mixture at room temperature in 3 μm memory cell at 20 V at (a) 200 mHz (Ch 1: 10 V/div, Ch 2: 0.020 V/div, time: 1 s), (b) 1 Hz (Ch 1: 10 V/div, Ch 2: 0.002 V/div, time: 200 ms), (c) 10 Hz (Ch 1: 10 V/div, Ch 2: 0.020 V/div, time: 20 ms), and (d) 50 Hz (Ch 1: 10 V/div, Ch 2: 0.020 V/div, time: 5 ms). Ch 1 shows driving time delayed square voltage and Ch 2 shows its optical response. The time scale per division is on *x*-axis

Figure 6 shows the optical response of glycerol/DHFLC mixture at different frequencies and at a fixed voltage. The optical response of the sample was observed by applying time delayed positive and negative square pulses, as shown in Fig. 6. As seen in the figure the optical transmission changes from maximum to minimum as the applied field reverses its polarity and there is almost no change when the applied field attains its 0 V state which confirms the memory effect. The optical response of pure DHFLC material has been observed by us earlier.[23,28] In earlier reports, we mentioned that whenever we had single peak in the electrical response the complete memory effect would be there. Indeed, we were able to observe the same, i.e., single peak in electrical response (Fig. 5) and complete memory effect in optical response in glycerol/DHFLC mixture.

The memory effect found in glycerol/DHFLC mixture by dielectric, electrical response, and optical response was further confirmed by observing the textures in deep SmC* phase by the application of 15 V bias. Figure 7 shows the memory (static) in glycerol/DHFLC mixture. The scattering state, the state before applying any bias, has been shown in

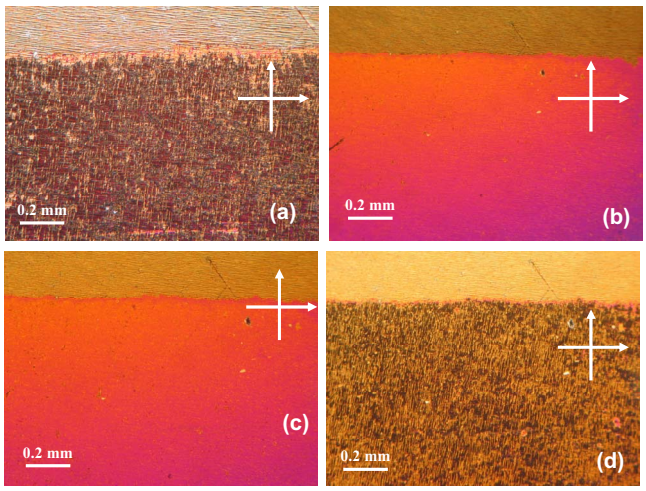

FIG. 7. (Color online) Optical micrographs of 3 μm glycerol/DHFLC mixture memory cell at room temperature at (a) 0 V, (b) 15 V bias, (c) after 12 h of removal of bias, and (d) again at 0 V. Scale bar: 0.2 mm. The cross arrows have been shown to indicate crossed polarizers.

Fig. 7(a). A switched state [Fig. 7(b)] was exhibited when 15 V bias was applied to the sample cell. Figure 7(c) (which was taken after 12 h after removal of bias) shows that the memory state was retained even after a prolonged time when the bias was removed. The memory state was switched back forcibly to the original state (scattering state) which has been shown in Fig. 7(d).

The observation of long lasting memory in glycerol/DHFLC mixture has been attributed to the following possible reasons.

(i) The glycerol is helping in the stabilization of helix deformation which plays a key role for having memory effect in glycerol/DHFLC mixture because the helix stabilization played important role in DHFLC material for having memory effect.[23]

(ii) The glycerol induces the resonance in between helix unwinding-winding (deformation) and molecular orientation processes which has been confirmed by electrical and optical responses (Figs. 5 and 6) in glycerol/DHFLC mixture.

(iii) The glycerol is also helping in the minimization of depolarizing field (which is caused by the charges developed on the surface of bounding glass plates as a result of attaining stable states when electric field is applied to the sample and this developed free charges on the bounding glass plates which turns into the creation of depolarization field) because glycerol possesses high dipole moment than the DHFLC material. If this field (depolarizing field) has been present the memory state would not have been remained in such mixture.

C. Material constants in glycerol/DHFLC mixture

The material parameters such as rotational viscosity, spontaneous polarization, and response time have been studied in glycerol/DHFLC mixture and compared with pure DHFLC material. Figure 8 shows the rotational viscosity, spontaneous polarization, and response time, respectively, as a function of applied voltage in the pure and glycerol/

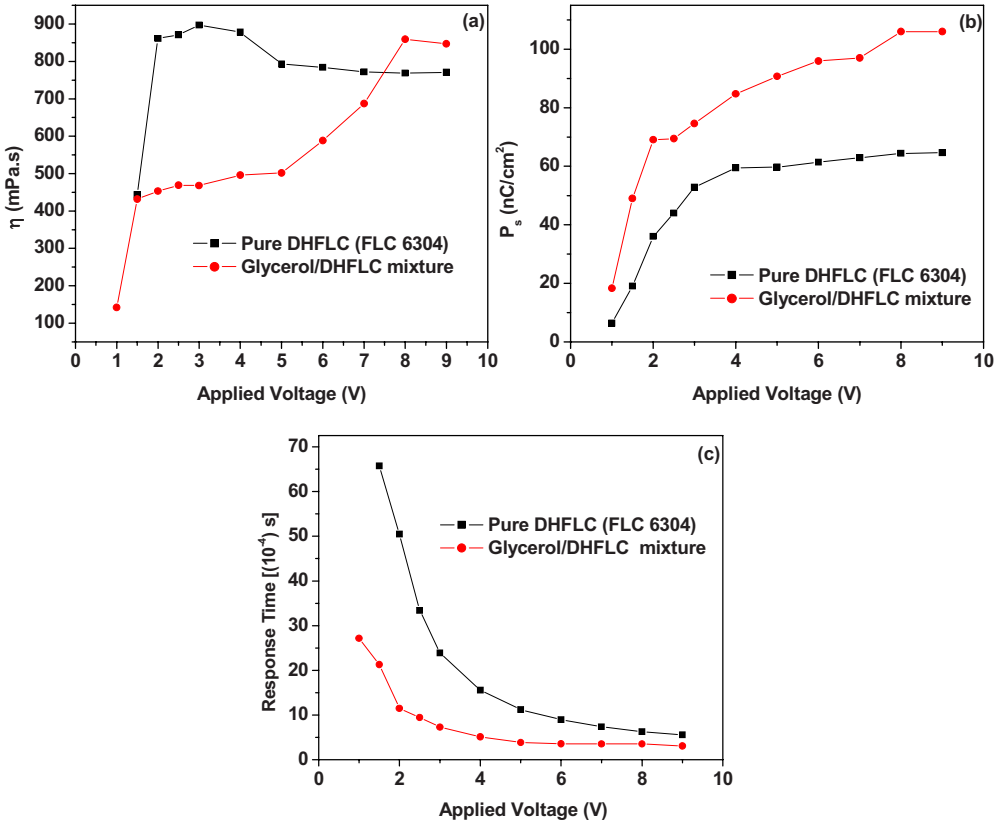

FIG. 8. (Color online) Behavior of (a) rotational viscosity, (b) spontaneous polarization, and (c) response time with applied voltage for glycerol/DHFLC mixture at room temperature.

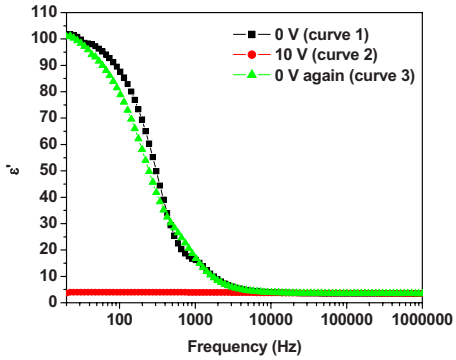

FIG. 9. (Color online) Dielectric permittivity as a function of log of frequency (log ν) at 0 V (curve 1), 10 V (curve 2), and again at 0 V (curve 3) of glycerol/FLC mixture at room temperature.

DHFLC mixture. The rotational viscosity with applied voltage has been shown in Fig. 8(a). The rotational viscosity in glycerol/DHFLC mixture is lower than the pure DHFLC material because the glycerol possesses lower viscosity than the DHFLC material. But if we see the behavior of spontaneous polarization with applied voltage, which has been shown in Fig. 8(b), we find increment in the polarization value for glycerol/DHFLC mixture. The combined effect of decrement in rotational viscosity and increment in spontaneous polarization can be seen in the behavior of response time with applied voltage, which can be seen in Fig. 8(c). These three are related with each other by the following relation

$$\tau = \eta / P_S E \tag{1}$$

where τ is response time, η is the rotational viscosity, P_s is the spontaneous polarization and E is the applied field, respectively. Both the decrement in rotational viscosity and the increment in spontaneous polarization helped to have lower response, which is desirous for fast LC display devices. Indeed, we observed the same what one should expect from Eq. (1), i.e., the response time has been lowered in glycerol/DHFLC mixture.

D. Memory effect in glycerol/FLC mixture

The glycerol/FLC mixture does not show memory effect either by dielectric or by electro-optical methods unlike the glycerol/DHFLC mixture which shows perfect memory. Figure 9 shows the dielectric permittivity as function of frequency in glycerol/FLC mixture. As can be seen from the figure, with the change in bias voltage of measuring field from 0 to 10 V, the permittivity value decreases to minimum which is due to the suppression of phason (Goldstone) mode which occurs due to phase fluctuation of the molecules.[27] Again on applying 0 V bias dielectric permittivity appears same as it was before applying the bias. This confirms that the cell does not remain in stable state and hence no memory effect. Figure 10 shows the optical response of glycerol/FLC mixture at different frequencies and at a fixed voltage. The output response appears as the same as the input unlike the glycerol/DHFLC mixture where the complete memory effect was observed by optical response.

Figure 11 shows the optical micrographs of the cell filled with glycerol/FLC mixture. Figure 11(a) shows the scattering

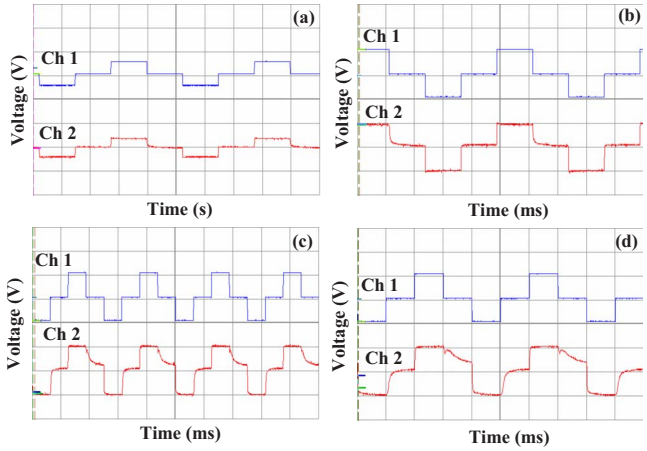

FIG. 10. (Color online) Optical response of glycerol/FLC mixture at room temperature in 3 μm memory cell at 20 V at (a) 200 mHz (Ch 1: 10 V/div, Ch 2: 0.020 V/div, time: 1 s), (b) 1 Hz (Ch 1: 10 V/div, Ch 2: 0.020 V/div, time: 200 ms), (c) 10 Hz (Ch 1: 10 V/div, Ch 2: 0.020 V/div, time: 20 ms), and (d) 50 Hz (Ch 1: 10 V/div, Ch 2: 0.020 V/div, time: 5 ms). Ch 1 shows driving time delayed square voltage and Ch 2 shows its optical response. The time scale per division is on *x*-axis

state before any bias application to the cell. A complete switched state [Fig. 11(b)] was achieved when 15 V bias was applied to the sample cell. Figure 11(c) shows that the sample cell has switched back immediately to the original scattering state and the bias was removed unlike the glycerol/DHFLC mixture where switched (memory) state was retained for a prolonged period (Fig. 7). The last scattering state [Fig. 11(d)] has been obtained after around 5 min after the removal of bias. Hence, there is no memory in glycerol/FLC mixture while as there was memory effect in glycerol/DHFLC mixture because the pitch value of the DHFLC material is in submicron range and hence the bistability (or memory) phenomenon is not like the conventional FLC material where it is due to surface stabilization effect. The memory effect or bistability in DHFLC is entirely a different concept related to helix deformation and molecular reorientation process.

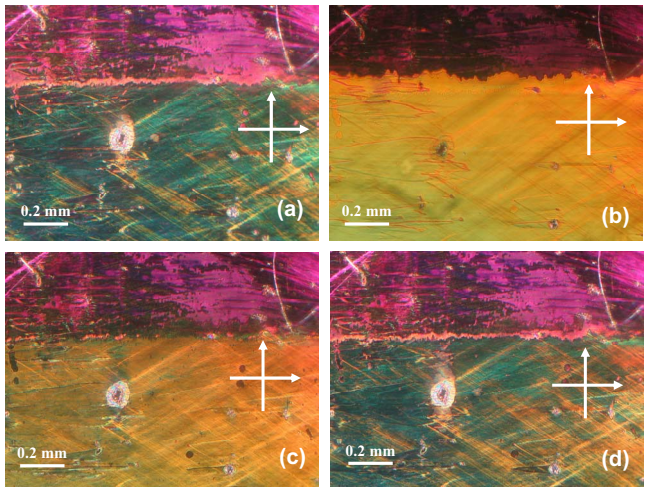

FIG. 11. (Color online) Optical micrographs of 3 μm glycerol/FLC mixture memory cell at room temperature at (a) 0 V, (b) 15 V bias, (c) after 5 min of removal of bias, and (d) again at 0 V. Scale bar: 0.2 mm. The cross arrows have been shown to indicate crossed polarizers.

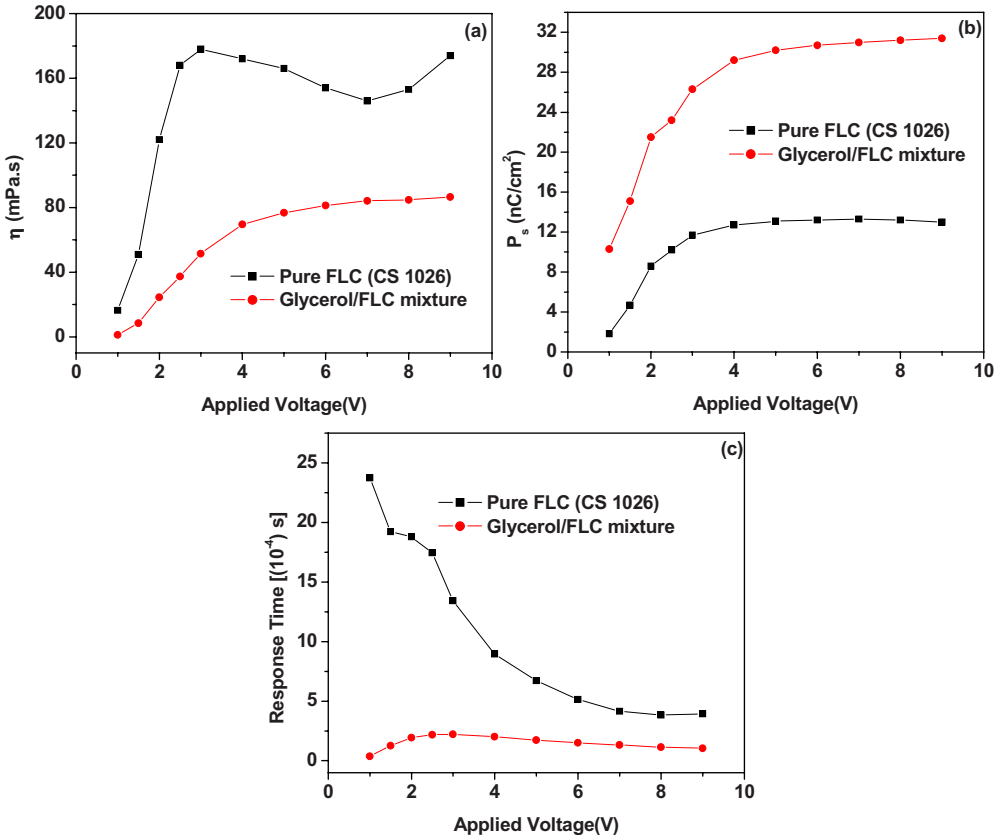

FIG. 12. (Color online) Behavior of (a) rotational viscosity, (b) spontaneous polarization, and (c) response time with applied voltage for glycerol/FLC mixture at room temperature.

E. Material constants in glycerol/FLC mixture

The material parameters such as rotational viscosity, spontaneous polarization, and response time have been studied in glycerol/FLC mixture and compared with pure FLC material. Figure 12 shows the rotational viscosity, spontaneous polarization, and response time, respectively, as a function of applied voltage in the pure and glycerol/FLC mixture. The behavior of material constants has been found the same as it was observed in the case of the glycerol/DHFLC mixture. The rotational viscosity decreases, the spontaneous polarization increases, and the response time decreases in accordance with the Eq. (1).

IV. CONCLUSIONS

The memory effect based on glycerol, a very common and versatile solvent, mixed with DHFLC has been proposed. The observed memory effect has also been compared with the glycerol mixed with FLC. It has been observed that the glycerol not only helps to improve the memory effect in glycerol/DHFLC mixture but also alters the physical parameters such as rotational viscosity, spontaneous polarization, response time, etc. too. The glycerol/DHFLC mixture based memory devices would have high potential for low cost, large area, high speed, high-density memory needed for future advance computers and digital electronics. We also hope that the memory devices based on underlying phenomena would have a tremendous impact on the future of information technology and electronic industry. These studies would also be helpful to optimize LC physical parameters for high quality display applications.

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