# SOME PHYSICAL STUDIES ON LIQUID CRYSTALS AND THEIR MIXTURES

A thesis submitted to the Bangalore University for the degree of **Doctor of Philosophy** in the Faculty of Science

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by

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September 1993

### DECLARATION

I hereby declare that the entire work embodied in this thesis is the result of the investigations carried out by me independently in the Liquid Crystal Laboratory, Raman Research Institute, Bangalore, and that no part of it has been submitted for the award of any Degree, Diploma, Associateship or any other similar title.

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## CERTIFICATE

I certify that this thesis has been composed by Ms. H. P. Padmini based on the investigations carried out by her at the Liquid Crystal Laboratory, Raman Research Institute, Bangalore, under my supervision. The subject matter of this thesis has not previously formed the basis of the award of any Degree, Diploma, Associateship, Fellowship or other similar title.

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# **CONTENTS**

	Preface	i-xiv
Chapter I	Introduction	1
Chapter II	Electromechanical effect in cholesteric liquid crystals with free boundary conditions	20
Chapter III	Electromechanical effect in cholesteric samples with fixed boundary conditions: Theoretical analysis	42
Chapter IV	Experimental study of electro- mechanical effect in samples with fixed boundary conditions	62
Chapter V	Electroclinic response of some ferroelectric liquid crystals: Part I	78
Chapter VI	Electroclinic measurements - Part II: Determination of the coefficients of the Landau theory	99

### Preface

This tlies describes some experimental studies on liquid crystals and their mixtures. It is concerned mainly with the effect of electric fields on liquid crystalline materials consisting of chiral molecules.

In Chapter I, we give a general introduction to the subject with particular reference to tlie phenomena reported in the rest of the tliesis. In particular some relevant properties of nematic, cholesteric, smeetic C, and smeetic C\* phases are outlined. The hydrodynamics of cholesterics, defects in cliolesterics and some properties of ferroelectric liquid crystals are the topics reviewed in this chapter.

In Chapter II, we give the results of our measurements of the electromeclianical coupling coefficient  $\nu_E$  using drops with zero anchoring energy. In particular we present the variation of  $\nu_E$  with the pitch of the cholesteric material.

Due to the macroscopic chiral arrangement in a cholesteric, it sustains novel cross couplings between hydrodynamic fluxes and forces. An excellerit example of such an effect is the *Lehmann rotation* phenomenon. Lehmann observed in 1900 that a vertical thermal gradient, which is a force with the characteristic of a polar vector, sets cholesteric drops into a rotational motion (see also Chandrasekhar, 1977). Leslie (1968) and others have worked out detailed hydrodynamic theories of

cholesterics and obtained solutions corresponding to the Lehmann effect. According to these models, the coupling constants depend on the macroscopic chirality associated with the helical arrangement of the director (de Gennes, 1975; Pleiner and Brand, 1987). In such a case in the simplest approximation, the thermomechanical coupling coefficients would be  $\propto q(=2\pi/P)$ , the wavevector of the helix. For the compensated cholesteric with q=0, the coefficients should vanish.

de Gennes has pointed out that any transport current should give rise to a similar effect. Recently Madhusudana and Pratibha (1987, 1989) demonstrated the electromechanical coupling which arises due to the transport of ions in a cholesteric sample. They found an electromechanical rotation in cholesteric drops with free boundary conditions.

If an electric field acts along the helical axis of a sample contained between two conducting glass plates, the torque balance equation takes a very simple form:

$$\gamma_1 \frac{\partial \phi}{\partial t} = K_{22} \frac{\partial^2 \phi}{\partial z^2} + \nu_E E \tag{1}$$

where  $\gamma_1 = \alpha_3 - \alpha_2$  is the difference between two Leslie viscosity coefficients,  $\phi$  the azimuthal angle,  $K_{22}$  is the twist elastic constant,  $\nu_E$  an electromechanical coupling coefficient. The above equation can be integrated to obtain a solution for  $\phi$  by using appropriate boundary conditions. An interesting solution is obtained if the anchoring energy for azimuthal orientation is zero on both the surfaces. The solution is then of the form (Leslie, 1968; de Gennes, 1975)

$$\phi = q_o z + \frac{\nu_E E}{\gamma_1} t + c \tag{2}$$

where c is a constant of integration. It corresponds to a continuous rotation of the structure with time, i.e., the *Lehmann Rotation*. The angular velocity is given by

 $(\nu_E E)/\gamma_1$ . The cholesteric drops which are found to follow this dynamical behaviour in our experiment contain a  $\chi$ -line defect and the above equation has to be modified to take into 'account the entropy production due to the motion of this defect.  $d\phi/dt$ is equal to  $(\nu_E E)/3\gamma_1$  in the presence of a line defect rotating with the structure.

The material chosen for our experiment consisted of a binary mixture of alkoxy phenyl trans-alliyl cyclohexyl carboxylates to get a room temperature nematic with  $\Delta\epsilon \approx -1$ . The dielectric anisotropy is chosen to be negative to avoid a change in the orientation of the director due to the dielectric coupling with the field. Cholesteryl chloride and methyl butyl benzoyloxy heptyloxy cinnamate were added to get cholesteric materials with left arid right handed helical arrangements respectively. The pitch was measured using the Cano wedge technique. By dissolvirig a small percentage of an epoxy compound, viz., Lixon, flat cholesteric drops were obtained. These drops were surrounded by the isotropic phase on all sides as Lixon has a strong affinity for glass. Thus the azimuthal anchoring energy at the surface of the drops was zero so that the structure of the drops could rotate freely. On application of a DC electric field to the electrodes, the structure was found to rotate.  $\nu_E$  was calculated using the slopes of  $d\phi/dt$  vs. E curves for samples with different values of the pitch which were got by varying the concentration of the cholesteric material. Indeed,  $\nu_E$  was found to be proportional to q, as expected on the basis of the phenomenological models. We found that a compensated cholesteric mixture which contains both cholesteryl chloride and methyl butyl benzoyloxy heptyloxy cinnamate does not show the rotation phenomenon, clearly demonstrating that the electromechanical effect is essentially of structural origin (Fig.1).

While the Lehmann rotation phenomenon remains the most convincing evidence for the cross coupling term in the cholesteric phase, experimentally it requires a very



Figure 1. Variation of the electromecharlical coupling coefficient  $\nu_E$  as a function of q (= $2\pi/p$ ).

special combination of chemicals to get the required type of drops. It is comparatively easier to prepare samples with fixed boundary conditions by having a strong anchoring of the director at the glass plates. In chapter III, a theoretical analysis of the electromechanical effect in cholesteric samples with rigid boundary conditions and subjected to both DC and AC electric fields is given.

When the director is fixed at both the boundaries z=0 and d, under the action of an applied DC field the medium has a static deformation. For a material with *negative* dielectric anisotropy the solution to equation (1) becomes

$$\phi(z) = \frac{\phi_d z}{d} + \frac{\nu_E E z}{2K_{22}} (d-z).$$
(3)

where the azimuthal angle of the director  $\phi = 0$  and  $\phi_d$  at z = 0 and d respectively.

In the absence of the applied field, the director has a uniform twist in the sample. When the field is applied, the variation of  $\phi$  across the sample thickness becomes non-uniform such that the thickness averaged value  $\overline{\phi}$  will be greater than or lower than that in the field free case depending on the sign E (and that of  $\nu_E$ ). It is usually preferable to apply AC rather than DC electric fields to liquid crystals to avoid electrolytic processes. This results in  $\phi$  oscillations. With the usual materials and typical sample thicknesses, the phase difference of the sample is much larger than the electromechanical contribution to the angle of twist. In such a case, the polarisation of the light beam incident along the z-axis follows the director (Mauguin criterion) and the deformation in  $\phi$ -profile cannot be detected by such a bearn.

This optical problem can be overcome by using a material which has a positive dielectric anisotropy. When an AC field is applied to the sample so that the applied voltage is above the threshold for the Freedericksz transition, a tilt-deformation is produced in the director field thus reducing the effective birefringence of the sample. This reduces the total phase difference introduced by the cell. Then the Mauguin criterion does not apply and we can detect the changes in the  $\phi(z)$  profile.

A detailed theoretical analysis of this problem is also given in chapter III. Under an AC electric field, the torque balance equation can be written as

$$\Gamma^{el} + \Gamma^{diel} + \Gamma^{EM} - \Gamma^{hydro} = 0 \tag{4}$$

where

 $\Gamma^{el}$  is the elastic torque,

 $\Gamma^{diel}$  is the dielectric torque,

 $\Gamma^{EM}$  is the torque due to electromechanical coupling, and  $\Gamma^{hydro}$  is the hydrodynamic torque.

It leads to two coupled non-linear partial differential equations in  $\theta$  and  $\phi$ . We have tried to solve these equations using a standard programme. The calculated  $\theta$  profile which is symmetric about the midpoint of the cell (Fig.2) is found to be practically independent of the sign and magnitude of q. The profile does not change sign with time as  $\theta$  oscillations arise due to the dielectric anisotropy of the sample and hence the relevant torque is quadratic in E. On the other hand, the  $\phi$  profile is asymmetric about the centre of the cell (Fig.3). This is because the torques  $\Gamma_2$  and  $\Gamma_3$  depend linearly on  $q \frac{\partial \phi}{\partial z}$  and  $q \frac{\partial \theta}{\partial z}$  respectively. The asymmetry changes *sign* during one period. This is because 4-oscillations are caused by the electromechanical coupling which is linear in E, which changes sign with time. As the sign of q is changed, the sign of  $\phi$  profile also changes.

We have also calculated the transmitted intensity as a function of time when the sample is kept between crossed polarisers (Fig. 4). We may note that there are two



Figure 2.  $\theta$  plotted as a function of position across the cell thickness at three different times. (a) t=0.225 T, (b) 0.5 T and (c) 0.775 T for  $q = 3 \times 10^5 m^{-1}$ . Cell thickness = 3  $\mu m$ ; applied voltage = 4.93 V, frequency = 18 Hz.



Figure 3.  $\phi$  plotted as a function of position across the cell thickness at three different times. (a) t=0.225 T, (b) 0.5 T and (c) 0.775 T for  $q = +3 \times 10^5 m^{-1}$ . Cell thickness = 3  $\mu m$ ; applied voltage = 4.93 V, frequency = 18 Hz.



Figure 4. Intensity of transmitted light plotted as a function of time for  $q = +3 \times 10^5 m^{-1}$ .

peaks within one period, but the second peak is slightly higher than the first for q > 0 showing that there is an f component which arises from the electromechanical coupling. When q < 0, the first peak is higher than the second one and the phase angle of the corresponding f component is opposite to that in the q > 0 case.

We describe in chapter IV our experimental studies on electromechanical effect in cholesteric samples taken in cells made of appropriately treated glass plates which provide fixed boundary conditions to the director.

In the case of materials with negative dielectric anisotropy, the average azimuthal angle of the distorted director field was sensed by a conoscopic technique using thick samples (d = 50  $\mu$ m) and voltages above the restabilisation threshold of EIID instability. The conoscopic pattern consists of two hyperbolic dark bands which rotate back and forth showing that the average value of  $\phi$  oscillates at the frequency of the applied field (Fig. 5). The value of the electromechanical coupling coefficient estimated by the conoscopic method is consistent with our earlier measurement of  $\nu_E$  using cholesteric drops with free boundary conditions.

We have also conducted electrooptic experiments on samples with a weak positive dielectric anisotropy. In this case at voltages above the Freedericksz threshold, a tilt-deformation is produced in the director field thus reducing the effective birefringence of the sample. It then becomes possible to detect optically the changes in the  $\phi(z)$  profile. For this purpose, we used mixtures of 82% of 2-cyano-4-heptyl phenyl-4'-pentyl-4-biphenyl carboxylate [7P(2CN)5BC], 18% of 4'-n-lieptyl-4-cyanobiphcnyl (7CB) and a cholesteric material.

At low enough frequencies of about a few IIz, the medium had a very clear linear electrooptic effect when kept between a pair of crossed polarisers and oriented such





Figure 5. Print from a video recording showing the rotation of the conoscopic figures of a 50  $\mu m$  thick negative  $\Delta \epsilon$  material when the voltage oscillates between +210 V (left) and -210 V (right) at 6 Hz.

that the surface alignment made an angle  $\psi$  of  $\pi/8$  radians with the polariser. The  $\phi$  oscillations due to the electromechanical coupling  $\psi$  give rise to a maximum optical signal at the frequency of the applied AC signal when  $ll = \pi/8$  radians.

The electrooptic signal oscillates at twice the frequency of the applied field whien  $\psi$  is changed to  $\pi/4$  radians. This signal arises from the **0** oscillations which occur due to the dielectric anisotropy of the sample which couples quadratically with E. The corresponding optical signal at twice the frequency of the applied AC voltage has its maximum value for  $\psi = \pi/4$  radians.

We also found that a nematic sample without any chiral molecules but with some twist angle shows the  $\psi$  angle dependence of the f and 2f signals which is similar to the one described above. When we twist a nematic we produce a macroscopic chirality in the sample and our observations again confirm that the electromechanical coupling has a contribution from the macroscopic helical arrangement of the director (Fig.6).

Our results on untwisted chiralised, twisted chiralised and twisted nematic samples indicate that the electromechanical coupling has a contribution which depends on q the natural wavevector, and a contribution which depends on the macroscopic twist.

We also present our measurements of the electromechanical coupling coefficient in systems whose pitch changes handedness as the temperature is varied across a *compensation* temperature. For this purpose a suitable cholesteric mixture was chosen consisting of 7P(2CN)5BC, 7CB and cholesteric chloride. The pitch and the handedness of the helix were determined from the Cano-Grandjean disclinations in wedge shaped cells. As the temperature increases, the electromechanical signal de-



**Figure 6.** The  $\psi$  angle dependence of the f and 2f signals from a twisted nematic sample without any chiral component and with weak positive dielectric anisotropy. Twist angle = 20°, f = 18 Hz. The signals were amplified through the transformer input of the lock-in-amplifier.

creases and changes sign at the compensation temperature (Fig.7). The signal again increases as the temperature is further increased. It is seen that as the frequency increases, the electromechanical signal decreases. We may note that the compensated mixture used in the experiment on cholesteric drops in chapter II has only about 10% of the chiral additives. But the compensated mixture used for the experiment in the present experiment contains  $\sim 56\%$  of cholesteric material. This again shows that the EM coupling constant predominantly depends on the macroscopic chirality associated with the helical arrangement of the director and not that of the molecule.

In Chapter V we give our results on measurements of the electroclinic coefficient in smectic A liquid crystals made of chiral molecules.

The electroclinic effect in the smeetic A phase of a chiral liquid crystal was discovered by Garoff and Meyer (1977, 1979). A remarkable property of this effect is the associated fast electrooptic response. When an electric field is applied parallel to the smeetic layers, a molecular tilt angle  $\theta$  relative to the smeetic layer normal is induced. Unlike the electromechanical effect which depends on macroscopic chirality, electroclinic effect (ECE) is essentially molecular in origin. The electroclinic coefficient strongly depends on the smeetic C\* like short range order.

Wlien an electric field is applied to the sample in the smectic A phase parallel to the smectic layers, it produces a non-zero average of the transverse component of the molecular polarization (P). This produces a 2-fold axis which, because of the lack of mirror symmetry produces a tilt in a plane perpendicular to P. In an aligned smectic **A** sample, the tilt of the director is directly related to the tilt of the optic axis. Therefore the ECE results in a linear electrooptic response.



Figure 7. Temperature variation of the f signal at 7 Hz (O), 17 Hz ( $\Box$ ), 27 Hz ( $\diamondsuit$ ), 37 Hz (A) and 57 Hz ( $\diamondsuit$ ).

Following the Landau theory of Garoff and Meyer, the mean field expression for the free energy density of a smectic A liquid crystal made up of chiral molecules is given by

$$F = F_o + \frac{1}{2}A(T)\theta^2 + \frac{1}{2}\chi_{\mathcal{P}}^{-1}\mathcal{P}^2 - \mathcal{P}E - \frac{\epsilon_{\infty}E^2}{2} - c\mathcal{P}\theta$$
(5)

Here  $F_o$  is the ground state free energy of the smectic A phase, A(T) is the temperature dependent Landau coefficient given by  $a(T - T_c)$ , **0** is the induced tilt angle,  $\chi_P$  is the generalized susceptibility,  $\mathcal{P}$  is the induced polarization, E is the external field, c is the electroclinic coupling constant between  $\mathcal{P}$  and  $\theta$  and  $\epsilon_{\infty}$  is the high frequency dielectric constant.

Minimising the free energy with respect to  $\theta$  and **P** 

$$\theta = \frac{\chi \mathcal{P}^c}{A(T^*)} E \tag{6}$$

and

$$\mathcal{P} = \chi_{\mathcal{P}} E + \frac{\chi_{\mathcal{P}}^2 c^2}{A(T^*)} E \tag{7}$$

where

$$A(T^*) = \tilde{a} \frac{(T - T_c^*)}{T_c^*}$$
$$T_c^* = T_c + \frac{\chi p^{c^2}}{a}$$

and 
$$\tilde{a} = aT_c^*$$

Due to the linear coupling  $c\mathcal{P}\theta$ , the transition temperature is displaced in a chiral compound compared to its non-chiral analogue. When a sinusoidal field  $Ee^{iwt}$  is applied to the cell,  $A(T^*)$  is to be replaced by  $[A(T^*)] + iwq$ , where  $\eta$  is the soft mode viscosity.

It can be shown that the amplitude of the  $\theta$  oscillations at the frequency w is

$$\theta_o = \frac{c\chi \mathcal{P}^E}{\left[w^2\eta^2 + \tilde{a}^2 \left(\frac{T - T_c^*}{T_c^*}\right)^2\right]^{1/2}}$$
(8)

The phase angle of the  $\theta$  oscillations with respect to the applied field is given by

$$\delta = tan^{-1} \left[ -\frac{w\eta}{\tilde{a} \left( \frac{T - T_c^*}{T_c^*} \right)} \right]$$
(9)

It is clear from equation (8) that as T approaches  $T_c^*$ , the viscous term restricts the divergence of  $\theta_o$  and thereby the amplitude tends to a saturation value. The relaxation time of the fluctuations of the order parameter  $\theta$ , viz.,

$$\tau = \frac{\eta}{\tilde{a}\left[\frac{T-T_c^*}{T_c^*}\right]} \tag{10}$$

also diverges as  $T_c^*$  is approached.

The experimental set-up used in this experiment is the same as that used to measure the electromechanical coupling coefficient. We use a PIN photodiode which has very fast response to monitor the optical signals. We have made the measurements on a homologous series, viz., [2S,3S]-4'-(2-chloro-3-methyl pentanoyloxy) phenyltrans-4"-n-alkoxy cinnamates (D series) synthesized in our laboratory and also on two commerical samples, viz., SCE-5, SCE-6.

We measured the electroclinic coefficient  $e = \frac{\theta}{E}$  where E is the amplitude of the applied electric field as a function of temperature. The electroclinic coefficient rapidly increases as the A-C\* phase transition temperature is approached. Some typical results are shown in figure 8 for SCE-5.

We have plotted the inverse electroclinic coefficient as a function of temperature for all the systems studied and a typical variation is shown in figure 9. As expected



**Figure** 8. Variation of electrooptical signal as a function of temperature for SCE-5.



Figure 9. Temperature dependence of the inverse electroclinic coefficient,  $e^{-1}$  of the 9th homologue at, 1960 Hz.

from the Landau theory, the variation of  $e^{-1}$  is quite linear at temperatures a little above  $T_c^*$ . As we approach  $T_c^*$ , however, the relaxation time increases, the dissipative contribution becomes prominent and the electrocliric coefficient saturates.

We have fitted our data of both the amplitude and phase of the electroclinic signal using equations (8) and (9) on the 8th homologue (Figures 10 and 11) and found the critical index  $\gamma = 1.0$  which agrees with the niean field value.

Chapter VI is an extension of the Vth chapter. Here we describe experiments to estimate several of the coefficients of the Landau expansion, using the procedure of Dupont et al. (1991). The main feature of this technique is to make both optical and current measurements in the A\* phase of the ferroelectric samples. The experiment was computer controlled and using appropriate softwares the signals were measured as functions of voltage, frequency and temperature. We measured the frequency dependence of the optical signal in the temperature range of a few degrees above the A-C\* transition temperature to the transition temperature for the compounds D7, D8, D9 and D10. A typical frequency variation is shown in figure 12, for the compound D9; which clearly shows the increase in  $\tau$  as  $T_{A-C*}$  is approached. The data was fitted to a Lorentzian using a non-linear least square fitting programme to calculate the relaxation frequency  $\tau^{-1}$ , which is plotted as a function of temperature in figure 13. The relaxation frequency increases linearly with temperature in accordance with the prediction of the Landau theory.

The electroclinic contribution to the dielectric constant relaxes beyond  $\tau^{-1}$ . It can be shown that by plotting  $\epsilon_{\perp}$  vs. e, the Y-intercept yields ( $\epsilon_o + \epsilon_o \chi_{\infty} - \chi_{\mathcal{P}}$ ) and the slope gives  $c\chi_{\mathbf{P}}$  (figure 15). We can estimate all the Landau coefficients from



**Figure 10..** Divergence of e in the 8th homologue as  $T_c^*$  is approached. The continuous line is the theoretical variation given by equation (5.10).



Figure 11. Temperature variation in the phase angle of the electroclinic signal in the 8th homologue. The continuous line is the theoretical variation given by equation (5.11).



Solid line gives the fitted Lorentzian.



Figure 13. Variation of  $1/\tau$  as a function of temperature for D9.



Figure 14. Variation of current as a function of frequency for the compound D9 at 342 K.



Figure 15. Dielectric permittivity as a function of the EC coefficient for the compound D9.

these measurements. The values of these coefficients which we have calculated for two compounds D9 and D10 are shown in the table.

These numbers are quite reasonable and are similar in order of magnitude when compared with the data on other compounds (Dupont et al., **1991**).

Some of the results presented in this thesis have been published in the following papers:

 Electromechanical effect in cholesteric liquid crystals with fixed boundary conditions.
 N.V.Madhusudana, R. Pratibha and H.P.Padmini Molecular Crystals and Liquid Crystals, 202, 35-49 (1991).

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### Table

Coefficients of 1	the l	Landau	theory
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Coefficients	Compound	
	D9	D10
$\chi_{\mathcal{P}}^{/\epsilon_o}$	11.6	8.5
$\chi_{\mathrm{P}^{c(Cm^{-2}rad^{-1})}}$	$3.73 \times 10^{-4}$	$4.87 \times 10^{-4}$
$c(NC^{-1}rad^{-1})$	$0.36 \times 10^{7}$	$0.65 \times 10^7$
$a(Nm^{-2} \ge K^{-1})$	0.78×10 <sup>4</sup>	$0.78 \times 10^{4}$
$\Delta T_{c}(K)$	1.7	0.4
$\eta \; (Nsm^{-2})$	0.01	0.01

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