

Dynamic Light Scattering and Nonlinear Optics in Liquid Crystals

by

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CERTIFICATE

This is to certify that the thesis entitled “**Dynamic Light Scattering and Nonlinear Optics in Liquid Crystals**” submitted by Amit Kumar Agarwal for the award of the degree of DOCTOR OF PHILOSOPHY of Jawaharlal Nehru University is his original work. This has not been published or submitted to any other University for the award of any other degree or diploma.

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DECLARATION

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Contents

1	Introduction	1
1.1	An overview of liquid crystals	1
1.1.1	The nematic phase	2
1.1.2	The cholesteric phase	4
1.1.3	The smectic phase	5
1.2	Static distortion in the director	8
1.3	Dynamic light scattering	10
1.3.1	Auto correlation function	10
1.3.2	Analysis of correlation function	14
1.3.3	Dynamic light scattering from nematics	16
1.4	Nonlinear optics	20
1.4.1	Nonlinear optics in liquid crystals	22
1.4.1.1	Light induced director reorientation	22
1.4.1.2	Thermal effects	24
2	Dynamic light scattering studies on a lyotropic discotic nematic liquid crystal	29
2.1	Introduction	29
2.2	Theory	31
2.3	Experimental	32
2.3.1	Instrumentation	32
2.3.1.1	The light source	32
2.3.1.2	The digital auto correlator	34
2.3.1.3	The light detector	36
2.3.1.4	The pre-amplifier and discriminator	36
2.3.1.5	The sample oven	36
2.3.2	PID temperature controller	37

2.3.2.1	Features of control program	37
2.3.3	Standard samples	40
2.3.4	Sample preparation	41
2.3.5	Experimental geometry	43
2.4	Results	44
2.5	Discussion	46
2.5.1	Estimation of the activation energy	47
3	The effect of polymer and salt on the viscoelastic modes of a lyotropic discotic nematic liquid crystal	53
3.1	Introduction	53
3.2	Experimental	55
3.3	Results	56
3.3.1	Refractive indices	56
3.3.2	Twist viscoelastic coefficient	56
3.4	Discussion	58
3.4.1	Estimation of activation energy	60
4	Viscoelastic coefficient of novel thiol terminated alkoxy-cyanobiphenyl liquid crystals	67
4.1	Introduction	67
4.2	Experimental	68
4.2.1	Sample cell preparation	69
4.2.2	Instrumentation and experimental geometry	71
4.3	Results	72
4.3.1	Refractive index measurement	72
4.3.2	Twist viscoelastic coefficient	72
4.4	Discussion	77
4.4.1	Estimation of activation energy	79
5	Thermo-nonlinear optics in cholesteric liquid crystals	83
5.1	Introduction	83
5.2	Theory	85
5.2.1	Effect of heating due to light absorption	87
5.2.2	Pitch variation with light intensity	88

5.3	Results and Discussion	89
5.3.1	Large optical nonlinearities in cholesterics	90
5.3.1.1	Rapid variation of pitch with intensity	90
5.3.1.2	Slow variation of pitch with intensity	90
5.3.1.3	Nonlinear optical rotation	93
5.3.2	Finite laser beams	96
5.3.2.1	Self phase modulation	96
5.3.2.2	Spatial solitons	96
5.3.2.3	New beam structures	97
5.3.2.4	Defect lattice in compensated cholesterics	97
5.3.3	Chiral smectic C	98
6	The effects of a laser field on phase transitions in liquid crystals	103
6.1	Introduction	103
6.2	Nematic - isotropic transition	106
6.2.1	Order parameter dependence on intensity	110
6.2.2	Order parameter dependence on temperature	112
6.3	Smectic A - nematic transition	114
6.4	Smectic C - smectic A transition	117
6.5	Cholesteric - isotropic transition	119

List of symbols used in the thesis

\mathbf{n}	Nematic Director
S	Nematic Order Parameter
P	Cholesteric Pitch
q_0	Cholesteric Wave Vector
n_x, n_y, n_z	Components of Director
ψ	Smectic Order Parameter
K_{11}, K_{22} and K_{33}	Splay, Twist and Bend Elastic Constant
\mathbf{i}	Incident Polarization Direction
\mathbf{f}	Scattered Polarization Direction
\mathbf{k}_i	Wavevector of Incident Light
\mathbf{k}_f	Wavevector of Scattered Light
T_t	Total Experimental Run Time
τ	Delay Time
τ_r	Relaxation Time
T	Temperature in $^{\circ}C$
T_A	Absolute Temperature
E	Electric Field of Light
I	Intensity of Light
$g_1(\tau)$	Electric Field Correlation Function
$g_2(\tau)$	Intensity Correlation Function
$\delta\mathbf{n}$	Deviation of Director from the Equilibrium
ϵ_{\parallel}	Dielectric Constant Parallel to the Director
ϵ_{\perp}	Dielectric Constant Perpendicular to the Director
ϵ_a	Dielectric Anisotropy
\mathbf{q}	Scattering Wave Vector
Γ	Relaxation Frequency
η	Viscosity
χ	Dielectric Susceptibility
\mathbf{P}	Induced Polarization
ω	Frequency of Light
n_o	Ordinary Refractive Index
n_e	Extraordinary Refractive Index
δn	Birefringence
\bar{n}	Average Refractive Index
λ	Wavelength of Light
q_{\parallel}	Component of Scattering Wavevector Parallel to Director
q_{\perp}	Component of Scattering Wavevector Perpendicular to Director
E_a	Activation Energy Corresponding to Twist Viscosity
k_B	Boltzmann Constant
K_{22}/η	Twist Viscoelastic Coefficient

N_D	Discotic Nematic Mesophase
ISO	Isotropic Phase
θ	Tilt Order Parameter
E_{app}	Applied Electric Field
ϕ	Scattering Angle
β	Optical Absorption Coefficient
$\bar{\beta}$	Average Optical Absorption Coefficient
β_a	Optical Absorption Anisotropy
d	Sample Thickness
κ	Thermal Conductivity
n_R	Refractive Index for Right Circularly Polarized Light
n_L	Refractive Index for Left Circularly Polarized Light
ρ	Mass Density
c_v	Specific Heat at Constant Volume
ρ_{opt}	Optical Rotatory Power
T^*	Super Cooling Temperature of Isotropic Phase
T^{**}	Super Heating Temperature of Nematic Phase
T_{NI}	Nematic to Isotropic Transition Temperature
T_{AN}	Smectic A to Nematic Transition Temperature
T_{AC}	Smectic C to Smectic A Transition Temperature
A_1, B_1 and C_1	Landau Free Energy Parameters for Nematic Phase
T_0	Temperature of Unilluminated Part of Sample
A_2, B_2 and C_2	Landau Free Energy Parameters for Smectic A Phase
A_3, B_3 and C_3	Landau Free Energy Parameters for Smectic C Phase

List of abbreviations used in the thesis

DLS	Dynamic Light Scattering
ACF	Auto Correlation Function
NLO	Nonlinear Optics
SHG	Second Harmonic Generation
CSPFO	Cesium Perfluoro-octanoate
PMT	Photo Multiplier Tube
ITO	Indium Tin Oxide
PID	Proportional Integral Derivative
RTD	Resistance Temperature Detector
GPIB	General Purpose Interfacing Board
DSC	Differential Scanning Calorimetry
POM	Polarizing Optical Microscope
PEG	Polyethylene Glycol
CsCl	Cesium Chloride
ODSE	Octadecyl Triethoxy Silane
LdG	Landau deGennes
N-I	Nematic to Isotropic
A-N	Smectic A to Nematic
C-A	Smectic C to Smectic A

Preface

This thesis deals with the dynamic light scattering and nonlinear optical studies of liquid crystals. Liquid crystals are “soft” materials. They possess the properties of liquids as well as crystals. For example, they flow like a liquid, while on the other hand they always have some degree of ordering in their molecular arrangement. Their response to an external electric or magnetic field finds many technological applications. Liquid crystals are classified into two types depending on the phase inducing factors.

- Lyotropic liquid crystals: In lyotropic liquid crystals, the ordering is mainly induced by the concentration of amphiphilic molecules in a solvent. In these liquid crystals, phase can be changed by changing the concentration of solute in a solvent, as well as the temperature of the system.
- Thermotropic liquid crystals: In thermotropic liquid crystals, phase transitions are brought about by change of temperature of the system.

In this thesis we deal with the investigations on both lyotropic and thermotropic liquid crystals. Chapter 1 gives an introduction to liquid crystals and a brief overview of dynamic light scattering. We briefly describe the nonlinear optics of liquid crystals in general.

The simplest liquid crystal phase is known as *nematic*. In nematics the molecules have long range orientational order and have no positional order. The average direction of molecular orientation is called the director and is denoted by an apolar vector \mathbf{n} . If the constituent molecules are chiral or a nematic is doped with some chiral molecules then, the director uniformly rotates in space, about a helical axis perpendicular to the director. The resultant helix has a pitch given by a 2π rotation of the director. Such liquid crystalline phases are known as *cholesterics*. A relatively more ordered liquid crystalline phase is the one where molecules are arranged in layers, known as *smectic* phase. In such phases, there is one dimensional positional ordering in addition to orientational order. There are many smectic phases. For

instance, when director is parallel to the layer normal then the phase is called *smectic A* phase and when the director makes an angle with the layer normal, it is called *smectic C* phase. If the director is not only tilted with respect to layer normal but it also precesses uniformly about the layer normal then the phase is known as *smectic C**. Apart from these, there are some other types of smectic phases. There are also some interesting systems like discotic phases, twist grain boundary phases and blue phases.

The interaction between the molecules of a liquid crystal is very weak. This is the reason for liquid crystals to have long wavelength thermal fluctuations even at room temperatures. Because of these fluctuations, the dielectric tensor of the medium also fluctuates and this results in the intense scattering of light. In fact, it is found that the intensity of scattered light in the liquid crystalline phase is about 10^6 times high compared to the intensity of light scattered by same material in the isotropic phase. By analysing the scattered light in properly chosen scattering geometries, one can obtain information about the viscoelastic coefficients of a liquid crystal. Viscoelastic coefficient is a material property, given by the ratio of elastic constant to corresponding viscosity coefficient. It is an important property to understand the switching characteristics of a display device. In chapter 1, we have briefly reviewed the theory of light scattering. This theory is useful to analyse our results on dynamic light scattering. Also, we have presented some basic nonlinear optics in context of liquid crystals. The interest in nonlinear optics of liquid crystals is mainly due to the observation of very large value of nonlinear optical coefficients. Liquid crystals possess dielectric anisotropy which can lead to a torque on the director in the presence of the electric field of a laser beam. This can result in the change in refractive index of the medium. In fact, the change in refractive index can be so high that the corresponding nonlinear optical coefficient is huge compared to other ordinary nonlinear materials.

In chapter 2, we have described our experimental apparatus including a temperature controller, interfaced to a computer *via* a general purpose interfacing board. We have employed dynamic light scattering technique to measure the twist viscoelastic coefficient of a lyotropic discotic nematic liquid crystal. We have taken Cesium perfluorooctanoate (CSPFO) which

forms disk like micelles when dissolved in water over a range of concentrations. These micelles arrange themselves in nematic order over a range of temperatures. A polarizing microscope texture of an unaligned nematic phase of CSPFO water system is shown in Fig. 1. The dynamic light scattering was carried out using a Malvern 4700C digital photon correlator. This photon correlator gives the intensity auto correlation function of the scattered light. A typical auto correlation function obtained from our experiments is shown in Fig. 2.



Figure 1: A polarizing microscope texture of CSPFO water system in the nematic (N_D) phase. The magnification was $200\times$ and sample was kept between crossed polarizers.

The relaxation frequency evaluated from the auto correlation function is related to the twist viscoelastic coefficient. We have systematically studied the variation of twist viscoelastic coefficient in the nematic phase of CSPFO water system as a function of temperature and concentration. We find that the twist viscoelastic coefficient increases with increase in temperature as shown in Fig. 3. As we increase the concentration of CSPFO in water, the twist viscoelastic coefficient at a given reduced temperature increases rapidly at higher concentrations. We have estimated the activation energy corresponding to twist viscosity. We find that activation energy is independent of the concentration of CSPFO in water.

The addition of dopants to a lyotropic system is important from the point of view of Bi-

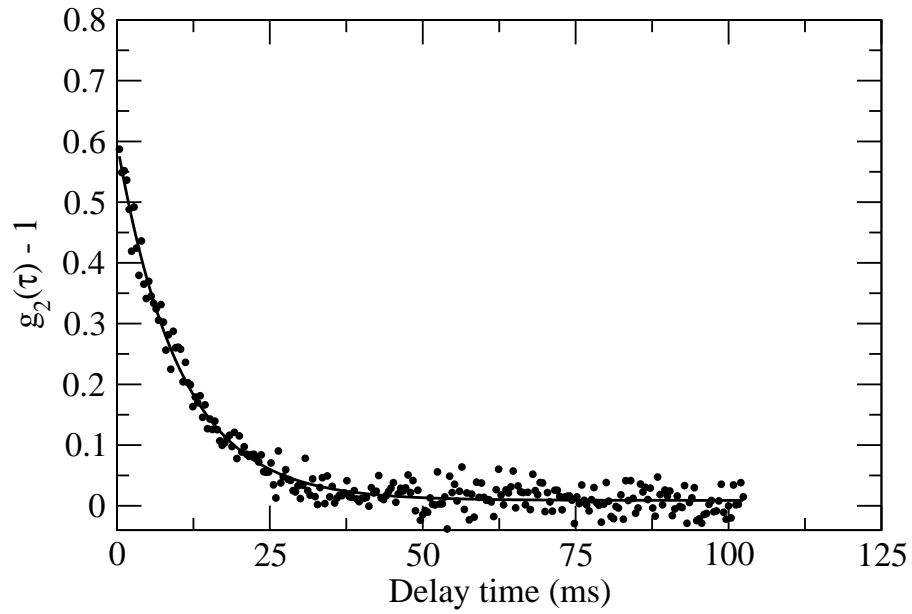


Figure 2: A typical auto correlation function obtained from our experiment for 45 wt% of CSPFO in water at 34.0°C . The scattering angle was 17.7° . Solid line is a single exponential fit. The value of relaxation frequency evaluated from the fit is 48.86Hz

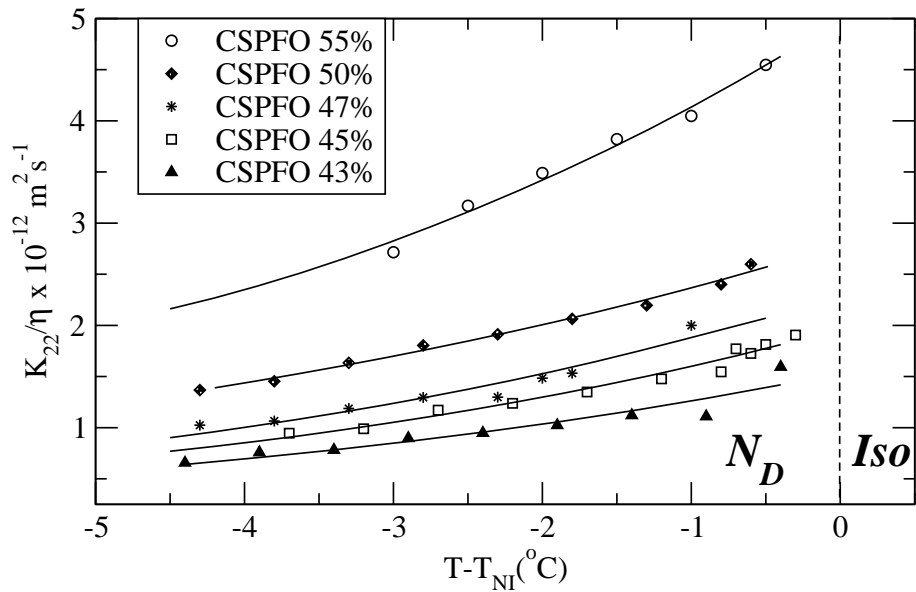


Figure 3: The variation of twist viscoelastic coefficient as a function of reduced temperature at various concentrations of CSPFO in water. Solid lines are cue to the eyes.

ology. Small quantities of polymers or salts in a lyotropic liquid crystal can mimic some biological phenomena that have relevance to applications. In chapter 3, we have studied the effects of small quantities of polymer as well as salt on the twist viscoelastic coefficient of a lyotropic discotic nematic liquid crystal. The presence of polymer above a certain critical concentration in CSPFO water system is known to suppress the lamellar phase. Our experiments show that the twist viscoelastic coefficient increases with an increase in the concentration of polymer. In the same system, the addition of salt reduces the micelle-micelle interaction giving flat longer aggregates which results in the suppression of the nematic phase. We have used Cesium chloride salt, having the same cation as that of CSPFO, as a dopant. We find that the viscoelastic coefficient decreases as we increase the concentration of salt in CSPFO water system. As a function of temperature, the twist viscoelastic coefficient increases in both the cases of polymer or salt doped liquid crystal. From our experimental data, we have estimated the activation energy corresponding to twist viscosity. We find that it increases with increase in concentration of polymer or salt dissolved in CSPFO water system.

In chapter 4, we have described the studies on the viscoelastic modes of novel thermotropic liquid crystals. The system studied was thiol terminated alkoxy-cyanobiphenyl liquid crystal. This was synthesized in our chemistry laboratory for the first time. The lower homologues of this molecule showed stable nematic phase near room temperature. Fig. 4 shows the structure of this molecule. We have studied the molecules having 5, 6, 7 and 8 carbon atoms in the alkoxy chain. We have measured the ordinary and extraordinary refractive indices using an Abbe refractometer and compared them with that of the well studied 4'-(hexyloxy)-4-biphenyl carbonitrile (6OCB) liquid crystal. This is shown in Fig. 5. The structure of 6OCB is same as that of thiol terminated alkoxy-cyanobiphenyl but it does not have a thiol group at the end. The twist viscoelastic coefficient was studied as a function of temperature and the length of alkoxy chain. We find that it increases as we increase the temperature in the nematic phase. The length of alkoxy chain has little effect on the viscoelastic coefficient. We have compared the values of twist viscoelastic coefficient with that of alkoxy cyanobiphenyl system (Fig. 6). We find that the values of twist viscoelastic coefficient are

smaller than that of alkoxy cyanobiphenyl systems. We suggest that the presence of bulky thiol group at the end of alkoxy chain increases the director relaxation time thereby decreasing the twist viscoelastic coefficient. We have estimated the activation energy corresponding to twist viscosity and find that the values are comparable to that of other rod like nematics. We have carried out dynamic light scattering experiments with applied electric field on the homeotropically aligned sample. From such experiments one can find the values of twist viscosity and elastic constant separately.

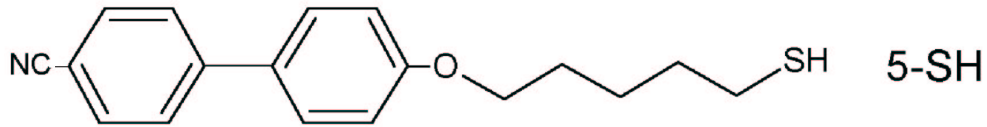


Figure 4: Structure of thiol terminated alkoxy-cyanobiphenyl molecule. We have named these molecules as nSH, where n is the number of carbon atoms in the alkoxy chain.

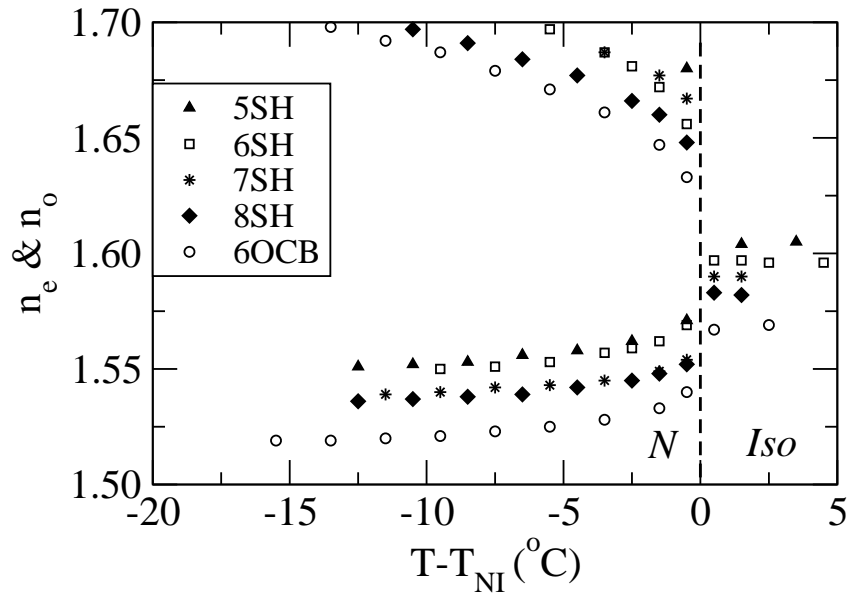


Figure 5: The variation of n_e and n_o for thiol-terminated alkoxy-cyanobiphenyl molecules as a function of reduced temperature for different lengths of alkoxy chains. For comparison we have plotted the values of n_e and n_o for 6OCB (open circles).

In dynamic light scattering experiments, we have used a low power laser source. The laser was just used as a probe beam. But, liquid crystals are known to have high nonlinearity.

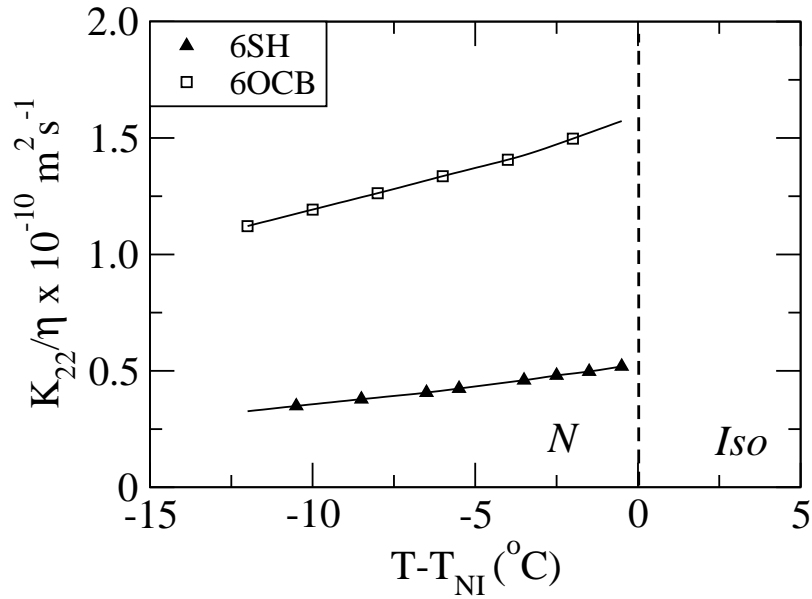


Figure 6: The twist viscoelastic coefficient of 6OCB (empty squares) and 6SH (filled triangle) as a function of reduced temperature. The values of K_{22}/η are 3 – 4 times higher in case of 6OCB. Solid lines are cue to the eyes.

A moderately intense laser beam can induce many interesting nonlinear effects in liquid crystals. In chapter 5, we describe our studies on thermal nonlinearities in absorbing cholesteric liquid crystals. Our computations show that in a right (left) handed cholesteric the nonlinear optical (NLO) coefficient ($\partial n/\partial I$) for the right (left) circular wave is more than a billion times greater than that found in ordinary nonlinear materials. It is about $10^{-4} \text{ cm}^2/W$ as compared to $10^{-14} \text{ cm}^2/W$ in case of usual nonlinear materials. It increases in magnitude as we approach the reflection band in the cholesteric phase. For the same structure, the left (right) circular wave has a nonlinear optical coefficient of the order of $10^{-6} \text{ cm}^2/W$, which is more than a million times greater than that found in the usual nonlinear materials. It monotonically increases when the pitch increases with increasing laser intensity. Further, when the pitch decreases with increasing laser intensity, it shows a smooth saturation behavior. The NLO coefficient for both the right and left circular waves is positive when the pitch increases with increase in laser intensity and negative when pitch decreases with increase in laser intensity. When the principle refractive indices vary with laser intensity then we find that for both the circular waves, the NLO coefficient is of the order of $10^{-5} \text{ cm}^2/W$. Further, the NLO coeffi-

cient even changes sign for the right (left) circular wave as we approach the reflection band. In the case of laser beams with a Gaussian intensity profile, we get a gradient in the pitch of the cholesteric across the beam. If the pitch variation is small compared to the intrinsic pitch then we find that in the de Vries limit both right and left circular states have a refractive index profile across the wavefront. This leads to self focusing and self divergence. On the other hand, in the Mauguin limit the base states are linear vibrations along and perpendicular to the local director. Here, the pitch variation with intensity can be very large. For the vibration parallel to the local director there is no torque on the director due to the electric vector of the laser beam. In such cases, we find a defect structure with a periodic array of disclination loops within the beam. We show that new beam structures can be generated by using the selective reflection of cholesteric liquid crystals.

If the laser intensity is very high, it will not only heat the system due to absorption but also change the order parameter of the liquid crystal. This leads to many interesting phase transitions in liquid crystals. In chapter 6, we have theoretically worked out the phase transitions in some liquid crystals in the presence of a laser field. A laser field can have two effects on the medium. One is due to the electric field of a laser beam which plays an important role if the intensity is high enough. Another is the absorption of the laser beam which increases the temperature of the medium. The electric field effect of a laser beam is equivalent to applying a low frequency electric field on liquid crystal sample except for the fact that dielectric anisotropy is low at optical frequencies. A high intensity laser beam can change the order parameter of a liquid crystal. On the other hand, absorption increases the temperature of the medium and hence decreases the order parameter. We consider the effects of these two parameters (electric field and absorption) on the phase transitions in liquid crystals. We generalize the Landau deGennes free energy density of a homogeneously aligned nematic sample where the polarization of incident laser beam is parallel to the director. In such a geometry, there is no dielectric torque due to the electric field of the laser beam on the director. The nematic to isotropic phase transition is a first order transition in the absence of any applied field. There is a small range of temperature in which both nematic and isotropic

phases can coexist with one of them being metastable. In the presence of an external field there exists a paranematic phase instead of an isotropic phase. This can also happen due to the electric field of a laser beam. In the absence of light absorption, if we start with a paranematic phase, then we find a one way first order phase transition from a paranematic to a nematic phase with increase of laser intensity. However, if we start with a nematic phase it continues to be in that phase as the laser intensity is increased. On the other hand, when the system is absorbing, even the nematic phase can go over to a paranematic phase *via* a first order phase transition. We find an interesting case where a nematic (paranematic) goes to a paranematic (nematic) and then to a nematic (paranematic) phase with increase of laser intensity. This is similar to a reentrant nematic (paranematic) phase *via* a paranematic (nematic). In smectic liquid crystals, a coupling between layering and orientational order can arise due to absorption anisotropy in the medium. Consequently, the nature of smectic A to nematic phase transition can change. In a particular geometry, smectic C to smectic A phase transition can change from a second order to a first order in the presence of a laser beam. In the case of cholesterics, we have considered the effect of high laser intensities on the cholesteric to isotropic phase transition.

During the course of this thesis we have published the following papers and some manuscripts are under preparation.

- Thermal Indexing in Cholesteric Liquid Crystals
Amit K Agarwal and G S Ranganath
Mol. Cryst. Liq. Cryst., vol. **437**, pp. 239/[1523] (2005)
- Dynamic Light Scattering Studies on a Lyotropic Discotic Nematic Liquid Crystal
Amit K Agarwal and K A Suresh
Proceedings of SPIE, vol. **5947**, pp. 59470A (2005)
- Structure and Dynamics in Solvent-Polarity-Induced Aggregates from a C_{60} Fullerene-Based Dyad
S S Gayathri, Amit K Agarwal, K A Suresh and Archita Patnaik

Langmuir, vol. **21**, pp. 12139 (2005)

- The Effects of a Laser Field on Phase Transitions in Liquid Crystals

Amit K Agarwal and G S Ranganath

Phase Transitions, vol. **79**, pp. 261 (2006)

- The Effects of Dopants on the Viscoelastic Modes of Lyotropic Discotic Nematic Liquid Crystals

Amit K Agarwal and K A Suresh (Manuscript under preparation)

- Twist Viscoelastic Coefficient of Novel Thiol Terminated Alkoxy-cyanobiphenyl Liquid Crystals

Amit K Agarwal, K A Suresh, S Kumar and Santanu K Pal (Manuscript under preparation)