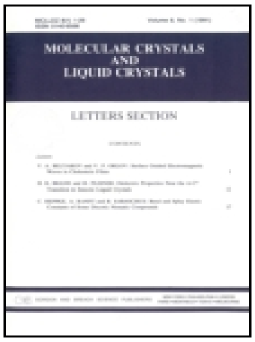

Molecular Crystals and Liquid Crystals

ISSN: 1542-1406 (Print) 1563-5287 (Online) Journal homepage: http://www.tandfonline.com/loi/gmcl20

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To cite this article: Manoj Marik, D. Jana, K. C. Majumder & B. K. Chaudhuri (2015) Dielectric Behavior in B1 and B2 Phases Composed of Unsymmetrical Bent Shaped Liquid Crystal Molecules, Molecular Crystals and Liquid Crystals, 606:1, 111-125, DOI: 10.1080/15421406.2014.915648

To link to this article: http://dx.doi.org/10.1080/15421406.2014.915648

Mol. Cryst. Liq. Cryst., Vol. 606: pp. 111–125, 2015
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ISSN: 1542-1406 print/1563-5287 online
DOI: 10.1080/15421406.2014.915648

Dielectric Behavior in B1 and B2 Phases Composed of Unsymmetrical Bent Shaped Liquid Crystal Molecules

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Detail dielectric measurements were carried out on two different liquid crystalline (LC) samples formed by bent core (BC) molecules, showing B1 and B2 phases in the temperature range 100–150°C. The thermal variation of real part of dielectric permittivity (ε') represents a tiny dip only in the saturated B1 phase which might arising from the dipole orientation disorder due to the inherent phase instability. The experimental data have been used to determine different relaxation parameters. Evidently only one relaxation mode has been detected in the dielectric relaxation spectrum for both phases, which is attributed to the long axis molecular rotation process.

Keywords Activation energy; bent core liquid crystal; B1 and B2 phases; dielectric spectroscopy

Introduction

The states of soft condensed matter are composed of thermo-tropic liquid crystalline (LC) compounds of wide variations in ordering with shape anisotropy [1, 2]. To a great extent, the shape of the mesogens determines the types of LC phases that may be produced. Rod like molecules (“calamatic” compounds) preferably form either nematic or smectic phases which possesses a one dimensional density modulation. Disc like molecules (“discotic” compounds) can also exhibit nematic phases, but they preferably form columnar phases with a two dimensional modulated structure. In 1907 Vorlander proposed his rule that “the LC state is obtained for the most linear of the molecules.” Since chemical construction has been understood to have a most significant effect on the mesophases formed by a LC molecule, it has become clear what type of molecule should be able to form a mesophase. Obviously a molecule having a linear structure seems to have an advantage in formation of mesophases. If the shape of the molecules significantly deviates from the classical rod or disc like shape new phases as well as sub phases of well identified phase limit might occur.

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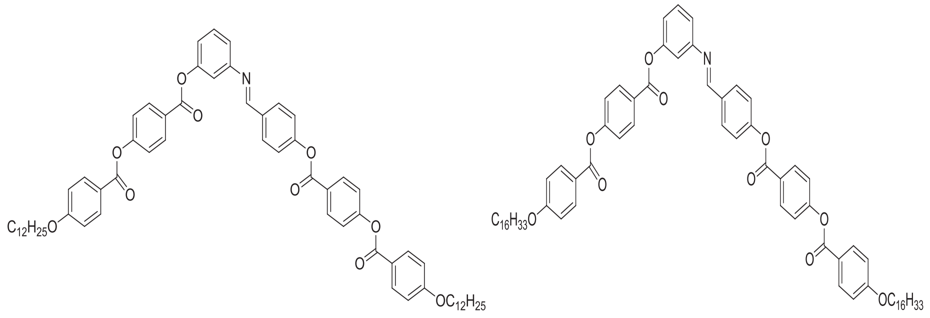
Table 1. Values of different fitting parameters for the two banana phases

Parameters	For B1 phase	For B2 phase
Value of A	0.003	0.654
Value of α_{eff}	0.080	0.036
Value of ν_C at $T = 130°\text{C}$ by Eq. (3)	98.58 kHz	72.50 kHz

Among these, *Y, λ, V, T, U*, *bent* or *banana*-shaped molecules have attracted a great deal of attention till date.

Bent core (BC) mesogens represent a relatively young member in LC families. The steric interactions due to their banana shape might lead to the occurrence of phases with unique ordering like the series of banana phases B1, B2, . . . , B8. The monolayer smectic phases are referred to as B1 phase, the antiferroelectric fluid smectic phase is referred to as B2 phase with the lower temperature phases of the same compound some time referred to as B3 or B4 phase and on the other hand intercalated smectic phase with layer spacing of half the molecular size is referred to as B5–B7 phases in the sequence of their discovery [3–5]. Most of the phases formed by BC mesogens have antiferroelectric interlayer structures [3, 6] except few special cases when the BC mesogens contain chiral centers in the terminal chains in each banana arm [7, 8]. In this consideration it is important to note that the use of chiral molecules is not necessary at all for exhibiting ferroelectricity in LC systems. Ferroelectricity may appear even in systems consisting of achiral molecules, if noncentro-symmetric molecules do not form centrosymmetric arrangements in their systems. For this reason, a great deal of attention has already been directed theoretically and experimentally towards achiral ferroelectric systems [9–11]. Tournilhac et al. first discovered achiral ferroelectric phase in Polyphilic molecules consisting of three or more chemically different units as a result of the separation of their units into homogeneous micro domains [9]. On the other hand, Niori et al. reported ferroelectricity in the SmA$_b$ and SmX1 phase of materials consisting of BC achiral low molecular weight molecules with restricted rotations [12–14].

The BC molecular topology is expected to produce a characteristic effect because of the coexistence of two different unsymmetrical molecular configurations in a single molecule. Apart from usual symmetrically armed BC molecules unsymmetrical BC molecules can



Compound-1 **Compound-2**

Figure 1. Molecular structure of compound-1 (B1-phase) and compound-2 (B2-phase).

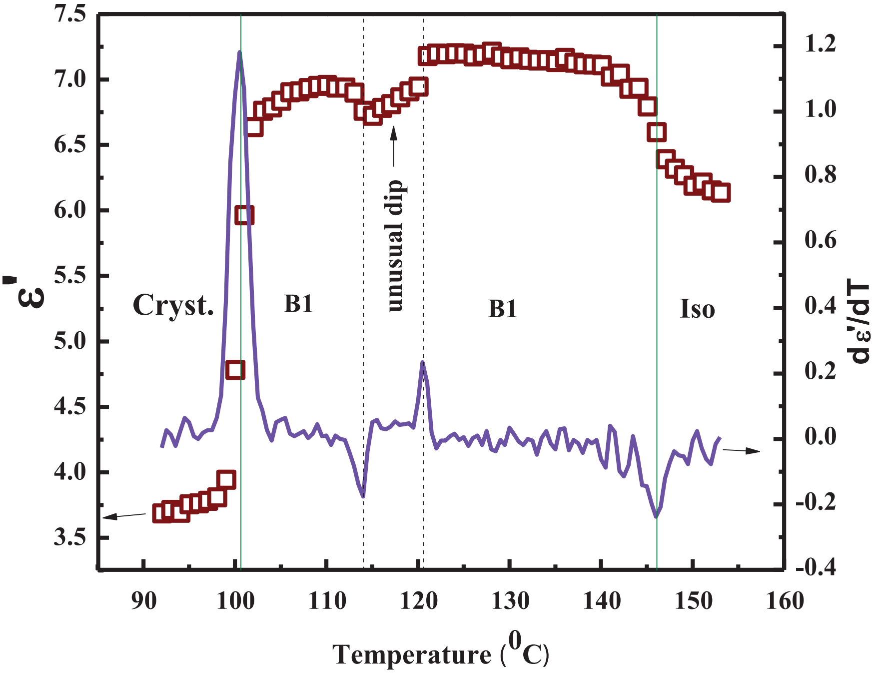


Figure 2. Temperature dependence of real part of dielectric permittivity (ε') and temperature derivative dε'/dT at an unbiased condition for a frequency of 3.162 kHz of a BC LC materials showing B1 phase. The peak of line curve confirms the phase transition temperatures.

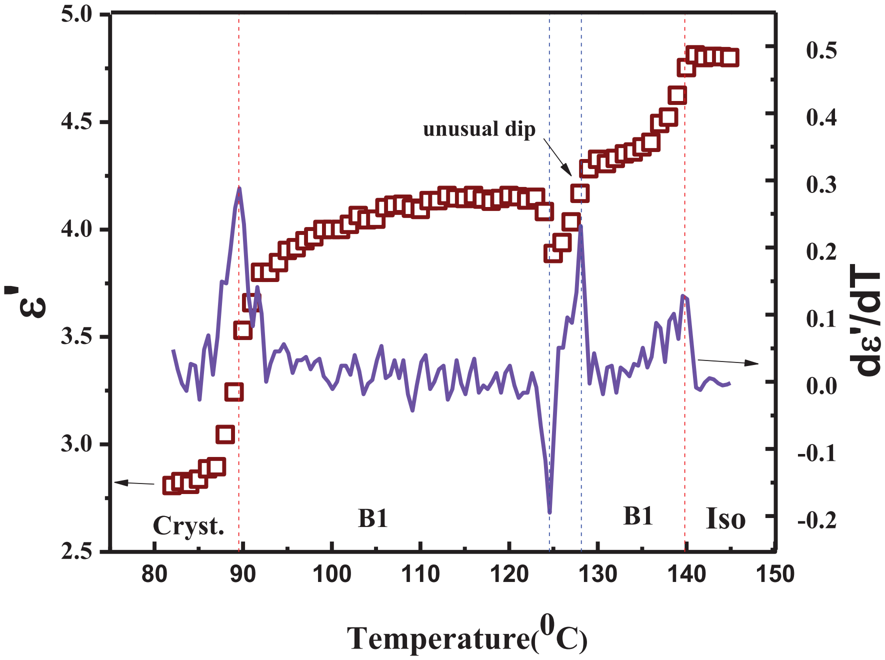


Figure 3. Temperature dependence of real part of dielectric permittivity (ε') and temperature derivative dε'/dT at a dc bias of 8V for a frequency of 3.162 kHz of a BC LC materials showing B1 phase. The peak of line curve confirms the phase transition temperatures.

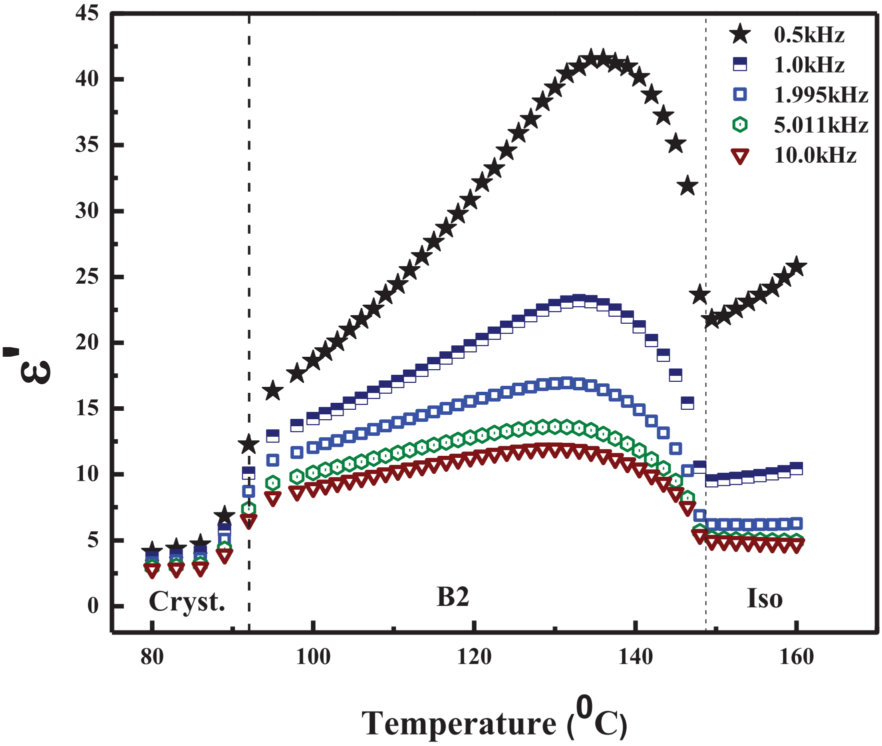


Figure 4. Temperature dependence of the real part of dielectric constant (ε') at different fixed frequencies for a cell of thickness 3.3 μm for BC sample showing B2 phase.

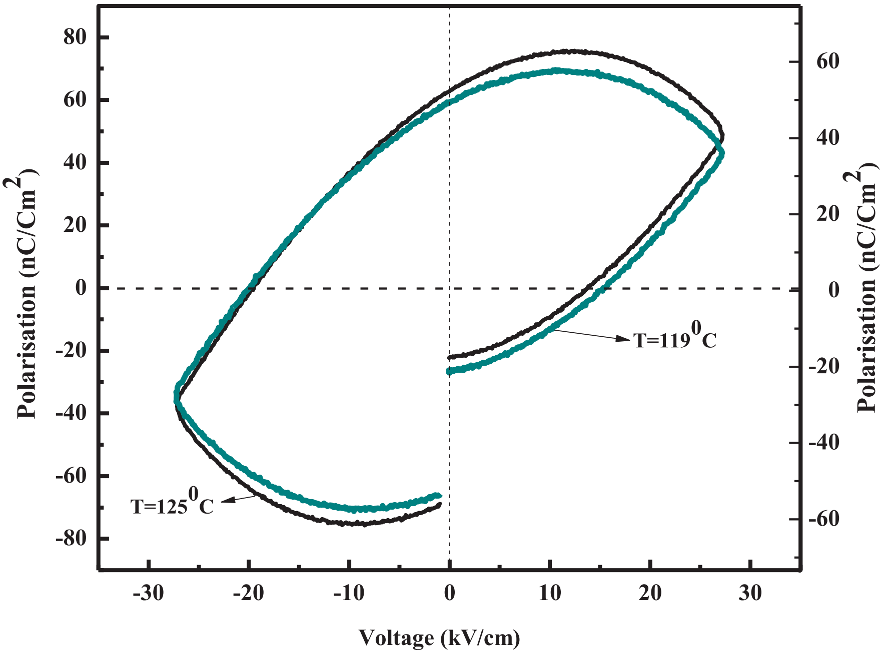


Figure 5. Field dependent hysteresis loop for B1 phase at dip ($T = 119$°C) and B1 phase (125°C).

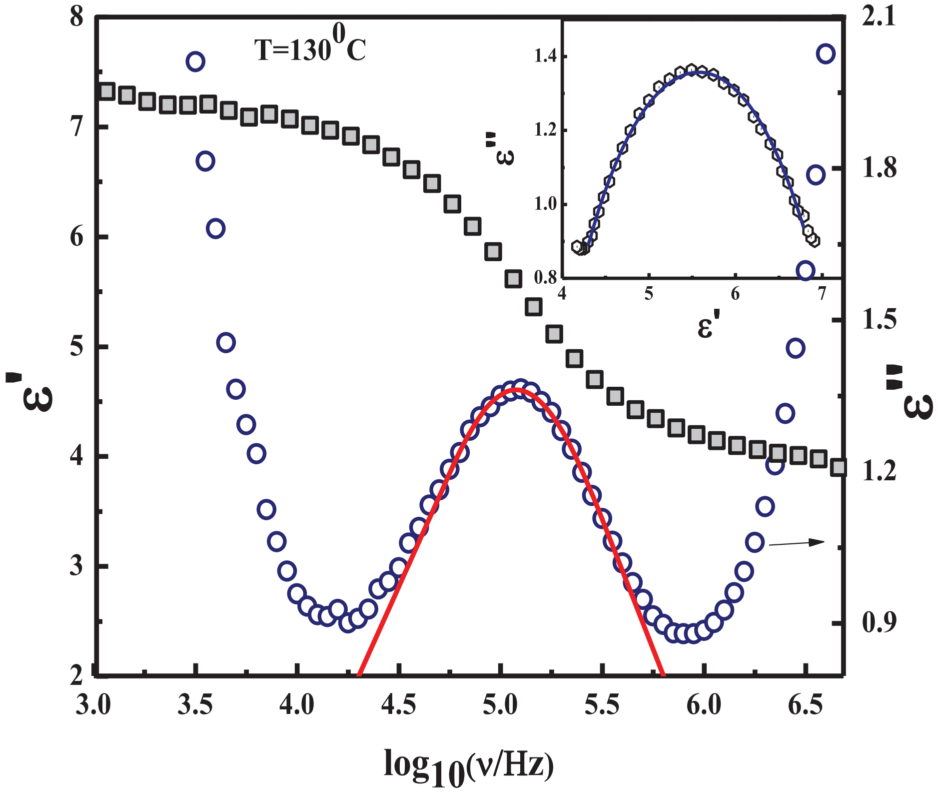


Figure 6. Dielectric dispersion (ε') and absorption (ε'') data for compound-1 in B1 phase for a limited frequency range. Cole-Cole plot for B1 phase at T = 130°C.

reduce the transition temperatures to an extent and possibly link the gap between the BC and rod like molecules. Dielectric investigations are a valuable tool for the characterization of BC molecular mesophases because they give additional information regarding reorientation times of the molecules, short range correlation and the dynamics of polar super structures. The result can further be related to the chemical structure of the molecules if the position of the dipole moment can be systematically changed.

With the above motivations, we would like to explore in detail a comparative dielectric study on two different BC LC samples formed by unsymmetrical six aromatic ring BC molecules of intermediate homologue (C_{12}) showing B1 phase and larger homologue (C_{16}) showing B2 phase. This can be thought of attaching a general aliphatic group (R = $C_nH_{2n+1}O$) to each of the last benzene ring on the two ends of the molecule. Although the structures are fairly rigid, there is often the possibility for internal molecular rotations that can modify the structures and contribute to the dielectric spectrum. It is inconceivable that it would be possible to separate these relaxations experimentally, though the theory shows the power of dielectric background. Hence to find some related important dielectric parameters to trace any intra-phase fluctuations, if exist and to continue with Arrhenius mechanism to show the thermal treatment of activation energy (E_A) with both B phases.

Experiment

Two homologous samples synthesized by Majumdar and coworkers [15] were thoroughly investigated. Both the samples have been synthesized from the same aromatic core

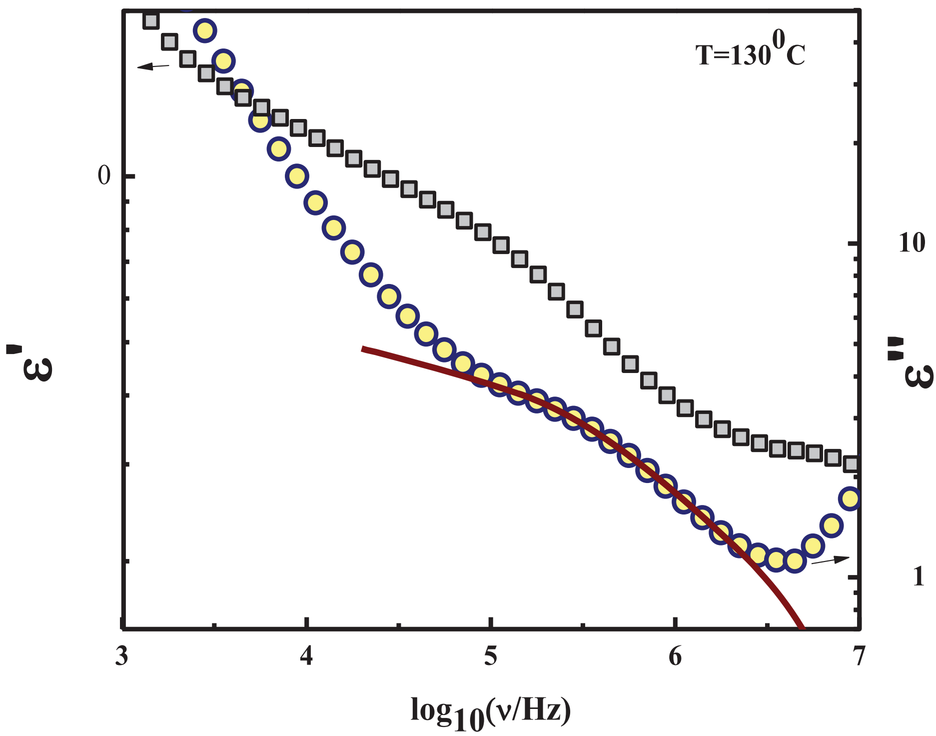


Figure 7. Dielectric dispersion (ε') and absorption (ε'') data for compound-2 in B2 phase for a limited frequency range.

with different chain length as shown in Fig. 1 and phase sequences (on cooling) as: ***compound-1***[(E)-4-((3-((4-((4-((4-(dodecyloxy)benzoyl)oxy)benzoyl)oxy)benzylidene) amino)phenoxy)carbonyl)phenyl 4-(dodecyloxy)benzoate] [***I 150°C B1 97°C crystal phase***], ***compound-2*** [(E)-4-((3-((4-((4-((4-(hexadecyloxy)benzoyl)oxy)benzoyl)oxy)benzylidene)amino)phenoxy)carbonyl)phenyl 4-(hexadecyloxy)benzoate] [***I 149°C B2 92°C crystal phase***].

The sandwiched type cells of thickness 3.6 μm (filled with compound 1) and 3.3 μm (filled with compound 2) with active area ~12 (4 × 3) mm^2 were prepared by indium-tin-oxide (ITO) coated glass plates (resistance ~50Ω cm^{-2}) following the process discussed in our previous work [16, 17]. Polyvinyl alcohol (PVA) was spin coated on the two glass substrates and baked at 140°C for 2 hr. Homogeneously aligned cells are obtained by rubbing two substrates with soft cloth in one direction. Mica spacers have been used to maintain uniform cell gap. First the empty cell was calibrated by using air and toluene as standard references. The cells were filled with investigating compounds in its isotropic phase by capillary action. To get better alignment of the molecules, the cell was subjected to slow cooling process. Temperature of the measuring sample cell was controlled by using Mettler FP82 hot stage along with Mettler FP90 controller with an accuracy of ±0.1°C. A computer controlled HP4192A impedance analyzer (frequency range 10 Hz–13 MHz) was used for dielectric measurements. The capacitance of the empty cell was found to be constant with increasing temperature. The loss exhibited by the empty cell was found to exhibit no peak in the above mentioned frequency range. This firmly indicates that neither the conductive coating of the cell plates nor any trace level impurities in the cell are exhibiting any relaxations in this frequency range.

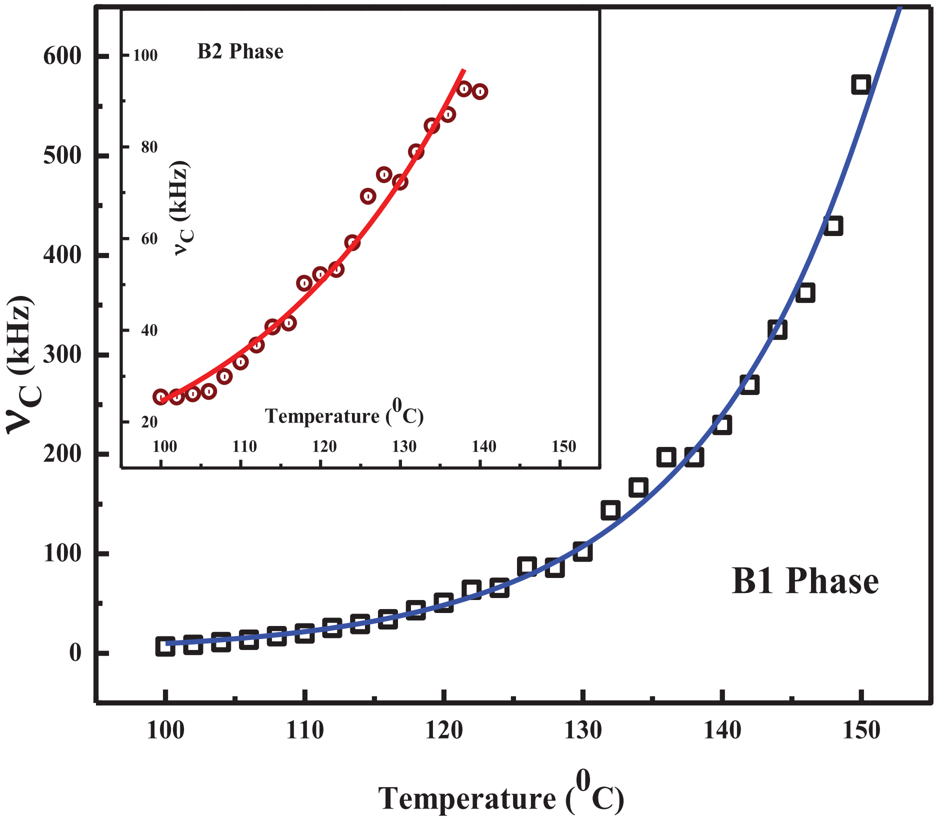


Figure 8. Variation of relaxation frequency (ν_C) with temperature for the single relaxation mode in B1 phase. The inset shows the same variation in the B2 phase. The solid line is the fitting to Eq. (3) in both the phase and the fitted values of $\alpha_{\text{eff}} = 0.08/^\circ\text{C}$ in B1 phase and $\alpha_{\text{eff}} = 0.036/^\circ\text{C}$ in B2.

Result and Discussions

The experimental dielectric data have been analyzed using Cole–Cole modified Debye theory [18]. According to this model, the angular frequency $(\omega) = 2\pi f$, (f is the linear frequency), dielectric strength $(\Delta\varepsilon) = \varepsilon_s - \varepsilon_\infty$ (ε_s is the static dielectric permittivity and ε_∞ is the high frequency dielectric permittivity), the distribution parameter (α) and the relaxation time $(\tau) = 1/2\pi f_r$ (where f_r is the relaxation peak frequency) follow the dispersion equation viz.

$$\varepsilon^* - \varepsilon_\infty = \frac{\Delta\varepsilon}{1 + (i\omega\tau)^{(1-\alpha)}} \tag{1}$$

where complex dielectric permittivity (ε^*) can be expressed as

$$\varepsilon^*(\omega, T) = \varepsilon'(\omega, T) - i\varepsilon''(\omega, T) \tag{2}$$

where $\varepsilon'(\omega, T)$ and $\varepsilon''(\omega, T)$ are the real and imaginary parts of the dielectric permittivity, respectively, and T is the absolute temperature of the medium.

Figures 2 and 3 depict here the thermal dependence (during cooling) of real part of dielectric permittivity $(\varepsilon\prime)$ and its temperature derivative $(\frac{d\varepsilon\prime}{dT})$ at 3.162 kHz for unbiased and

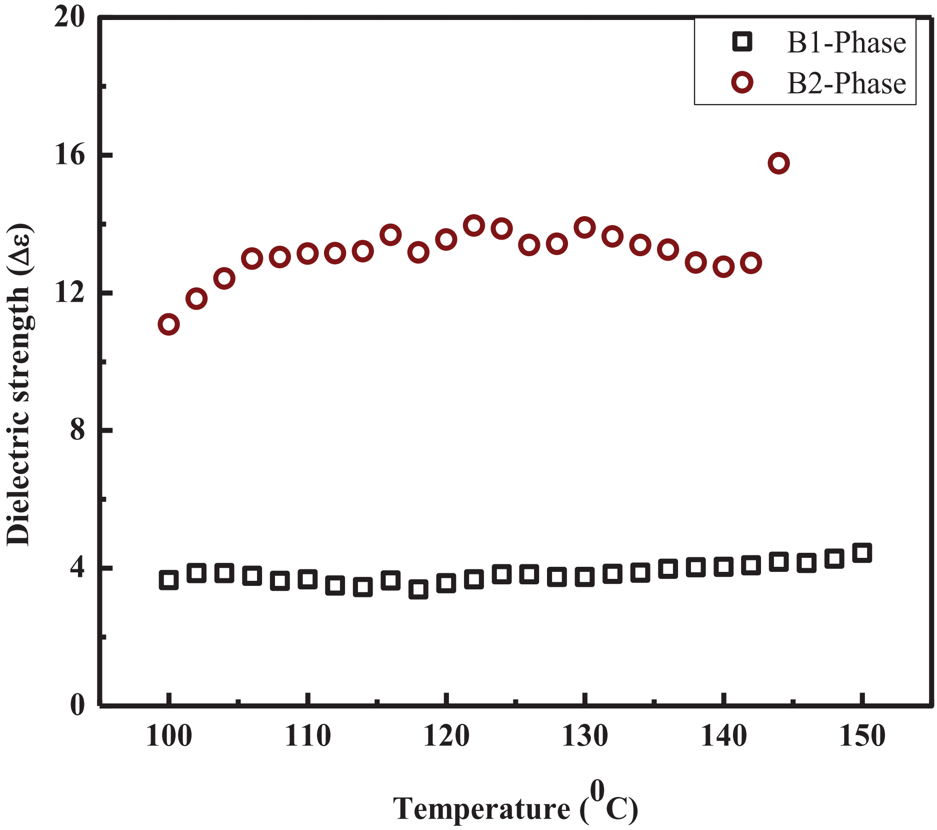


Figure 9. Temperature dependence of dielectric strength of the single mode of both the samples.

also at biased condition (8 V) for isotropic, B1 and crystalline phases with distinct phase transitions. The thermal variations of ε' of both plots show clearly the consequent phase transition temperatures with minor deviations from the earlier report. Due to the immediate effect of applied bias permittivity values changed slightly [15]. However these deviations might be originated as a result of the influences of different cell thicknesses, quality of the used cell, bias and also on other unavoidable state of affairs [17].

In Fig. 4, the thermal activities of ε'in the B2 phase also has been presented for various frequencies (viz. 0.5 kHz, 1 kHz, 2 kHz, 5 kHz, 10 kHz) for a 3.3 μm thick cell. This plot clearly shows the phase transition temperatures for I-B2 and Cryst.-B2 are 148°C and 92°C, respectively, and all these are well within the range of earlier reported values [15] and most interestingly no dip is present here.

Therefore, by considering all these thermal dependences of ε' and $\frac{d\varepsilon'}{dT}$, the important aspect here is the appearance of a tiny dip in the saturated B1 phase only, though both the B1 and B2 phase samples have been prepared from the same aromatic core by adding different aliphatic chains.

In general when the chain length of BC LC molecules are sufficiently long, the molecules have strong biphilicity and there is segregation of the aromatic cores and the aliphatic chains, resulting in a layering order (smectic like) of the B2 phase [12, 19–21]. The intermediate chained molecules show here, the presence of monotropic B1 phase with

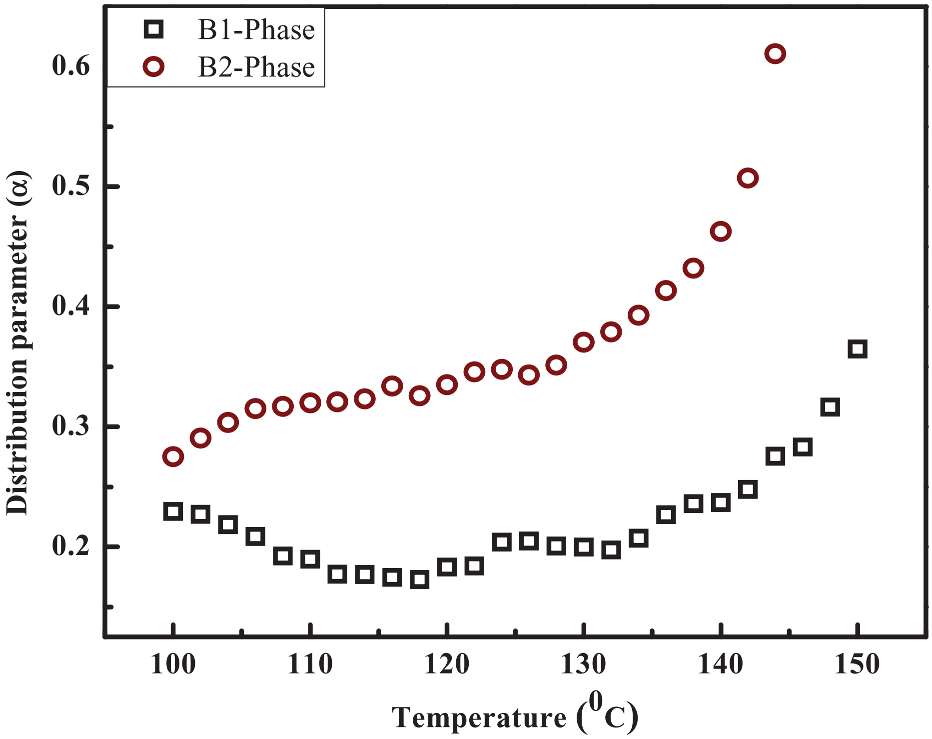


Figure 10. Temperature dependence of distribution parameters of the singly found mode for both the samples.

uniquely appeared tiny dip in thermal variation of ε'. In most BC molecules, the bend angle (between each arm and the bow axis) is larger than 60° and also it is greater than the magic angle of 54.7° which indicate the presence of negative dielectric anisotropy in the system. The BC molecules cannot rotate freely about the long (bow) axes, and pack with an in-layer polar order. The aromatic cores are associated with several dipolar groups having components in the molecular plane that can give rise to a tilting about the arrow axis. This dipolar mechanism is similar to the case of the smectic C LC formed by rod shaped molecules, where the off-axis components of the dipoles can produce tilting of the molecules [21, 22]. On the other hand, larger molecular cross-section of chains compared to the cores in these types of molecules can also produce tilt [21]. Though the individual BC molecules are achiral, as a result of the polar ordering of the molecules and the tilt, the layer becomes chiral [21, 23]. In a typical symmetric V-shaped molecule there is no dipole moment perpendicular to the C_2 axis but there is always a permanent dipole moment present along the C_2 axis. Though the individual molecules are achiral, as a result of the polar ordering of the molecules and the tilt, the layer becomes chiral [23]. Due to this formation of local layer chiral domain in the layered structure we should always expect a low value of spontaneous polarization ($\sim$nC/cm^2) as well as dielectric permittivity in B2 phase. The B1 phase has a somewhat controversial 2D frustrated structure and has generated considerable interest in recent times as it has been shown that under the influence of high electric fields B1–B2 transition may occur and ferroelectric–antiferroelectric (F-AF) like switching may appear [24]. Thus, it can be expected to perceive some more fascinating features of B1 samples over the B2, especially in dielectric environment.

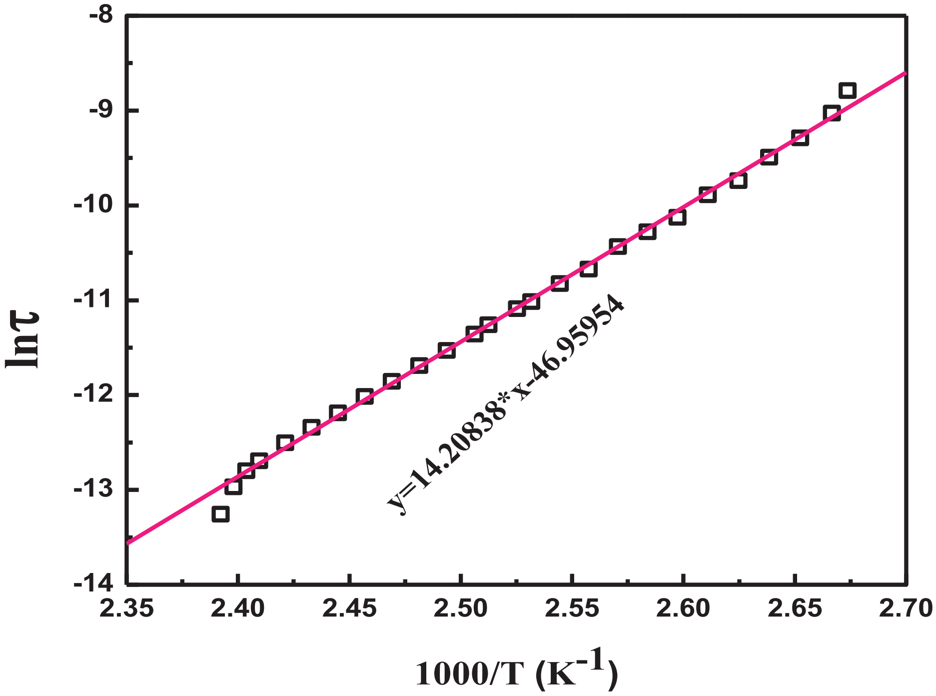


Figure 11. The inverse temperature dependence of natural logarithm of relaxation time (ln τ) in B1 phase at unbiased condition. The solid line shows the fitting with Arrhenius Eq. (4) and give the value of activation energy as $E_A = 118.13$ kJ/mol.

Thus the presently studied unsymmetrical six aromatic ring BC LC molecules draws motivating consideration from the view point of above all aspects. The attachment of $C_{12}H_{25}O$ in the aromatic chain (B1) evidently modifies the capacitance within the specified window of temperature (dip region) while such a behavior is absent in B2 phase by attaching $C_{16}H_{33}O$. The observed behavior of dip in B1 phase can be associated with appropriate variation of dipolar correlation with temperature [4]. The voltage dependence of the hysteresis loop in B1phase also has been measured.

Figure 5 shows bias variation of hysteresis loop in B1 phase ($T = 125$°C) and also in the dip ($T = 119$°C) region. It has been clear that, there is hardly any F-AF like transition occurred in this dip at all. The polarization values increases by small amount for systematic thermal activities of the bent shaped B1 phase molecules. Thus, this small part (dip) appeared in the saturated region of B1 phase solely due to thermal ambiguity. These self generated small sub-phases in the saturated LC phases give always some extra features to LC physics and its technological industries. If one wants to categorize these two LC B-phases from dynamical point of assessment, one should become aware of the two main orientations: the rotation around the molecular long and short axes [20, 25].

From the structure of the BC materials it is evident that the rotation around the short axis is accompanied by very small change of dipole moment. Every molecular ordering gives a constraint to the dynamics which can be seen by dielectric method. A superstructure can be observed by this technique if it causes a linear response to the applied external electric field. In order to study such effects the sample should show low electrical conductivity. To explore the different relaxation modes, we show in Figs. 6 and 7 the frequency dependence

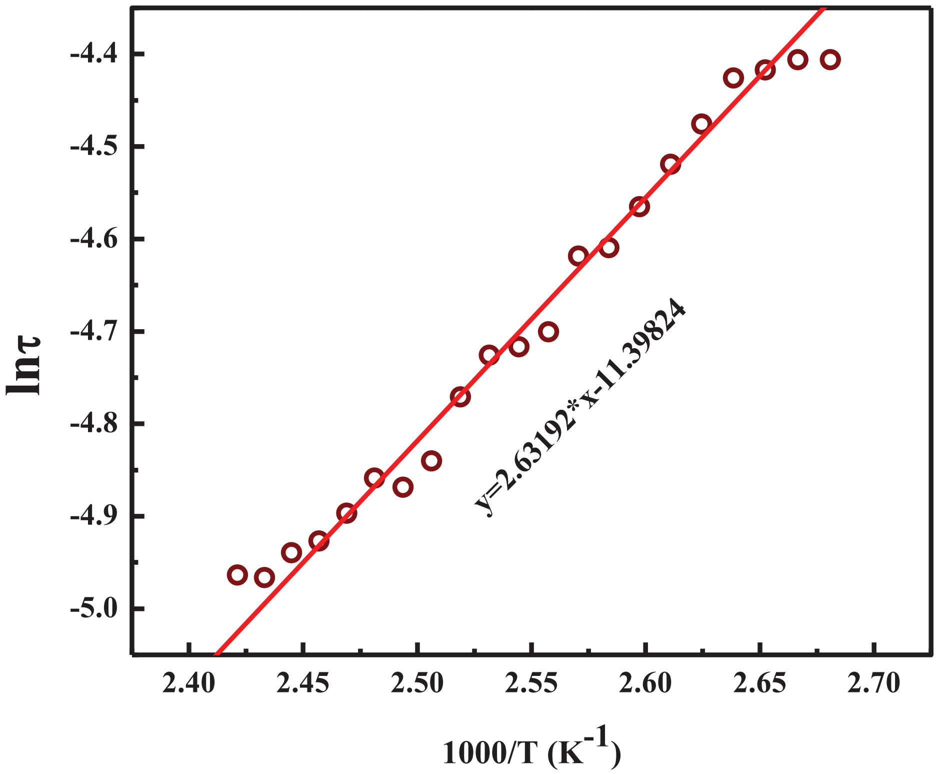


Figure 12. The inverse temperature dependence of natural logarithm of relaxation time (ln τ) in B2 phase. The solid line shows the fitting with Arrhenius Eq. (4) and give the value of activation energy as $E_A = 21.88$ kJ/mol.

of real (ε') and imaginary parts (ε'') of dielectric permittivity of both the compounds at $T =$ 130°C in absence of bias field.

In both the present investigation we cannot detect any low frequency relaxation mode which might be due to the presence of large ionic contributions at low frequency. Also from the above mentioned figures the relaxation modes at few 100 kHz ranges have been found which might be originated due to the rotation of the molecules around the long axis. This observation is in conformation according to Pelzl [26] and Heppke *et al.* [27].

The solid lines here are the Cole–Cole fitted curve to the dielectric data. From these fittings, we obtain here some important parameters which might be helpful to explore other relevant features. The relaxation frequency (ν_C) of the observed single mode has been extracted from the theoretical fit of the Cole–Cole Eq. (1), to the experimental data points of both the samples. The thermal variation of ν_C is presented in Fig. 8. In the present study ν_C is few times higher in B1 than B2 phase. It is also clear from the figure and also from the inset that ν_C increases sharply with temperature and fitted with following equation given by [28]

$$\nu_C(T) = A\exp(\alpha_{eff}T) \tag{3}$$

where ν_C is the observed relaxation frequency in kHz at any arbitrary temperature T, α_{eff} signifies the presence or absence of any fluctuations in the mesophase. The precise outcome here is that, the fluctuation level in both the BC LC phases is minor. In comparison

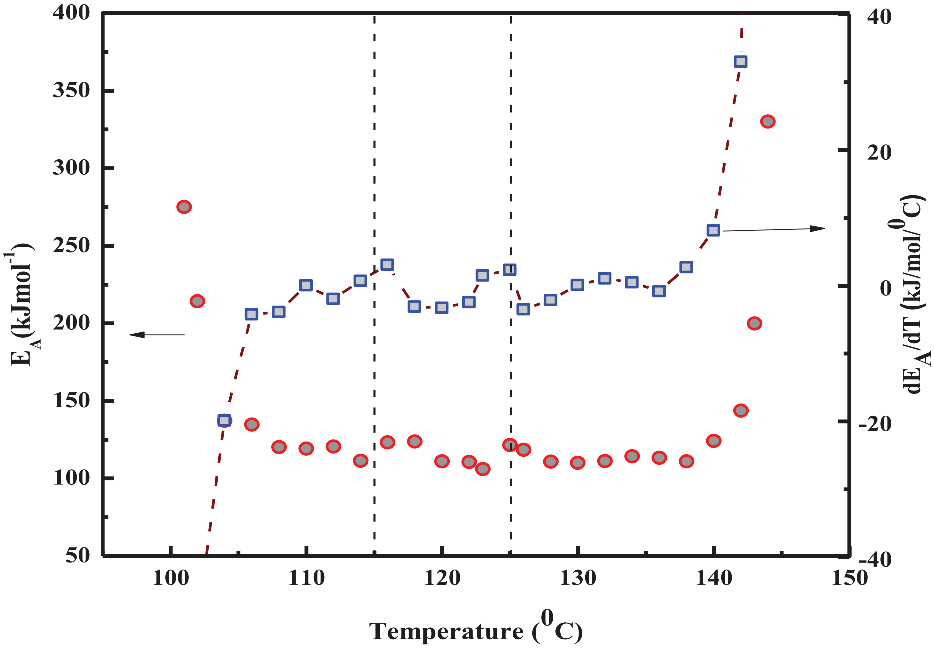


Figure 13. Thermal variation of activation energy (E_A) and its temperature derivative (dE_A/dT) for in B1 phase at unbiased condition.

B1 phase ($\alpha_{eff} = 0.08/°C$) is slightly more fluctuated over B2 phase ($\alpha_{eff} = 0.036/°C$) [29, 30].

The variation of dielectric strength ($\Delta\varepsilon$) with temperature for the two samples is shown in Fig. 9. The increase in $\Delta\varepsilon$ is due to increment in spontaneous polarization (P_S), which indicates that the dipole moment of the molecules plays an important role for the enhancement of dielectric constant [31, 32]. Thermal variation of $\Delta\varepsilon$ here is very minute but the $\Delta\varepsilon$ value of B1 sample is one third that of B2. In this respect P_S of B1 sample is surely lower by a margin than B2. The temperature dependence of distribution parameter (α) of the two samples is shown in Fig. 10.

The values of the distribution parameters of both the samples are almost constant initially up to 135°C and above 135°C, it increases sharply towards I-B1 and I-B2 transition points. The value of α increase significantly near I-B1 or I-B2 transition clearly indicates the presence of more than one relaxation modes near such a phase transition temperature. The lower value of α in the B1 phase naturally signifies that the obtained mode of relaxation is dominating over other modes, if any. The mode of B2 phase here is less dominating in comparison to B1 following the same analogy.

The existence of a tiny dip in the saturated B1 phase here composes the result interesting in large scale and motivates us to explore in detail the thermal variation of activation energy (E_A) in the B1 phase of the investigating compound. In LC devices the relaxation time (τ) is of extreme importance, which further depends on cell gap, degree of molecular alignment and many other external and internal parameters. Besides, the application of high voltage can effectively change τ and all these related properties on which E_A depends on. Note that E_A plays a crucial role in many device applications. Therefore, E_A can be calculated using the following Arrhenius relation [33]

$$\ln \tau(T) = \ln \tau_A + \frac{E_A}{RT} \tag{4}$$

where the dependence $\ln \tau(T)$ on T^{-1} is linear, τ is relaxation time τ_A is pre-exponential factor, R is the universal gas constant and T is the absolute temperature. Figures 11 and 12 show the variation of natural logarithm of relaxation time [$\ln(\tau)$] with inverse temperature in B1 phase as well as in B2 phases separately. This plot clearly manifests the ever expected linear thermal dependence. The experimental data points are fitted to Eq. (4) also to extract the value of E_A separately in B1 and B2 phases. We obtain E_A in the B1 phase for unbiased condition as 118.13 kJ/mol, and value of E_A in B2 phase is 21.88 kJ/mol.

Here, the obtained values of E_A are very closer to the previously reported values for other banana phases [34, 35]. As a result from Arrhenius formula, the derivative $\partial \ln \tau(T) / \partial T^{-1}$gives directly value of E_A, at a given temperature. Thus, one can analyze the thermal behavior of E_A [29, 36]. Generally, it is assumed that E_A should be temperature independent. However, there is no physical reason to assume constant E_A for LC material, as the basic physical properties, the number density, etc. are all temperature dependent.

Figure 13 manifests an unusual comparative thermal behavior of E_A and its temperature derivative (dE_A/dT) in B1 phase. In the earlier half of this manuscript we had reported the existence of an unexpected tiny dip in the middle range of saturated B1 phase. With the help of Eq. (3) we had also established that a minute additional fluctuation did exist in B1 phase over B2 phase. Thus Fig. 13 might be useful to give some kind of support to the observation of tiny dip in B1 phase. More regular thermal behavior has been depicted for B1 phase. But there is a little bit of irregularity accompanied in the range 115–125°C in B1 phase which we can easily understand from Fig. 13. Coincidentally the newly found dip exists there for B1 phase. So, one can easily match the two in formations to support strongly each other.

Conclusion

In the present study, we have determined the dielectric properties of two BC liquid crystal materials as a function of temperature and frequency.

1. Thermal analysis of $\varepsilon'$and $\frac{d\varepsilon'}{dT}$ in the B1 phase for both in biased and unbiased conditions gives a tiny dip with confined well boundary and it is of special importance.
2. Relaxation frequency (ν_C) of both the phases depends on temperature and ν_C was fitted with Eq. (3) in both cases. It is evident from the value of α_eff that a minute excess fluctuation exists in B1 phase over B2 phase, which might be due to incipient structural differences.
3. The dielectric strength of both the samples is almost temperature independent. The dielectric strength of sample-1 is less than sample-2 due to its lower spontaneous polarization value.
4. The value of distribution parameter (α) was found to be lower than 0.5 everywhere throughout the B1 phase. The value of α in B2 phase exceeds the value 0.5 only at the transition point. So from these two behaviors it is evident that both the samples follow the Cole–Cole type of relaxation mechanism with one relaxation mode.
5. The almost temperature independent thermal behavior of activation energy in B1 phase signifies a small region with up rights to show some support in evidence of the tiny dip.

Acknowledgments

The authors are grateful to the Council of Scientific and Industrial Research (CSIR), Government of India, for partial financial grant. One of the authors (BKC) is grateful to the Alexander von Humboldt (AvH) Foundation for providing the LCR Meter. Author KCM is thankful to UGC New Delhi for an Emeritus Fellowship. Author (MM) is grateful to The Indian Association for The Cultivation of Science (IACS, Kolkata) for providing the experimental facilities. We thank the anonymous reviewer for critical comments and suggestions to improve the quality of the work.

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