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Liquid Crystals

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Birefringence, permittivity, elasticity and rotational viscosity of ambient temperature, high birefringent nematic liquid crystal mixtures

D. Venkata Sai^a, P. Sathyanarayana^a, V.S.S. Sastry^a, J. Herman^b, P. Kula^b, R. Dabrowski^b and Surajit Dhara^{a*}

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We prepared a few binary nematic liquid crystal mixtures with high birefringence (≈ 0.4 at room temperature) and studied the temperature variation of physical properties. All the mixtures show a small, positive dielectric anisotropy and the parallel component of dielectric constant exhibits anomalous temperature dependence. Orientational order parameter, both splay, bend elastic constants and rotational viscosity are comparatively larger than conventional low birefringent liquid crystal mixtures. The figure of merit (FoM) of the mixtures are also calculated as a function of temperature and expected to be useful for various applications.

Keywords: nematic liquid crystal; high birefringence; viscoelasticity; figure of merit

1. Introduction

Liquid crystals used in flat panel display devices are optically anisotropic, i.e., birefringent. The typical values of birefringence of many commercial mixtures are in the range of 0.05–0.2. Liquid crystals with high birefringence have attracted attention because of their non-display applications. For example, they are essential for laser beam steering,[1] tunable focus lenses,[2] optical switches for telecommunication, infrared special light modulators,[3] and directional reflectors.[4] They also have significant importance for radio frequency applications in the GHz and THz frequency range,[5,6] broad range filters and holographic devices. Moreover, high birefringence enhances the display brightness and contrast ratio of polymer-dispersed liquid crystal (PDLC), holographic PDLC, cholesteric liquid crystal display and liquid crystal gels.[7–9] The birefringence of a liquid crystal strongly depends on the molecular structure and π -electron conjugation.[10,11] Usually more linearly conjugated liquid crystal exhibits larger birefringence.[12] The increase of conjugation length leads to high melting temperature, decrease of nematic range, increased viscosity and poor solubility to form eutectic mixtures. One possible way to increase nematic range is to prepare eutectic mixture of high birefringent liquid crystals. There are some reports on the synthesis and optical measurement of high birefringent nematic liquid crystals.[13–23] However, the detail physical characterisation such as dielectric and viscoelastic measurements are rare. In this paper, we report on the preparation and detail characterisation

of a few high birefringent mixtures containing quarterphenyl and phenylethynyltolane cores.

2. Experimental

The experimental cells were made of two indium tin oxide (ITO)-coated glass plates with circularly patterned electrodes. The plates were spin coated with polyimide (AL-1254) and cured at 180°C for 1 h and rubbed antiparallel way for planar or homogeneous alignment of the sample. For homeotropic alignment, ITO plates were spin coated with polyimide (JALS-204) and cured at 200°C for 1 h. Cells were made by placing the two plates together, ensuring that the active electrode area overlaps. The separation was controlled by glass-bead spacers with diameter 5 μm . The thickness of the empty cell was measured with $\pm 1\%$ accuracy by an interferometric technique using spectrometer (Ocean Optics, HR-4000). Alignment of the sample was observed using a polarising optical microscope (Nikon, LV100 POL) and a temperature controller (Instec, mk1000). The dielectric constants are measured by using a LCR metre (Agilent 4980). The perpendicular component of the dielectric constant was measured in planar cell below the Freedericksz threshold voltage. To measure the parallel component of dielectric constant, the effective dielectric constant was measured as a function of voltage (0.02–20 V) at a fixed temperature. The experimental variation of the voltage dependent dielectric constant tends to saturate at high voltages. The linear part of the dielectric constant is plotted

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against $1/V$ and extrapolated to $1/V = 0$ to obtain ϵ_{\parallel} at various temperatures. The measured value is in good agreement with the measurement made in an independent cell.

The optical retardation was measured by using a phase modulation technique with the help of a Helium–Neon laser (633 nm), a photoelastic modulator (PEM-100) and a lock-in amplifier (DSP-7265). A sinusoidal voltage of frequency 4111 Hz and an amplitude up to 20 V at steps of 0.02 V were used for the measurements of dielectric constant by using the LCR meter. The dielectric dispersion was measured by an impedance analyser (Novocontrol, Alpha-A). The retardation and also the sample capacitance were measured simultaneously as a function of temperature and voltage. The splay elastic constant (K_{11}) is obtained from the Freedericksz threshold voltage (V_{th}) and is given by $K_{11} = \epsilon_0 \Delta\epsilon (V_{th}/\pi)^2$, where $\Delta\epsilon = \epsilon_{\parallel} - \epsilon_{\perp}$ is the dielectric anisotropy. K_{33} was estimated from the fitting of the voltage-dependent optical retardation with the theoretical calculations.[24–27] Rotational viscosity (γ_1) was measured using the phase-decay-time measurement technique.[28–31]

3. Results and discussion

3.1 Birefringence and orientational order parameter

We prepared and studied three different mixtures using four different nematic liquid crystals. The chemical structure of the individual compounds and the phase transition temperatures of the mixtures are shown in Figure 1. At room temperature, they are nematic and highly turbid. In each mixture, one compound is common and non-polar with larger wt%. The second compound is polar with isothiocyanate terminal group containing laterally alkyl, fluorine and chlorine substituted and less in wt%. The polar compounds are mixed with the non-polar host with maximum solubility. All the mixtures have high clearing point and wide nematic range. The synthesis and phase transition temperatures of the individual compounds are recently reported.[11] The temperature variation of birefringence (Δn) of the mixtures are shown in Figure 2. Δn jumps to about $\simeq 0.22$ from 0 at the nematic–isotropic (NI) transition in all the mixtures and reaches to a high value of $\Delta n \sim 0.4$ at room temperature. The birefringence of the mixtures increases as follows: $\Delta n_{Mix-1} > \Delta n_{Mix-2} > \Delta n_{Mix-3}$. This can be understood based on the molecular structure of the individual

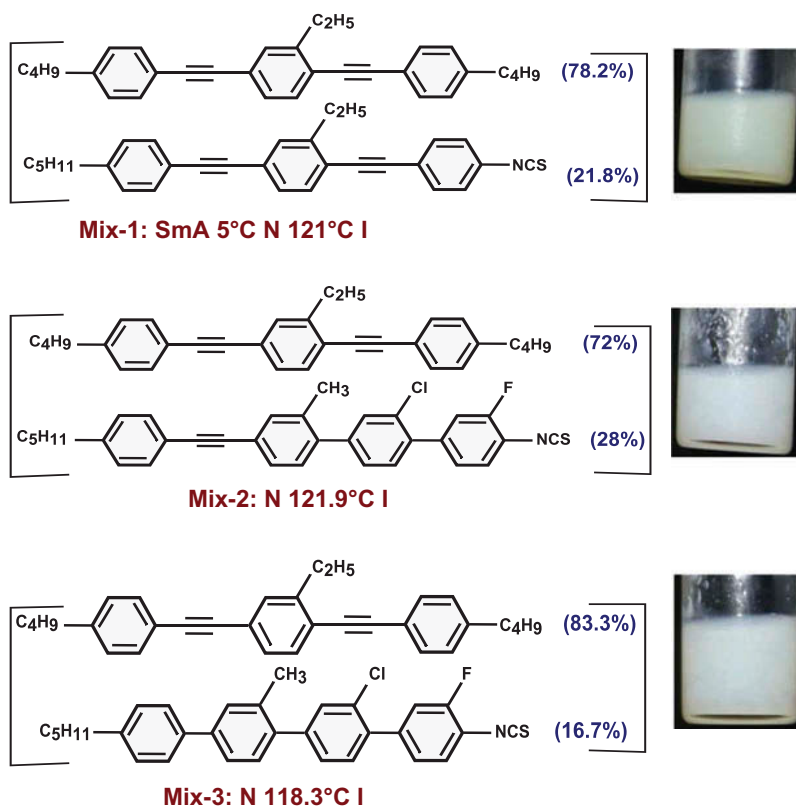


Figure 1. (colour online) Molecular structure and wt% of the individual compounds and the phase transition temperature of the mixtures (in cooling). Nematic \rightarrow N and Smectic-A \rightarrow SmA. The physical appearance of the samples in the bottles at room temperature are also shown.

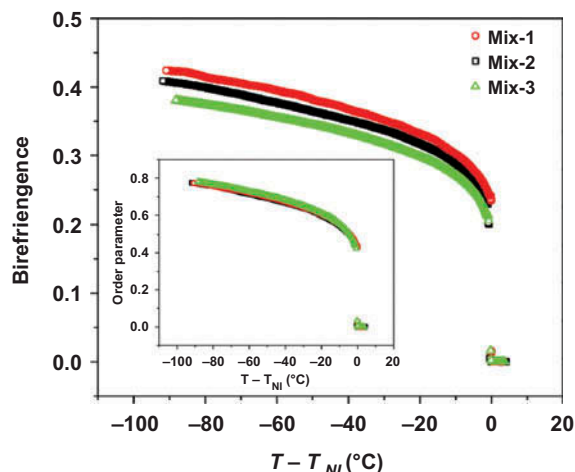


Figure 2. (colour online) Variation of birefringence (Δn) and (inset) orientational order parameter (S) of the mixtures as a function of shifted temperature.

components in the mixture. In all the compounds, a significant contribution to the Δn is due to the presence of $-C\equiv C-$ bonds and phenyl rings. Usually, $-C\equiv C-$ contributes more to Δn than phenyl rings.[11] As we proceed from Mix-3 to Mix-1, we notice that apart from a small variation in the lateral groups, the number of phenyl rings decreases and $-C\equiv C-$ groups increases. Thus the effective conjugation length and hence Δn of the mixture increases.

To estimate order parameter, the temperature variation of Δn can be approximated by the Haller extrapolation formula.[32] $\Delta n = \Delta n_0(1 - T/T_1)^\beta$, where β , T_1 are the adjustable parameters, and Δn_0 is the birefringence of the perfectly aligned sample. The fit parameters are listed in Table 1. T_1 is slightly higher than T_{NI} , and β is about ≈ 0.18 . Similar value of β was also reported in many other liquid crystals.[33] The order parameter S of the long molecular axis was estimated using the relation $S \approx \Delta n/\Delta n_0$. The temperature variation of S is shown in Figure 2. The value of S increases with decreasing temperature and at room temperature it is quite high (about $S \approx 0.8$) and larger than that of many common liquid crystals. Similar large order parameter in high birefringent nematic liquid crystals were also reported by Sekine et al.[34]

Table 1. Fit parameters of different mixtures.

Mixture	Δn_0	T_1 (°C)	T_{NI} (°C)	β
Mix-1	0.55	125	121	0.18
Mix-2	0.53	125.4	121.9	0.18
Mix-3	0.49	120	118.3	0.17

3.2 Dielectric constants

The variation of parallel (ϵ_{\parallel}) and perpendicular (ϵ_{\perp}) components of dielectric constants and the average, $\bar{\epsilon} = (\epsilon_{\parallel} + 2\epsilon_{\perp})/3$, as a function of temperature is shown in Figure 3(a). The dielectric anisotropy is positive, i.e., $\Delta\epsilon (= \epsilon_{\parallel} - \epsilon_{\perp}) > 0$ and almost the same in all the mixtures. ϵ_{\parallel} increases up to a certain value and then decreases significantly near the room temperature. Similar unusual behaviour of ϵ_{\parallel} was observed in many compounds showing nematic to smectic phase transition.[25,35] In addition, in those compounds, it was found that ϵ_{\perp} also tend to increase at the same temperature and eventually $\Delta\epsilon$ can change sign. It was shown that due to the presmectic order, the antiparallel correlation of the dipoles parallel to the director increases and as a result the effective dipole moment reduces in the nematic phase leading to the decrease of ϵ_{\parallel} . Similar argument was given for increase of ϵ_{\perp} . However, in the present mixtures only Mix-1 exhibits N – SmA transition around 5°C and

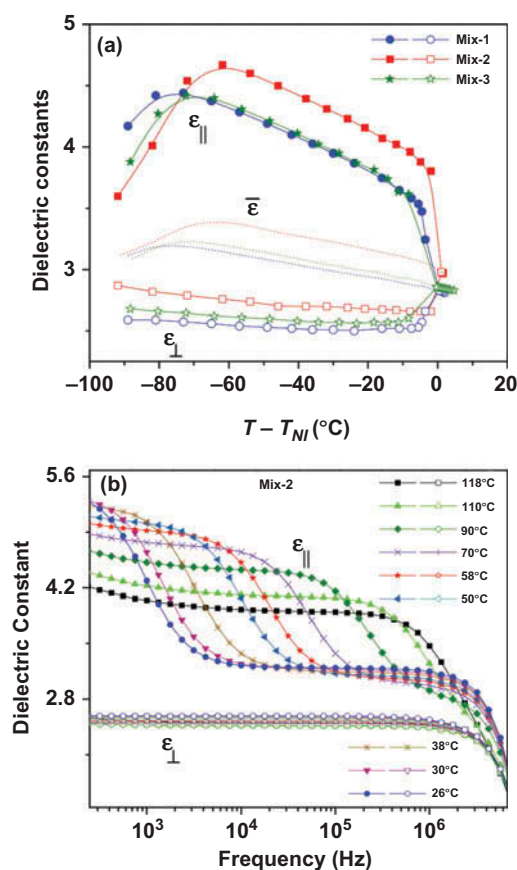


Figure 3. (colour online) (a) Variation of parallel (ϵ_{\parallel}), perpendicular (ϵ_{\perp}) components of dielectric constant and $\bar{\epsilon} = (\epsilon_{\parallel} + 2\epsilon_{\perp})/3$ as a function of shifted temperature. (b) The frequency dispersion of real part of the dielectric constants, i.e., $\epsilon_{\parallel}(f)$ and $\epsilon_{\perp}(f)$ at various temperatures of Mix-2. Solid lines are drawn as guides to the eye.

Mix-2 and Mix-3 do not show any smectic phase but some kind of solidification was noted around this temperature. In addition, ϵ_{\perp} does not show any increasing tendency at the same temperature. Thus, the unusual temperature dependence of ϵ_{\parallel} of the mixtures can not be understood based on the antiparallel correlation of dipoles. To understand this, we measured the frequency dispersion of both $\epsilon_{\parallel}(f)$ and $\epsilon_{\perp}(f)$ in one sample (e.g. Mix-2) in the frequency range of 100 Hz – 10 MHz (Figure 3(b)). We note that in both the components, one relaxation is common (beyond 1 MHz) which comes from the finite resistance of the ITO electrodes. $\epsilon_{\perp}(f)$ is almost independent of temperature and does not show any dielectric relaxation. On the other hand $\epsilon_{\parallel}(f)$ exhibits another dielectric relaxation below 1 MHz and the relaxation frequency decreases rapidly with decreasing temperature. For example, relaxation frequency at 90°C is about 237 kHz and reduces to 1 kHz at room temperature (26°C). The temperature-dependent dielectric constant was measured at a fixed frequency 4.11 kHz and the relaxation frequency decreases below this value around 50°C. Hence the unusual decrease of ϵ_{\parallel} near room temperature is due to the decrease of relaxation frequency below the measuring frequency. The detailed dielectric dispersion measurements of all the samples will be reported elsewhere.

3.3 Elastic constants

The variation of splay (K_{11}) and bend (K_{33}) elastic constants as a function of temperature is shown in Figure 4. Though K_{11} is almost the same within the experimental accuracy, K_{33} appears to be slightly different in the three mixtures. Both K_{11} and K_{33} increase with decrease of temperature and these values are

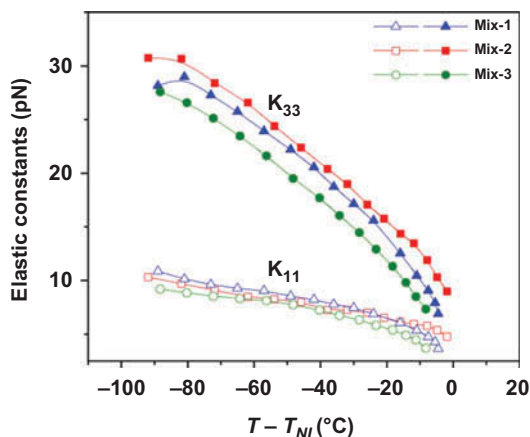


Figure 4. (colour online) Variation of splay (K_{11}) and bend (K_{33}) elastic constants as a function of shifted temperature. Solid lines are drawn as guides to the eye.

comparatively larger than that of many common (low birefringent) nematic liquid crystals. For instance, in 5CB, $K_{11} = 5$ pN and $K_{33} = 9$ pN whereas in the present mixture (Mix-1), these numbers are 10 and 30 pN, respectively at room temperature. Since $K_{ii} \propto S^2$, [36] the large elastic constant could be due to the large orientational order of the mixtures. It is also noticed that K_{33} tends to decrease slightly near the room temperature at which ϵ_{\parallel} shows anomalous dependence. This is due to the decrease of the dielectric anisotropy.

3.4 Rotational viscosity

Finally, we discuss the rotational viscosity (γ_1) of the mixtures in the nematic phase. The intensity of the transient response of various mixtures are measured. A representative variation of $\ln[\delta_o/\delta(t)]$ with time (t) is shown in Figure 5(a). γ_1 was calculated from the measurement of relaxation time (τ_o) and K_{11} of the mixtures. The variation of γ_1 as a function of temperature is shown in Figure 5(b). γ_1 increases very rapidly with decrease of temperature. For instance, at room temperature, this is $\simeq 1$ Pa s (Mix-2) which is about two orders of magnitude larger than conventional (low birefringent) nematic liquid crystals. The rotation viscosity can be written as $\gamma_1 \sim S \exp(W/kT)$, W being the activation energy, S the order parameter and k the Boltzmann constant. [35] Since $S \propto \Delta n$, we plotted $\ln(\gamma_1/\Delta n)$ with $1/T$ in Figure 5(b) (inset). The estimated activation energy for the three mixtures are 330, 350 and 380 meV respectively and these are comparable to the values reported for many low birefringent nematic liquid crystals. [30]

To characterise the performance of the liquid crystal materials, we estimated figure of merit (FoM) using the formula, $\text{FoM} = K_{11}\Delta n^2/\gamma_1$ as a function of temperature. Figure 6 shows the temperature variation of the FoM. Since, $K_{11} \propto S^2 \propto \Delta n^2$, the temperature dependence of FoM can be written as follows:

$$\text{FoM} = A(1 - T/T_1)^{3\beta} \exp(-W/kT). \quad (1)$$

FoM is largest for Mix-1 and reduced in Mix-2 and Mix-3. At higher temperature, the FoM of all the mixtures are reasonably larger than conventional low birefringent nematic liquid crystals but it decreases at room temperature. For instance, for Mix-1, at 95°C, FoM is 15 $\mu\text{m}^2/\text{s}$ and decreases to 1.5 $\mu\text{m}^2/\text{s}$ near room temperature. The reports on FoM at room temperature, high birefringent nematic liquid crystal is rare hence not available for comparative discussion. Though the birefringence is large at

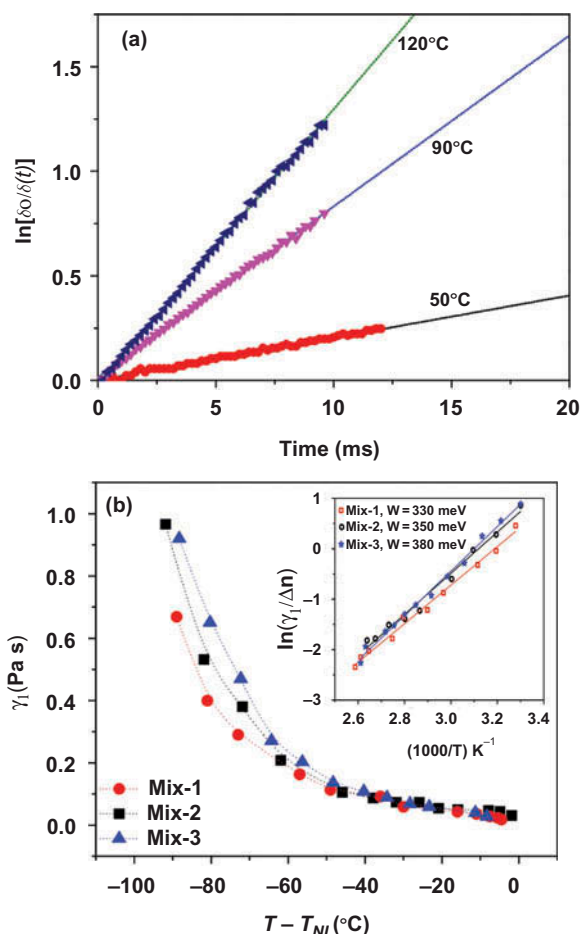


Figure 5. (colour online) (a) Linear variation of $\ln[\delta_0/\delta(t)]$ with time(t) at various temperatures of Mix-1. Solid lines are theoretical fits to the experimental data. (b) Variation of rotational viscosity γ_1 of different mixtures as a function of shifted temperature (inset). Linear variation of $\ln(\gamma_1/\Delta n)$ with $1/T$. Solid lines are theoretical fits to the corresponding equations as described in the text.

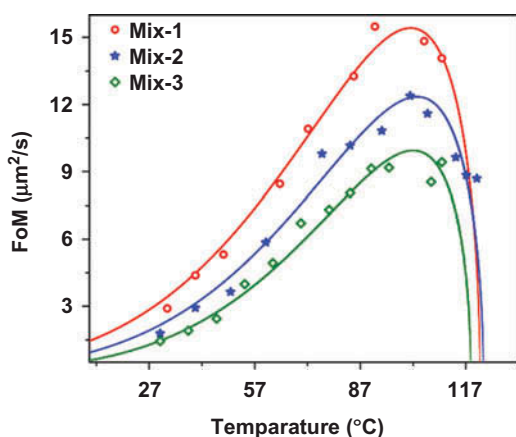


Figure 6. (colour online) Temperature variation of figure of merit (FoM). Solid lines are theoretical fits of the experimental data to Equation (1).

room temperature, high viscosity is disadvantageous especially where fast switching is needed.

4. Conclusion

In conclusion, we prepared and measured various physical properties of three high birefringent room temperature nematic liquid crystal mixtures. The orientational order parameter, both splay and bent elastic constants are comparatively larger than that of many common liquid crystal mixtures. The unusual temperature dependence of parallel component of dielectric constant near the room temperature is due to the rapid decrease of dielectric relaxation frequency with decreasing temperature. The FoM at higher temperature is reasonably large but reduces at room temperature due to the very high viscosity. Thus elastic and dielectric properties of high birefringent materials are suitable but high viscosity is a matter of concern for application and hence needs to be reduced.

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