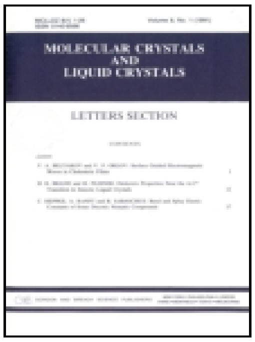

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Optical and dielectric properties of an isothiocyanato liquid crystal compound and comparison of its behavior with that of a homologous member

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ABSTRACT

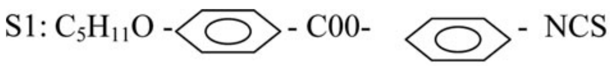
The properties of an isothiocyanato liquid crystal compound (code name S1) has been studied with a view to compare its behavior with that of another member of its homologous series (code name S5). Optical properties, such as refractive index, polarisability, and their anisotropies, have been evaluated with varying temperature and the orientational order experimentally determined from the study of polarisability anisotropy and compared with theoretically computed values. Dielectric studies have been conducted to determine not only the dielectric anisotropy but also the effective molecular dipole moment and its temperature dependence. The temperature variation of the angle of inclination (β) of molecular dipole moment with the director direction has also been determined and compared with that of S5. In order to determine the nature of molecular association in the mesophase, the molecular correlation factor (g) has been estimated from the measurements of dipole moments of compound in solution and compared with the values obtained for S5.

KEYWORDS

Birefringence correlation factor; dielectric anisotropy; dipole moment; order parameter; polarisability anisotropy; refractive index

1. Introduction

The mesogenic compound S1 is a member of the homologous series 4-isothiocyanatophenyl–4′-n-alkyloxybenzoate [1] with $n = 5$, and has been synthesised by Czuprynski [2,3]. Preliminary X-ray work and related investigations on some members of the series have been reported [1–5]. Studies similar to the present work on optical and dielectric properties have been carried out on the homologous member S5 (having $n = 10$) [4–6]. The ultrasonic stability of some liquid crystals containing cyano and isothicyanato terminal groups have been studied [7]. Work has also been done on fluoro-isothiocyanated mesogens [8]. Optical properties of isothiocyanato fluorinated mixture are also determined [9]. However, no systematic optical and dielectric studies have been reported to date on S1.The compound S1 has the following structural formula and is known to undergo the following phase transition temperatures:



Cr 63.8 °C Sm A1 72.1 °C N 83.5 °C Iso

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Comparison of the properties of the above compound is made with that of S5, which has the following structural formula and exhibits the following phase transition temperatures:

S5:

Cr 77 °C Sm A 97 °C Iso

It is evident from the above phase transition temperatures that the nematic phase in S1 disappears in the higher member S5, which exhibits only the SmA phase. However, in S1 the phases exist within a narrow range of temperature contrary to the case of S5, which exhibits the SmA phase over a wide span of ∼20°C.

The present work focuses on the optical and dielectric properties of S1 and draws comparison of these properties with those of S5.

2. Experimental

2.1. *Optical microscopy*

Routine optical microscopic studies were conducted to confirm phase transition temperatures. The sample was introduced into a hot stage (Mettler FP82 HT) and the temperature was raised at a rate of 1°C min^{-1} during heating. Optical microscopy studies confirmed the phase transition temperatures of the Cr→SmA1 and SmA1 to N phases with an accuracy of 0.5°C. However the nematic to isotropic transition was observed to occur at 83.5°C,and the repeated observations confirmed the above findings.

2.2. *Birefringence studies*

Birefringence studies were conducted using a He-Ne source (wave length $\lambda = 633$ nm) on the basis of the Chatelain–Wedge principle. Each sample was introduced into a glass prism of angle between 1° and 2°. The prisms were formed with glass slides whose inner surfaces were treated with polyvinyl alcohol for planar surface alignment. The liquid crystal sample was introduced into the prism and sealed. The prism encapsulated in a sample holder, whose temperature was regulated up to ±1°C with the help of a temperature controller, was placed in an aligning magnetic field of 8 K Gauss such that the direction of rubbing (along the prism edge) is along the magnetic field. The laser beam from the source was made incident on the sample through a hole in the sample holder, and the emerging beams (ordinary and extraordinary) were projected on a screen held several metres (4.5 m) away. The sample was taken through a number of temperature cycles in the presence of a magnetic field to ensure an aligned monodomain sample. The sample was heated at a rate of 1°C m^{-1} to temperatures beyond its isotropic temperature and was allowed to cool at the same average rate. From the changes in the patterns observed on the screen, the transition temperatures could be confirmed. From angular deflection measurements on the screen, the refractive indices n_e and n_0 of extraordinary and ordinary rays could be determined with the knowledge of prism angle. The prism angle was determined prior to the introduction of sample by measuring the angular deviations of the laser beam reflected from the front and back surfaces of prism. Measurements of both top and bottom ends of each of the circular spots (∼0.6 cm in diameter) were taken, and the mean value of these was used for calculation.

To obtain polarizabilities α_e and α_o, and thereby the order parameter from the birefringence studies, the densities have to be determined as a function of temperature. The densities of sample at various temperatures were obtained by introducing weighed sample in molten form in a dilatometer, which was then placed in a heat bath. The length of sample column in dilatometer tube was measured with a traveling microscope at temperature intervals of 1°C, and the density of sample was calculated. The polarizabilities were determined using the Vuks formula [10], namely

$$(n_{\Upsilon}^2 - 1)/(n^2 + 2) = (4\Pi)/3\, N\alpha_{\Upsilon},$$

where n_{Υ} is the refractive index, $\Upsilon = \mathrm{e}$ or o, $n = n_{\mathrm{avg}} = [n^2{}_{\mathrm{e}} + 2n^2{}_{\mathrm{o}}]/3$; N is the number of molecules per cubic centimeter and is given by $N = \mathrm{N_A} d/M$, where $\mathrm{N_A}$ is the Avogadro number, d is the density of the sample, and M the molecular weight. The orientational order parameter $<\mathrm{P}_2>$was calculated using the de Gennes expression [11] in the following form:

$$<P_2> \;=\; (\alpha_{\mathrm{e}} - \alpha_{\mathrm{o}})/(\alpha_{\mathrm{II}} - \alpha_{\perp}),$$

where $(\alpha_{\mathrm{II}} - \alpha_{\perp})$ was determined by adopting the Haller's extrapolation [12] procedure and by plotting In $(\alpha_{\mathrm{e}} - \alpha_{\mathrm{o}})$ versus In $(T_{\mathrm{C}} - T)$ and extrapolating the straight line to obtain $\mathrm{T} = 0$, that is to In T_{C} to obtain $(\alpha_{\mathrm{II}} - \alpha_{\perp})$ from the intercept.

2.3. *Dielectric study*

Dielectric studies were conducted using indium tin oxide (ITO)-coated glass plates separated with thin cover slips on three sides to form a sample cell (capacitor).Typical dimension of a sample cell is 4.0 × 2.5 × 0.05 cm^3 and has a capacitor value of ~21 pf in air. The cell was calibrated at different operating frequencies (10 and 100 KHz) using standard liquids (benzene and p-xyline) with the help of an LCR meter (6471 Fobes Tinsely). The sample was introduced into the cell as an isotropic liquid through the open end of capacitor cell, which was subsequently sealed. The sample was encapsulated in a sample holder whose temperature may be regulated with an accuracy of ±1°C and heated up to 150°C. The sample was taken through a number of temperature cycles in the presence of an aligning magnetic field (~8 KGauss) to obtain an aligned monodomain sample. Readings were taken at intervals of 2°C to a temperature beyond the nematic–isotropic temperature for the parallel and perpendicular components of dielectric permittivities from which the capacitor values was evaluated by a standard procedure of calibration.The bridge voltage across the sample was maintained sufficiently low (~0.3 V) so as not to produce any electric field-induced instability. The effect of electrode polarization or any other parasitic phenomena is cancelled out during the process of standardization of cell using standard liquids.

2.4. *Determination of isolated molecular dipole moment*

Solutions of the compound of different concentrations were prepared in a non-polar solvent (p-xylene). Each solution was introduced into sample cell, and dielectric studies were performed at 10 KHz to obtain dielectric permittivities ε_{12} of solutions at a particular temperature of 80°C. The dielectric permittivity ε_1 of the solvent was determined at the same temperature.

Birefringence studies of solutions of different concentrations were performed at the same temperature (80°C). Solutions of different concentrations (by %wt) were introduced in a glass prism (angle: $1^\circ \sim 2^\circ$), which was inserted in a sample holder, whose temperature was regulated with an accuracy of ±1°C. Details of the experimental procedure are given in Section 2.2.

The required equation for computing molecular dipole moment in solution μ_{sol} is

$$\mu_{\mathrm{sol}}^{2} = \frac{27KT[(\varepsilon_{12} - n_{12}^{2}) - (\varepsilon_{1} - n_{1}^{2})]}{4\Pi \mathrm{N}(\varepsilon_{1} + 2)(n_{1}^{2} + 2)c,}$$

where suffices 1 and 12 refer to solvent and solution parameters respectively, c is the concentration (mol cc^{-1}) at temperature T (K), and N and K are Avogadro number and Boltzmann constant respectively.

The dipole moment μ_{iso} of the isolated molecule was obtained by extrapolating the curve of μ_{sol} versus concentration (c) to infinite dilution. The values obtained in esu cm may be converted to a Coulomb-meter or Debye using a factor of 3.3356×10^{-12}.

3. Results and discussion

3.1. *Optical studies*

Variations in ordinary and extraordinary refractive indices, n_{o} and n_{e} with temperature for S1 are depicted in Fig. 1(a).The average refractive index $n_{\mathrm{avg}} = [n^{2}{}_{\mathrm{e}} + 2n^{2}{}_{\mathrm{o}}]/3$ shows an increase from SmA to nematic phase and also a slight but perceptible discontinuity from nematic to

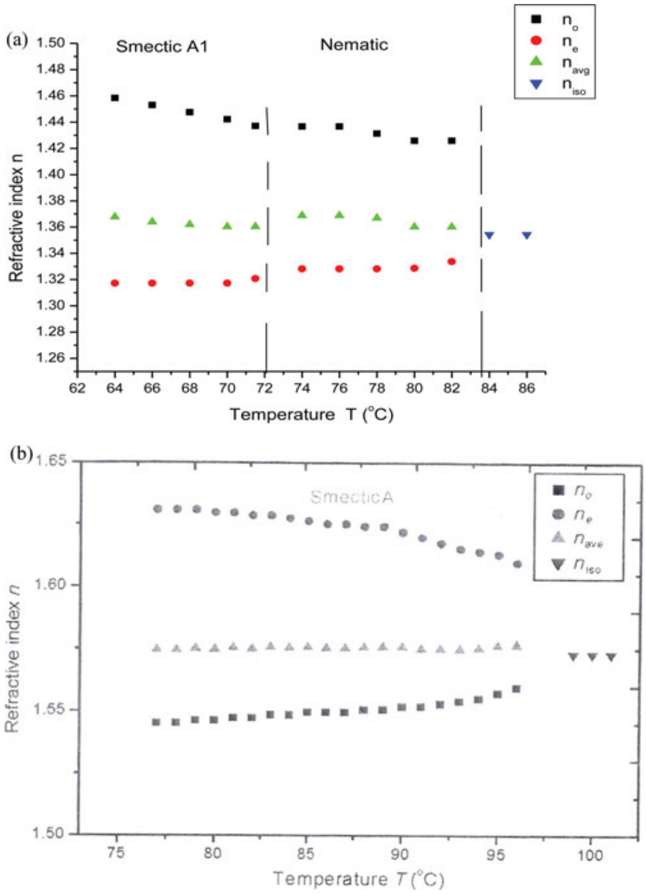

Figure 1. (a) Variation of refractive indices n_{e}, n_{o}, n_{avg}, and n_{iso} with temperature T of S1. (b) Variation of refractive indices n_{e}, n_{o}, n_{avg}, and n_{iso} with the temperature of S5.

isotropic transition. Variations in the refractive indices of S5 have been reproduced here (Fig. 1(b)) from [6] for easy reference and show that the average refractive index is almost constant throughout the SmA range and continuous with respective n_{iso} values at T_{iso} (Fig. 1(b)). This behavior is generally attributed to non-polar compounds for nematic–isotropic transitions [13] whereas the discrepancy between n_{avg} and n_{iso} values at nematic–isotropic transition temperature is suggested to be exhibited by the molecules of higher molecular dipole moments [14].

The discrepancy between n_{avg} and n_{iso} values at nematic–isotropic transition is suggested to be exhibited by the molecules of higher molecular dipole moments [14]. However, the same possibly may not be inferred for SmA-1 transition as it is expected that S5 having a terminal NCS will have a strong molecular dipole moment. It is found that n_{avg} at 80°C (N phase) is 1.365 for S1 and 1.58 for S5 at the same temperature (SmA phase), and Δn values of S1 at 70°C (SmA phase) and 80°C (nematic phase) are 0.125 and 0.097 respectively. On comparing with the Δn value of 0.0834 (80°C, SmA phase) for S5, we note that Δn for S5 is slightly less than that of S1 at the same temperature.

Figure 2(a) depicts the thermal variation of polarisabilities α_{e} and α_{o}. A slight discontinuity in polarisability is observed at the nematic to isotropic phase transition for S1. However,

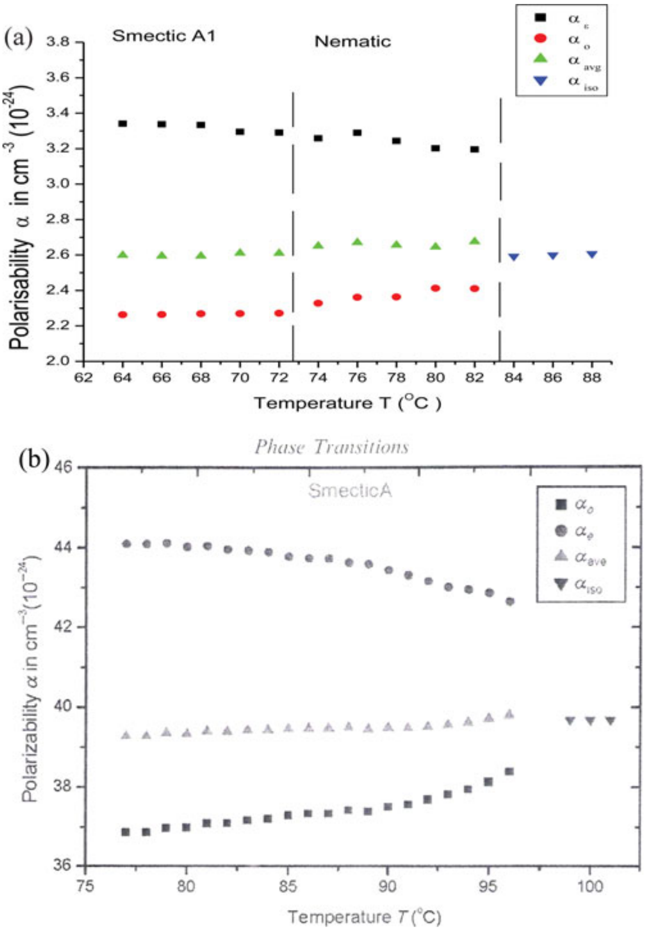

Figure 2. (a) Variation of polarisability α_{e}, α_{o}, α_{avg}, and α_{iso} with temperature T of S1. (b) Variation of polarisability α_{e}, α_{o}, α_{avg}, and α_{iso} with temperature T of S5.

for S5 the average polarisability $\alpha_{\text{ave}} = (\alpha_e + 2\alpha_e)/3$ is constant throughout the SmA range and continuous with the respective α_{iso} value at T_{iso} as shown in Fig. 2(b). The average polarisability $\alpha_{\text{ave}} = 26.42 \times 10^{-24}$ cm^{-3} for S1 (nematic phase, 80°C) whereas the value for S5 is much higher, i.e. 39.33×10^{-24} cm^{-3} (SmA phase, 80°C).

Figure 3(a) shows the variation of the order parameter of S1 with temperature. Experimental values of orientational order parameters have been calculated and compared with the theoretical values from the Maier–Saupe [15] and McMillan [16] theories. Although the Maier–Saupe values are relevant to ordering in the nematic phase (orientational ordering), they have been depicted for the entire mesogenic range for S1 to enable comparison of the orientational aspects of ordering in presence/absence of other type of ordering, namely translational. Theoretical values of order parameter have also been calculated on the basis of the McMillan potential [16] for SmA phase, namely

$$V(z,\ \cos\theta) = -v_o[-\alpha\delta\cos(2\Pi z/d) + \{\eta + \sigma\alpha\ \cos(2\Pi z/d)\}P_2(\cos\theta),$$

where α and δ are two adjustable parameters, z is the displacement along the layer normal, d is the layer thickness, $\eta = <\mathrm{P}_2(\cos\theta)>$ is the orientational order parameter, whereas $\tau = <\cos(2\pi \mathrm{z}/\mathrm{d})>$ is the translational order parameter and $\sigma = <P_2(\cos\theta)\cos(2\pi z/d)>$

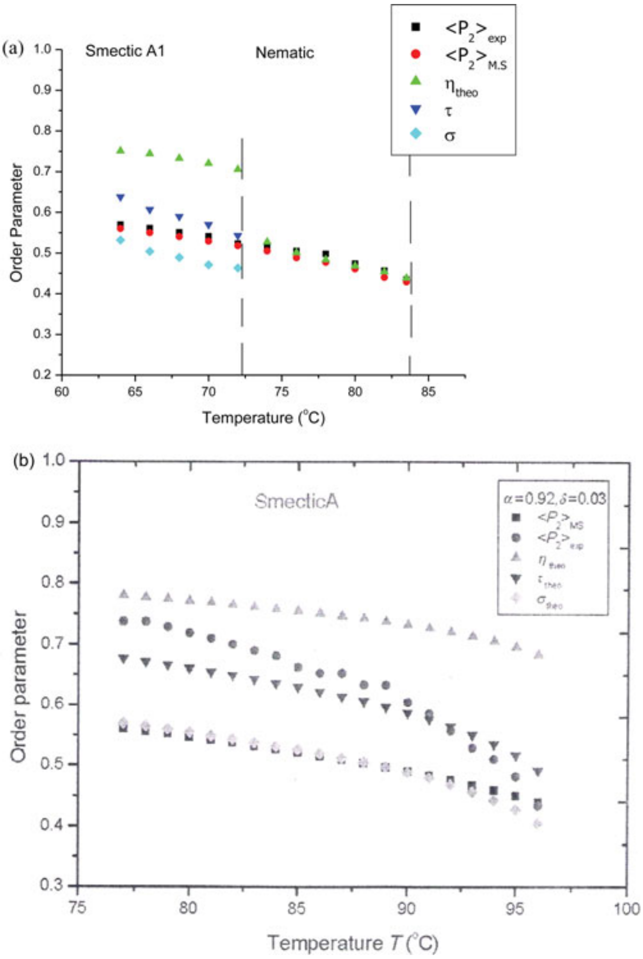

Figure 3. (a) Variation of order parameter with the temperature of S1. (b) Variation of order parameter with the temperature of S5.

is the mixed translational and orientational order parameter; ν_o determines the nematic–isotropic transition temperature and fixes the temperature scale of model. McMillan [16] has shown that the nematic–isotropic transition considering only η and σ at $T_{NI} = 0.2202\ \nu_o$ (given by the Maier–Saupe theory) is valid for $\alpha < 0.98$, and that the SmA–nematic transition temperature increases with α and reaches T_{NI} at $\alpha = 0.98$. For $\alpha > 0.98$, the SmA phase melts directly into the isotropic phase. For the values of α below 0.98, there can be an Sm–N–Iso transition with an Sm–N transition of first order. In case of lower values of α (<0.70), an Sm–N–Iso transition occurs with an Sm–N transition of second order. In case of S1, which has SmA–N–Iso transition, the best reproducibility of transition temperature was obtained with $\alpha = 0.85$ and $\delta = 0.045$. Since this value of α falls below the 0.98 limit, we may expect SmA–N–Iso transition to be of first order, although in the present case not only η and σ but τ too has been considered in the potential. In the case of S5, a higher value of $\alpha = 0.92$ with $\delta = 0.03$ gave the best reproducibility and a reasonable fit to S5 data where there is only SmA–Iso transition [4]. Here also the value of α falls below the 0.98 limit. Variations in the order parameters of S5 with temperature are reproduced in Fig. 3(b) from [6] for easy comparison. In the case of S1, the agreement between $<P_2>_{exp}$ and η_{theo} in the SmA phase leaves much to be desired, η_{theo} always remaining greater than $<P_2>_{exp}$ However, agreement in the nematic phase between $<P_2>_{exp}$ and $<P_2>_{MS}$ is excellent. The η_{theo} values predictably are also nearly coincident with $_{MS}$ as should ideally be the case in the nematic phase. The nature of variation of $<P_2>_{exp}$ in the case of S1 is distinctively different from that of S5, showing only a slow and gradual variation along the entire mesogenic range that may be treated as nearly constant. $<P_2>_{exp}$ of S5 on the contrary exhibits a sharp decline from values ~0.77 (80°C) to 0.43 at SmA–Iso transition.

3.2. *Dielectric studies and dipole moments*

The nature of thermal variation of dielectric permittivities ε parallel ($\varepsilon_{||}$) and ε perpendicular ($\varepsilon_{\perp}$) at frequencies of 10 and 100 KHz are depicted in Fig. 4(a) and (b). The average dielectric permittivity values, ε_{avg} are 7.00 and 6.75 for 10 and 100 KHz respectively at 80°C. In the case of S5, the corresponding values at 10 and 100 KHz at 80°C are 6.76 and 7.42, indicating a reversal in trend with increasing frequency as compared with S1 [6]. In the case of S1, both $\varepsilon_{||}$ and $\varepsilon_{\perp}$ increase with temperature at both the frequencies, resulting in an upward trend in thermal variation of ε_{avg}. This feature is in contrast with the nature exhibited by S5, where ε_{avg} remains more or less constant throughout the SmA range. The discrepancy of ε_{avg} with ε_{iso} at T_{NI} in the case of S1 is significant. This feature, although not as prominent, is also observed in the optical behavior, where n_{avg} shows a slight but perceptible discontinuity with n_{iso} at the N–Iso transition. This behavior is attributed to the molecules with relatively high or moderate molecular dipole moment [14] as discussed earlier. For S5, the discontinuity of ε_{avg} with ε_{iso} at $T_{SmA\text{–}Iso}$ is hardly perceptible [6]. This behavior is also in agreement with the optical behavior of S5 where discrepancy between n_{avg} and n_{iso} at $T_{SmA\text{–}Iso}$ is hardly discernable. Although in nematic–isotropic transitions, this is generally attributed to non-polar compounds [13], the same may possibly not be inferred for SmA–Iso transition as pointed out earlier.

Dielectric anisotropy $\Delta\varepsilon$ is 0.348 for S1 (80°C at 10 KHz) and declines with frequency to 0.295 (80°C at 100 KHz). The same trend is observed for S5, where $\Delta\varepsilon$ values at 10 and 100 KHz are 1.79 and 1.42 respectively at 80°C [6].The estimated error in the determination of dielectric permittivity is about 2%.

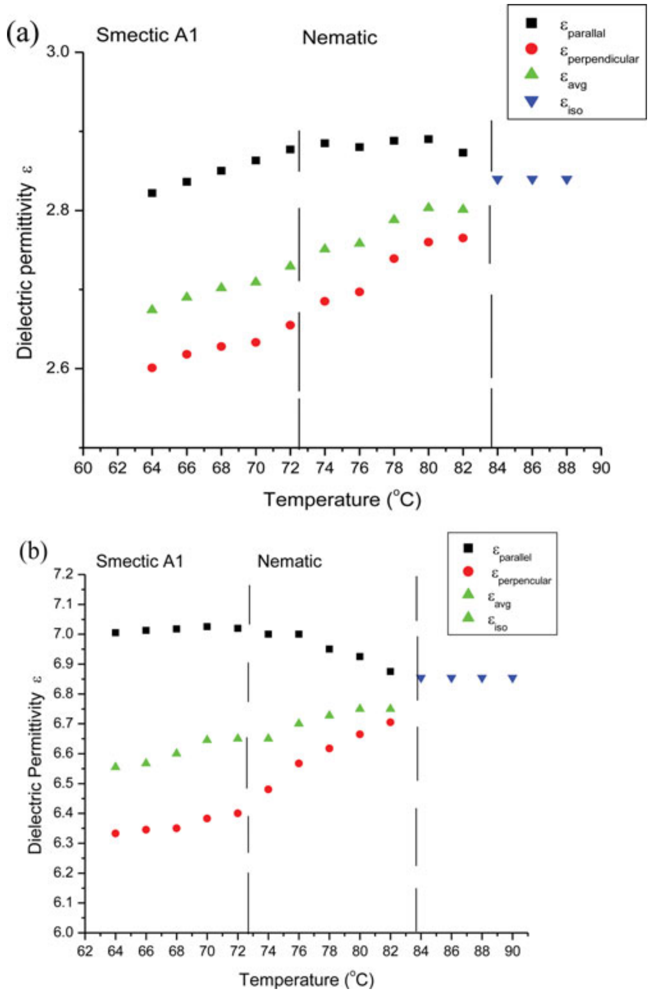

Figure 4. (a) Variation in dielectric constant with temperature *T* at 10 KHz for S1. (b) Variation in dielectric constant with temperature *T* at 100 KHz for S1.

The effective dipole moment μ_{eff} and the angle of inclination β have been computed using the following relations [17]:

$$\varepsilon_{\mathrm{avg}} = 1 + 4\Pi\mathrm{NhF}(\alpha_{\mathrm{avg}} + \mathrm{F}\,\mu_{\mathrm{eff}}\,2/3\mathrm{KT})$$

and

$$\Delta\varepsilon = \frac{4\Pi\mathrm{NhF}\{\Delta\alpha - \mathrm{F}\,\mu_{\mathrm{eff}}^{2}(1 - 3\cos^{2}\beta)\}}{2\mathrm{KT}} < P_2 > ,$$

where 2KT

$$\varepsilon_{\mathrm{avg}} = (\varepsilon_{\mathrm{II}} + \varepsilon_{\perp})/3, \qquad \alpha_{\mathrm{avg}} = (\alpha_{\mathrm{e}} + 2\,\alpha_{\mathrm{o}})/3\,,$$
$$f = 4\Pi\mathrm{N}(\varepsilon_{\mathrm{avg}} - 2)/\{3(\varepsilon_{\mathrm{avg}} + 1)\}, \qquad h = 3\,\varepsilon_{\mathrm{avg}}/(2\varepsilon_{\mathrm{avg}} + 1),$$
$$F = 1/(1 - \alpha_{\mathrm{avg}}\mathrm{f}).$$

The values of polarisability anisotropy $\Delta\alpha$, average molecular polarisability α_{avg}, and orientational order parameter $<\mathrm{P}_2>$ at different temperatures have been taken from our optical data for the computation of $\mu_{\mathrm{eff.}}$

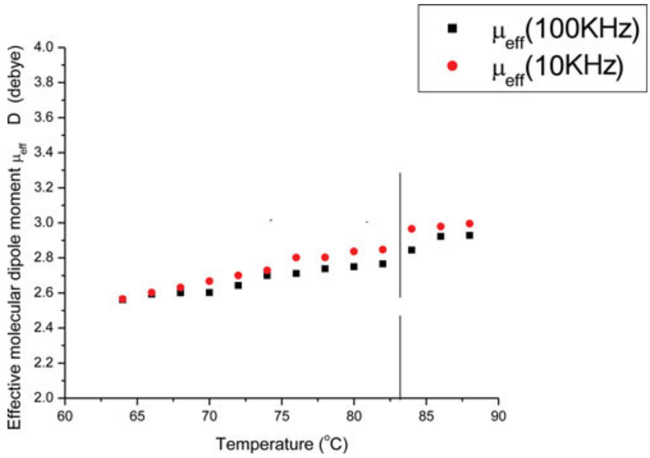

Figure 5. Variation of effective molecular dipole moment (μ_{eff}) with temperature *T* for S1.

Variation in μ_{eff} with temperature at two different frequencies of 10 and 100 KHz of S1 are shown in Fig. 5; μ_{eff} increases gradually with temperature over mesogenic range for both frequencies and is slightly greater for the lower frequency of 10 KHz. Typical values at 80°C for frequencies of 10 and 100 KHz are 2.75 and 2.85 Debye. For S5, μ_{eff} is greater for higher frequency at any temperature, values respectively being 2.96 and 3.16 Debye at 10 and 100 KHz at 80°C [6], a trend distinct from the S1 case.

The temperature dependence of the angle of inclination β of S1 at frequencies of 10 and 100 KHz is depicted in Fig. 6. β values increase with an increase in temperature, showing a sharp discontinuity in behavior, especially at the nematic–isotropic transition. In fact, the trend in SmA and nematic phases are distinctly different at higher frequency of 100 KHz. In SmA phase, β values at the two frequencies are almost the same, the difference in behavior at two different frequencies being manifested in nematic phase.The β values at 100 KHz are slightly higher than at 10 KHz in general, typical value being 63.37° at 80°C at 10 KHz, and for S5, typical value is 47.47° (80°C at 10 KHz) [6].

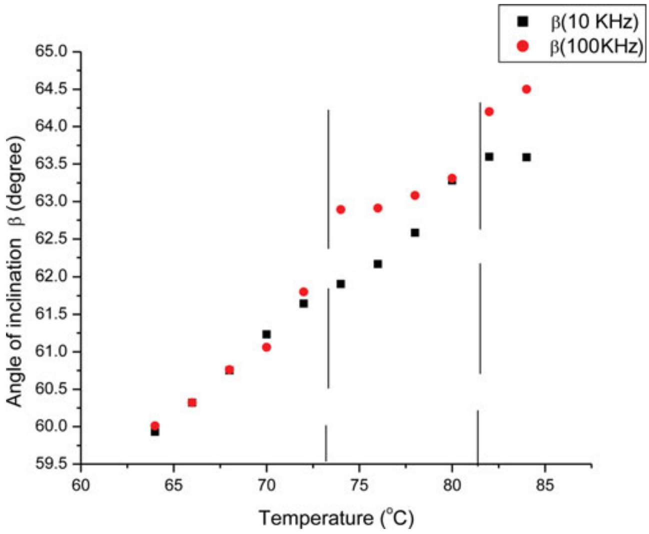

Figure 6. Variation of angle of inclination (β) with temperature *T* for S1.

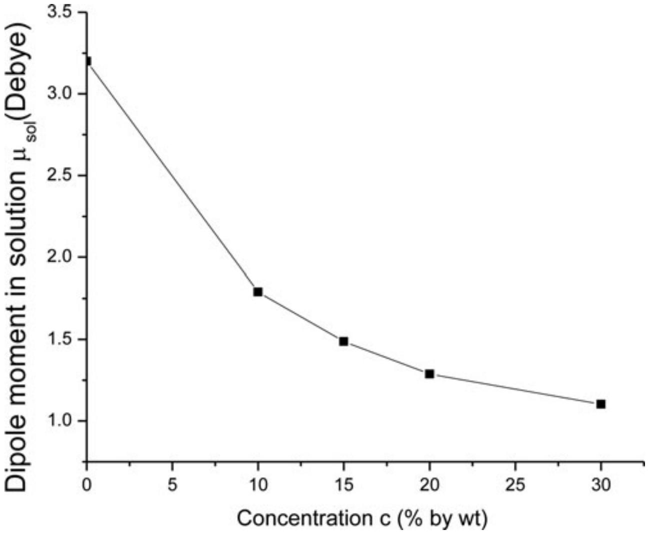

Figure 7. Variation of molecular dipole moment in solution (μ_{sol}) with concentration at 80°C of S1.

3.3. *Dipole moment in solution*

Variation of dipole moment in solution μ_{sol} with concentration c (%wt) is shown in Fig. 7 at a temperature of 80°C and an operating frequency of 10 KHz. By extrapolating the curve to an infinite dilution, the isolated or free molecular dipole moment of S1 is obtained and is found to be 3.20 Debye. In the case of S5, the corresponding value of dipole moment is 3.15 Debye [6], only slightly less than for that for S1, i.e. both the molecules possess significant dipole moment values. Comparing this value of μ_{iso} for S1 with its μ_{eff} value (at 80°C and 10 KHz), the value of correlation coefficient $g = (\mu_{eff}{}^2/\mu_{iso}{}^2)$ is found to be 0.73, which is less than the value of the correlation coefficient for S5, which is 0.89. The correlation factor g, as defined in the Kirkwood–Frolich theory [18,19], gives a measure of molecular association between a reference molecule and its nearest neighbors, and its deviation from unity is indicative of molecular association. With $g = 1$ corresponding to no association between molecules, the Kirkwood–Frolich equation reduces to the Onsagar equation [20]. The value of $g < 1$ is indicative of antiparallel orientation of neighboring molecules, whereas $g > 1$ is indicative of parallel orientation. The value of g for S1 indicates an antiparallel orientation, which is stronger than that of S5, which has lower g value.

4. Conclusions

The effect of terminal subsituents such as NCS group in liquid crystal compounds and mixtures have been extensively studied, and it is well established that terminal isothiocyanto groups lead to increased optical and dielectric anisotropy. The present study on S1 also confirms high optical and polarisability anisotropy values comparable with those of S5, which also has a terminal isothiocyanto group and differs structurally from S1 only in chain length. Our study reveals that the presence of isothiocyanto (NCS) group in terminal position, in addition to increasing the anisotropies, causes a significant contribution to the increase of molecular dipole moments, both S1 and S5 having isolated molecular dipole moment values of 3.20 and 3.15 Debye respectively.

Compound S1 is found to have an antiparallel orientation of neighboring molecules similar to that of S5, although the association is stronger in the case of S1 as suggested by greater deviation of correlation coefficient from unity.

The variation of experimentally obtained orientational order parameter is more gradual for S1 as compared with S5, indicating the greater functional stability of S1.

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