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PAPER

Splay-bend elasticity and rotational viscosity of liquid crystal mixtures of rod-like and bent-core molecules

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We report temperature dependent measurements of birefringence (Δn), splay (K_{11}), bend (K_{33}) elastic constants and rotational viscosity (γ_1) of mixtures of rod-like and bent-core molecules. K_{11} and Δn remain almost constant for all the mixtures whereas K_{33} is always lower than K_{11} and further decreases with increasing concentration of bent-core molecules in the mixtures. Apart from the large jump in Δn at the nematic-isotropic transition (NI) a significant jump in Δn at the nematic-smectic transition (NS) is also observed that increases with increasing mol% of the bent-core compound in the mixtures. Rotational viscosity increases by a factor of four when the concentration of the bent-core compound increased from 0 to 10 mol%. The results are discussed on the basis of their structural similarity and mutual alignment in the nematic phase.

I. Introduction

The interests in investigating the structure-property relation in unconventional liquid crystals has increased significantly in the past decade after the discovery of the electrooptical switching in bent-core liquid crystals.¹ Several new and complex structures known as 'banana' or B-phases (e.g., B_1 , B_2 , etc) owing to their shape in such unconventional liquid crystals are already reported.²⁻⁴ Apart from the B-phases these bent-core compounds occasionally exhibit some phases of calamitic liquid crystals like nematic and smectic. The physical properties of the nematic phase composed of bent-core molecules have been reported to show several interesting and new results. For example, it has been found that bent-core nematic liquid crystals exhibit sign inversion of elastic anisotropy,^{5,6} unusual electroconvection,⁷⁻⁹ giant flexoelectricity,^{10,11} large viscosity,^{12,13} Kerr coefficient,¹⁴ induced¹⁵ and spontaneous biaxiality.^{16,17} Nematic phases composed of a mixture of rod-like and bent-core compounds are also interesting as they often exhibit unusual physical properties.¹⁸⁻²² For example, there are some reports on the unusual elastic properties in the mixture of rod and bent-core liquid crystals. In particular, Dodge *et al.* showed that addition of a small concentration of bent-core liquid crystals in a calamitic compound can reduce K_{33} by a factor of two or more.^{18,19} Kundu *et al.* reported anomalous trends in the temperature dependence of splay (K_{11}) and bend (K_{33}) elastic constants in the nematic phase of mixtures of 4-n-octyloxy 4-cyanobiphenyl (8OCB) and the present bent-core compound (Fig. 1(a)) and explained the results on the basis of

their mutual orientation²⁰ in the presence of short-range smectic fluctuations. In this paper, we present the measurements of Δn , K_{11} , K_{33} and γ_1 as a function of temperature and show that these physical properties of the mixture are significantly different than those previously reported in other binary systems due to the

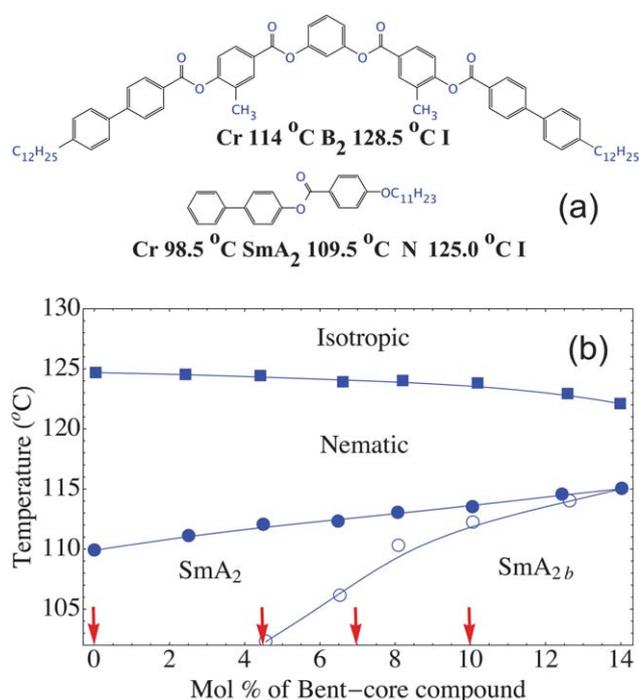


Fig. 1 (a) Chemical structures of the compounds and their phase transition temperatures. (b) Phase diagram of the mixtures of the above two compounds. Vertically down red arrows indicate the mol% chosen for the experiments.

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structural similarity, *i.e.*, the aromatic and aliphatic parts of the rod are matched with one half of the bow in the present system.

II. Experimental

Liquid crystal cells were prepared by using two indium tin oxide (ITO) coated glass plates. These plates were treated with polyimide (AL-1254) and cured at 180 °C for 1h and rubbed anti-parallel for planar or homogeneous alignment of the sample. The cell thickness was measured by using a fibre optic spectrometer (Ocean Optic, HR4000). Typical cell thickness used in the experiment was $\sim 9 \mu\text{m}$. The cell was filled with the sample in the isotropic phase and a well aligned nematic phase was obtained on cooling. The perpendicular component of the dielectric constant (ϵ_{\perp}) was measured in a planar cell below the Freedericksz threshold voltage by using a LCR meter (Agilent 4980A). To measure the parallel component of dielectric constant (ϵ_{\parallel}) the effective dielectric constant was measured from 0.02 to 20V. The experimental variation of the voltage dependent dielectric constant tends to saturate at high voltages. The linear part of dielectric constant is plotted 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 measurements made in an independent cell.

The optical retardation was measured by using a phase modulation technique²³ with the help of a Helium-Neon laser, a photoelastic modulator (PEM-100) and a lock-in amplifier (SR-830). A sinusoidal voltage of frequency 4111 Hz and amplitude up to 20 V at steps 0.02 V were used for the measurements by using the LCR meter. The retardation and also the sample capacitance were measured simultaneously as functions of temperature and voltage. The splay elastic constant (K_{11}) is obtained from the Freedericksz threshold voltage (V_{th}) and is given by $K_{11} = \epsilon_0 \Delta\epsilon (V_{\text{th}}/\pi)^2$, where $\Delta\epsilon = \epsilon_{\parallel} - \epsilon_{\perp}$ is the dielectric anisotropy. At strong surface anchoring conditions, the voltages above the threshold voltage V_{th} and the retardation ($\Delta\phi$) are given by the parametric equations:²⁴

$$\frac{V}{V_{\text{th}}} = \frac{2}{\pi} \sqrt{1 + \gamma \sin^2(\phi_m)} \int_0^{\frac{\pi}{2}} \sqrt{\frac{1 + \kappa \sin^2(\phi_m) \sin^2(\psi)}{(1 + \gamma \sin^2(\phi_m) \sin^2(\psi))(1 - \sin^2(\phi_m) \sin^2(\psi))}} d\psi \quad (1)$$

$$\Delta\phi = 2\pi \frac{n_e d}{\lambda}$$

$$\left(\frac{\int_0^{\frac{\pi}{2}} \sqrt{\frac{(1 + \gamma \sin^2(\phi_m) \sin^2(\psi))(1 + \kappa \sin^2(\phi_m) \sin^2(\psi))}{(1 - \sin^2(\phi_m) \sin^2(\psi))(1 + \nu \sin^2(\phi_m) \sin^2(\psi))}} d\psi}{\int_0^{\frac{\pi}{2}} \sqrt{\frac{1 + \gamma \sin^2(\phi_m) \sin^2(\psi)}{(1 - \sin^2(\phi_m) \sin^2(\psi))}} d\psi} - \frac{n_o}{n_e} \right) \quad (2)$$

where d is the cell thickness, ϕ_m is the tilt angle at the middle of the cell and the other terms of the reduced quantities are: $\gamma = (\epsilon_{\parallel}/\epsilon_{\perp}) - 1$, $\kappa = (K_{33}/K_{11}) - 1$, $\nu = (n_e^2/n_o^2) - 1$, $\sin(\phi) = \sin(\phi_m)\sin(\psi)$. The sample retardation at higher voltage is fitted to the above

equations by an iterative procedure to get the bend elastic constant K_{33} .^{20,25}

To verify the anchoring condition we estimated the pretilt angle (α) of the mixtures by putting the lower limit of the integration in eqn (1) and (2) as a fit parameter α . In all the mixtures it was found to be less than 1° indicating the strong anchoring at the surface hence α was assumed to be zero in further analysis. It may be pointed out that a pure bent-core nematic also exhibits a very low pretilt angle on the same alignment layer.^{13,26} Independent measurements were also carried out to measure the temperature dependent anchoring energy of the mixtures which also indicated strong anchoring and the details will be published elsewhere.

The phase-decay-time measurement technique was used to measure the rotational viscosity (γ_1). A signal generator (Tektronix, AFG3102) was used to apply voltage at a frequency of 4.111 kHz and a photomultiplier tube (Hamamatsu, H6780-01) was used as a detector to measure the time dependent transmitted intensity. A small voltage (V_b) corresponding to the first maxima or minima was applied depending on transmission intensity such that the total phase retardation of sample was $n\pi$, where n is an integer. At time $t = 0$, the bias voltage (V_b) was removed and the relaxation transmission intensity change of the liquid crystal cell was measured with an oscilloscope (Tektronix, TDS 2012B).

The time dependent intensity at a particular temperature is given by:²⁷

$$I(t) = I_0 \sin^2\{\Delta_{\text{tot}} - \delta(t)\}/2, \quad (3)$$

where I_0 is the maximum intensity change and Δ_{tot} is the total phase difference. The optical phase difference $\delta(t)$, for small director distortion can be approximated as $\delta(t) = \delta_0 \exp(-2t/\tau_0)$, where δ_0 is total phase difference of liquid crystal under bias voltage (V_b) that is not far from the Freedericksz threshold voltage (V_{th}). In the case where δ_0 is close to $n\pi$, $\delta(t)$ becomes $\delta(t) = \delta_0 \exp(-4t/\tau_0)$. The slope of the plot $\ln[\delta_0/\delta(t)]$ with time (t) yields the relaxation time (τ_0). The rotational viscosity (γ_1) of the liquid crystal is given by:

$$\gamma_1 = \frac{\tau_0 K_{11} \pi^2}{d^2} \quad (4)$$

where d is the thickness of liquid crystal sample. This procedure was repeated in the entire nematic range for all the mixtures.

III. Results and discussion

We studied various mixtures of compounds 4-biphenyl-4'-*n*-undecyloxybenzoate and 1,3-phenylene-bis[4-(3-methylbenzoyloxy)]-4'-*n*-dodecylbiphenyl-4'-carboxylate. The chemical structures of the compounds and their individual phase transition temperatures are shown in Fig. 1(a). The mixture exhibits some new phase transitions and the detailed phase diagram is reported by Pratibha *et al.*^{28,29} The part of phase diagram showing NI phase transition is reproduced (Fig. 1(b)) and is the interest of the present study. The NI transition temperature decreases slightly (Fig. 1(b)) and the N-SmA₂ transition temperature increases with increasing mol% of bent-core compound and SmA_{2b} appears beyond ~ 4 mol%. Very recently we have made detailed calorimetric measurements and showed that the critical heat anomaly

associated with the SmA_2 to SmA_{2b} transition is described with a Fisher-renormalized form of the usual scaling expression.³⁰ The temperature dependent variation of the birefringence (Δn) of the pure compound and various mixtures are shown in Fig. 2(a). Δn jumps to ~ 0.06 from 0 at NI transition in the pure as well as in all the mixtures and increases with decreasing temperature in the nematic phase. Δn is almost constant in all the mixtures at a fixed temperature which suggests that the orientation order, S ($\propto \Delta n$) is not affected by the inclusion of the bent-core molecules. A significant jump in Δn is also observed at the NS transition in all the mixtures including the pure compound and indicate that the NS transition is first order and consistent with the calorimetric measurements.³⁰ Further, the relative jump in Δn across the NS transition appears to increase with increasing mol% of the bent-core compound and indicates the increase in the coupling of the smectic order parameter ψ and fluctuation in the nematic order parameter S as the McMillan parameter (α) increases from 0.96 to 0.97 in the present mixtures. Measurement of Δn , K_{11} and K_{33} of mixture of the present bent-core compound with 8OCB were reported by Kundu *et al.*²⁰ They reported inverse effect *i.e.*, Δn decreases significantly with increasing concentration of bent-core compound.

The temperature dependent variation of dielectric anisotropy for the various mixtures ($\Delta\epsilon = \epsilon_{\parallel} - \epsilon_{\perp}$) is shown in Fig. 2(b). The dielectric anisotropy is positive and relatively small and decreases with increasing mol% of bent-core compound at a fixed temperature. The resultant dipole moment of the rod-like

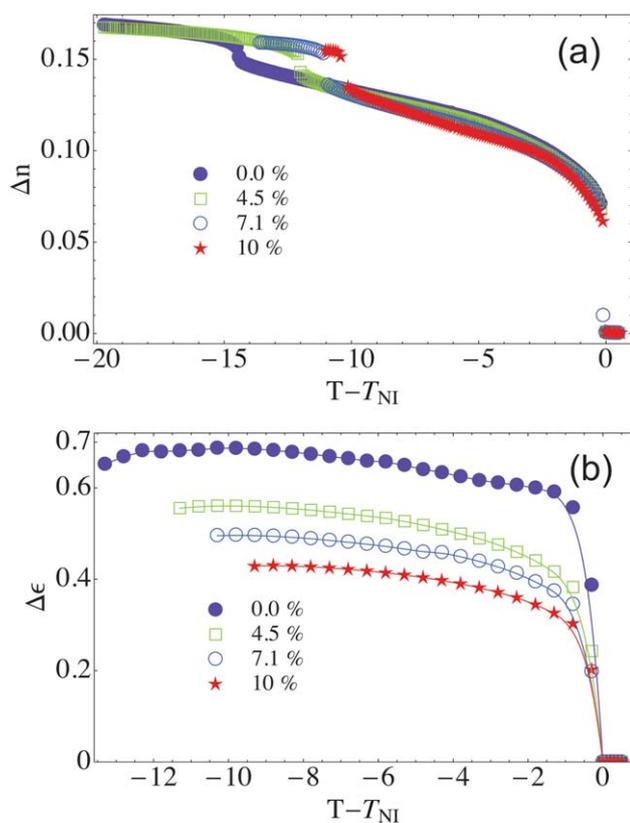


Fig. 2 Variation of (a) birefringence (Δn) and (b) dielectric anisotropy ($\Delta\epsilon$) of the mixtures as a function of temperature. Continuous lines are drawn as a guide to the eye.

molecules is along the long axis whereas the bent-core molecules have transverse dipole moments. The reduction in dielectric anisotropy with increasing concentration of the bent-core compound in the nematic phase, keeping the orientational order almost the same, suggests that the long axes of the bent-core molecules on an average are parallel to the long axes of the rod-like molecules as shown schematically in Fig. 3(a).

The optical phase difference ($\Delta\phi$) of the sample was measured as a function of applied voltage to measure splay (K_{11}) and bend (K_{33}) elastic constants at various temperatures. A typical variation of experimental data together with the best fit to eqn (1) and (2) is shown in Fig. 4(a). The temperature-dependent variation of splay (K_{11}) and bend (K_{33}) elastic constants are shown in Fig. 4(a) and Fig. 4(b) respectively. K_{11} increases with decreasing temperature in the nematic phase and show a little change of curvature as the smectic phase is approached. Interestingly at a fixed temperature there is no significant change in K_{11} with increasing mol% of the bent-core compound similar to Δn (Fig. 2a). Since Δn ($\propto S$) do not change with the mol% of the bent-core compound, K_{11} ($\propto S^2 \propto \Delta n^2$) is also not affected. On the other hand K_{33} decreases significantly (Fig. 4(b)) with increasing concentration of the bent-core compound. Variation of K_{11} and K_{33} at two temperatures namely $T - T_{\text{NI}} = 3.8^\circ$ and 7.8° is shown in Fig. 4(b) (inset). K_{11} remains constant and K_{33} is reduced by a factor of ~ 1.7 and tends to saturate as the concentration is increased from 0 to 10 mol%. Measurement of K_{11} and K_{33} in the mixture of the present bent-core compound with 8OCB was also reported by Kundu *et al.* and they found that both decreases with increasing concentration of bent-core compound in the mixtures.²⁰ The strikingly different results *i.e.*, both Δn and K_{11} remains constant in the present mixtures is attributed to the structural similarity, *i.e.*, the aromatic and aliphatic parts of the rod are matched with one half of the bow. Further as mentioned earlier the bow axes of the bent-core molecules are parallel to the long axes of the rod-like molecules (Fig. 3(a)), and the bent molecules do not facilitate splay fluctuations hence K_{11} remains almost the same for all mixtures. The decrease of bend elastic constant (K_{33}) is due to the coupling of the bent shape of the bent-core molecules with bent distortion as

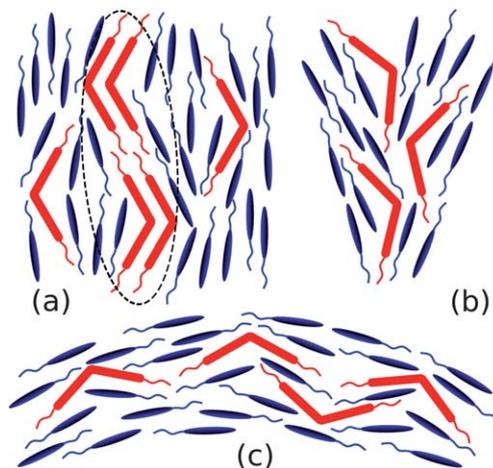


Fig. 3 (a) Mutual alignment of rod-like and bent-core molecules in nematic phase. Dotted region show a temporary cluster in the nematic phase. Schematic representation of (b) splay and (c) bent distortion of the director in the mixtures.

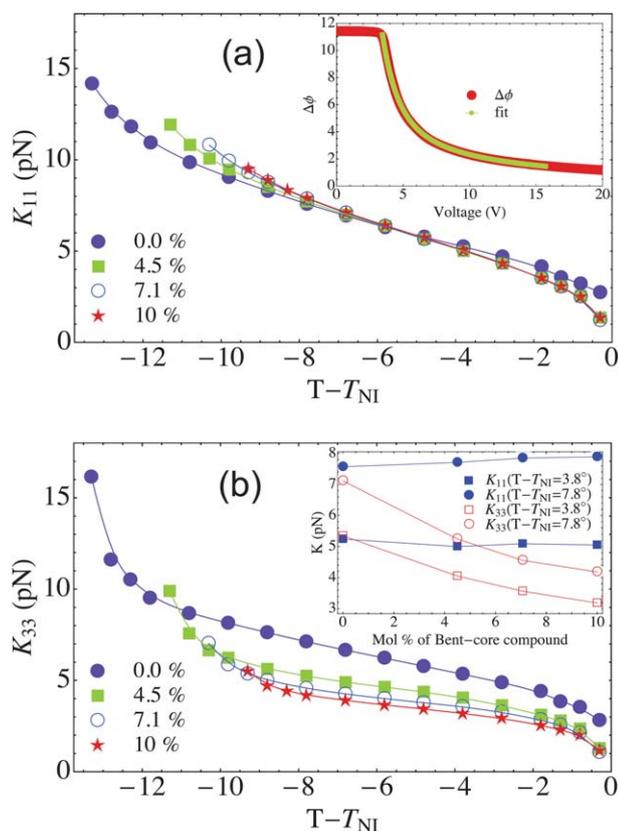


Fig. 4 (a) Variation of splay elastic constant (K_{11}) of various mixtures as a function of temperature. (Inset) Retardation as a function of applied voltage at $T - T_{NI} = -7^\circ$. Continuous green line is a theoretical fit to eqn (1) and (2). (b) Variation of bend elastic constant (K_{33}) of various mixtures as a function of temperature. (Inset) Variation of both K_{11} and K_{33} with the concentration of bent-core compound. Continuous lines are drawn as a guide to the eye.

described by Sathyanarayana *et al.*⁵ They suggested that the strain in the bent distortion is partly relieved due to the coupling and as a result K_{33} can be significantly lower than K_{11} . A schematic representation of the splay and bend distortions in the mixture is shown in Fig. 3(b) and 3(c) respectively. The pretransitional divergence of K_{33} in pure compound as the N-SmA₂ transition is approached is due to smectic short-range fluctuations in the sense that the bent fluctuations are suppressed in the onset of smectic short-range order. Such pretransitional divergence of K_{33} in the mixture is also observed in other calamitic and unconventional mesogens.^{5,19,25,31} Furthermore, it is noticed that the relative divergence in K_{33} in the mixtures compared to the pure compound as the N-SmA transition is approached is reduced with increasing mol% of the bent-core compound. This is a consequence of the reduced K_{33} in the sense that bent fluctuations are relatively larger in the mixtures compared to the pure compound.

Finally we discuss the rotational viscosity (γ_1) of the mixtures in the nematic phase. The normalised intensity of the transient response of various mixtures is shown in Fig. 5(a). Time dependence of the retardation was estimated from the normalised intensity by using eqn (3). A representative variation of $\ln[\delta_o/\delta(t)]$ with time (t) of experimental data (10 mol%) together with the best fit is shown in Fig. 5(a)(inset). γ_1 was estimated from

eqn (4) by measuring the relaxation time τ_o and K_{11} of several mixtures. The temperature-dependent variation of γ_1 is shown in Fig. 5(b). γ_1 of the pure compound is relatively low and comparable to many known calamitic compounds.³² It increases with decreasing temperature in the nematic phase as expected due to the increase in the orientational order parameter (S).³³ In the mixture γ_1 increases significantly compared to the pure compound with increasing mol% of bent-core compound. For example, at two temperatures namely $T - T_{NI} = 1.8^\circ$ and 6.8° , it increases by a factor of four when the concentration of the bent-core compound is increased from 0 to 10 mol% (Fig. 5(b), inset). It may be mentioned that in the mixture the increase in γ_1 can be solely attributed to the increase in τ_o since K_{11} almost remains constant. The temperature dependent variation of γ_1 was reported to be higher in the pure bent-core compounds than conventional calamitic compounds.^{13,34,35} In some cases it was reported to be 10 times higher and was explained due to the formation of temporary smectic clusters that originate from the bent-shape of the molecules.^{6,34} Existence of such clusters in the nematic phase is revealed in dynamic light scattering³⁶ and rheological studies.³⁷ In the present study γ_1 is almost 4 times larger even at a relatively small concentration (10 mol%) of the bent-core compound and expected to be contributed by such

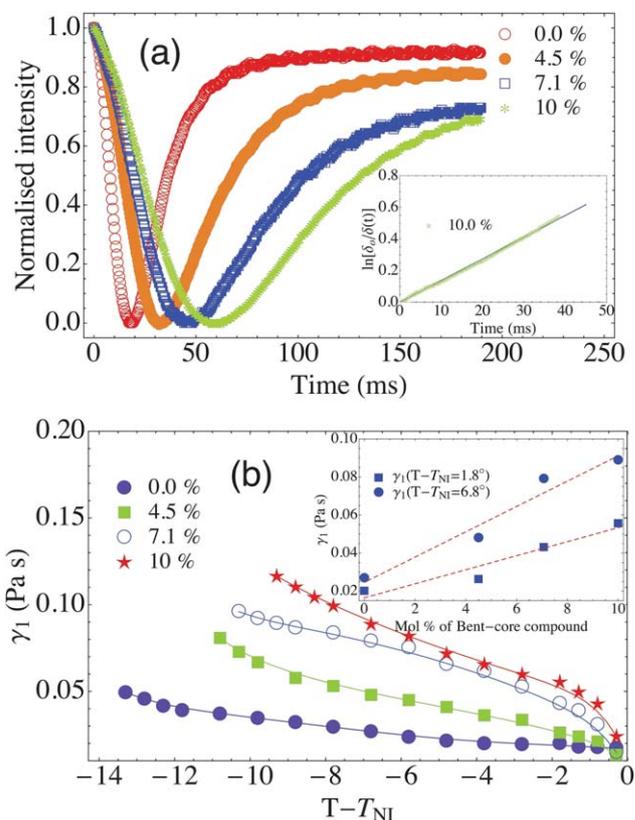


Fig. 5 (a) Time dependent normalised transmitted intensity after the removal of the bias voltage V_b at $T - T_{NI} = -2^\circ$ for various mixtures. (Inset) Linear variation of $\ln[\delta_o/\delta(t)]$ with time (t) at the same temperature for the mixture with 10 mol % bent-core compound. (b) Variation of the rotational viscosity (γ_1) of mixtures as a function of reduced temperature. (Inset) Variation of γ_1 with the concentration of bent-core molecules at two different reduced temperatures. Continuous lines are drawn as a guide to the eye.

temporary clusters of bent-core molecules. A schematic representation of such temporary clusters in the nematic phase is depicted in Fig. 3(a). The size and the number of clusters can increase with increasing concentration of bent-core molecules and γ_1 of the mixture can increase.

IV. Conclusion

In conclusion, we have measured the birefringence, splay and bend elasticity and rotational viscosity of several mixtures of rod-like and bent-core molecules in which the aromatic and aliphatic parts of the rod are matched with one half of the bent-core molecule. We observed that the birefringence and hence the orientational order (S) remain the same in all mixtures. The dielectric anisotropy decreases with an increase in concentration of the bent-core molecules indicating that bow axes of bent-core molecules align parallel to the long axes of rod-like molecules. The splay elastic constant remains constant, whereas the bend elastic constant decreases significantly with increasing concentration of bent-core molecules suggesting the coupling of the bent-shape with the bend distortion. The increase in rotational viscosity with concentration suggests the presence of temporary smectic clusters of bent-core molecules in the mixtures.

It may be mentioned that the flexoelectric effect in such mixtures could be significant and can affect K_{33} measurements. It was shown by Brown *et al.*³⁸ from voltage dependent dielectric measurements that it can slightly underestimate K_{33} when the flexoelectric effect is neglected. Further it was shown by Kundu *et al.*²¹ that the mixture of bent-core and rod-like nematic samples exhibits homeotropic alignment because of a large positive self-energy due to a high flexoelectric coefficient at higher concentrations of the bent-core molecules. In our present study the highest concentration of bent-core molecules is higher than their mixtures but no such effect was observed suggesting that the flexoelectric coefficient may not be significantly large. In our experiment we used retardation data to measure K_{33} and found it is reduced by a factor of ~ 1.8 times at 10 mol%. Such a large reduction in the present data can not be accounted for by merely the flexoelectric effect. A detail theoretical investigation is needed to find the effect of flexoelectric as well as order electric polarization on the voltage dependent retardation measurements.

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