

# Miscibility Study of Nematic Liquid Crystal with Cholesteric Liquid crystal

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**Abstract:** Liquid crystals are characterized by high molecular orientation order in their mesomorphic states. The mixture of liquid crystals influences the molecular orientation of liquid crystal compound consequently affecting their physical properties. In this paper, the main focus is on the study of the mixtures of Nematic liquid crystal and Cholesteric liquid crystal in different proportions. We have used Differential scanning calorimetry (DSC) technique to measure the Phase transition temperatures. Fourier Transform Infrared Spectroscopy (FTIR) and Ultraviolet-Visible (UV) spectroscopy are used to analyze the relative concentration of liquid crystals in the mixtures and their spatial distribution. The values of pitch for Cholesteric liquid crystal and the mixtures were calculated from ordinary refractive indices. Because of its low phase transition temperatures and less requirement of energy, the mixture of Cholesteric chloride with Cholesteryl oleate in 30%+70% proportion holds promise in the optical as well as non-optical applications of liquid crystals.

**Keywords:** Liquid Crystal (LC), Differential scanning calorimetry (DSC), Fourier Transform Infrared Spectroscopy (FTIR), Ultraviolet Visible (UV) Spectroscopy, Pitch.

## I. INTRODUCTION

Liquid crystals have become a consumer product with many everyday applications. A liquid crystal is a mesophase which has partially or completely lost the long range order of ordinary crystals, but still possesses one or more dimensional long range orientational order of certain anisometric units. [1], [2],[3] The mixture of liquid crystals bring the melting point down which helps or gives wide applicability of its use in various applications. These mixtures show phase transition temperatures and other physical properties which are different from their constituents.[4],[5]. In the present study the main focus is on the miscibility of Nematic liquid crystal and Cholesteric liquid crystal. Arnold,Sackman and Demus developed the miscibility rules which can be summarized as follows. [6], [7], [8], [9].

- i) If two LCs are miscible, they are isomorphic and therefore belong to the same type of mesophase,
- ii) If two LCs are isomorphic, they need not be necessarily miscible.

When two compounds are isomorphic within a certain mesophase, both their thermal transition temperatures and corresponding thermodynamic parameters exhibit continuous dependence on their components. This means that both the components of mixtures behave like an ideal solution. Therefore, by knowing the transition temperatures and thermodynamic parameters of the parent compounds, we can apply the equations of Schroeder and Van Lar (equation 1) to predict the phase diagram of the mixtures.

$$F_1 = \left\{ 1 - \frac{\Delta H_1^0 T_2 (T - T_1)}{\Delta H_2^0 T_1 (T - T_2)} \right\} \dots \dots \dots \text{Equation 1}$$

$F_1$  = Molar fraction of components

$T_1^0$  C and  $\Delta H_1^0$  are the transition temperature and enthalpy change for pure component A.

$T_2^0$  C and  $\Delta H_2^0$  are the transition temperature and enthalpy change for pure component B.

$T^0$  C is the transition temperature corresponding to the molar fraction  $F_1$ .

$T^0$  C should be lower than  $T_2^0$  C and higher than  $T_1^0$  C. ( $T_1^0$  C <  $T^0$  C <  $T_2^0$  C)

A phase diagram that exhibits a higher or lower transition temperatures than the maximum or minimum  $T_2^0$  C or  $T_1^0$  C respectively violates the temperature criteria. The negative, positive or linear dependence of  $T^0$  vs  $F_1$  is determined by the parameter X from equation 1 where  $X = \Delta H_1^0 T_2 / \Delta H_2^0 T_1$

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## II. RELATED WORK

In studying liquids crystals, we often want to expand the range of certain phases so the phase is more stable.[11] We also to alter the physical properties such as viscosity. An easy way to do this is to dope the liquid crystal with another liquid crystal.[12]The mixtures help in realignment of the liquid crystal molecules which can have a large effect on the electrooptical properties of a liquid crystal.[13],[14] .The previous researchers studied various properties of the liquid crystal mixtures using different techniques. V.S,Chandel,A.K.Singh,S.Manohar,J.P.Shukla and R.Manohar[15] studied phase transitions in binary mixture of Cholesteric liquid crystals. Their main focus is on different phases of liquid crystal mixtures. They not only observed new phases in the mixtures but also found higher thermal stability in the mixtures. Authors [16] measured dielectric, refractive, elastic and viscous properties of the HBLCMs.The mixtures measured high optical and dielectric anisotropies and relatively low rotational viscosities. This has applications in different kinds of liquid crystal electro-optical devices with low response times, operating in visible and near infrared regions. PK Mukherjee[17] studied the mixtures of HBAB  $\{p\text{-}[(p\text{-hexyloxy-benzylidene)-amino] benzonitrile\}$  and CBOOA [*N-p-cyanibenzylidene-p-n-octyloxyaniline*].The mixtures exhibited a two phase region where both smectic-A and nematic phase coexists. is discussed by means of Landau formalism. The problem of the first or second order nature of the nematic-smectic-A phase transition is explored. General Landau theory for coupled orientational, translational order parameters and concentration is developed by the author to discuss the reason for this two phase region. Authors [18] formulated two exemplary high birefringence and low clearing temperature Liquid crystal mixtures, using the laterally substituted isothiocyanato tolane compounds. They observed decrease in the melting point (below 20<sup>0</sup>C) in the mixtures. The temperature derivative of the mixtures was enhanced in the mixtures.

## III. LIST OF CHEMICALS AND SAMPLE PREPARATION

The present study was done using Nematic liquid crystal, Cholesteric liquid crystal and their mixtures in four different proportions. (Both the samples were procured from ALDRICH)

Sample A: Name of the sample: Cholesteryl oleate (Nematic phase)

Molecular formula: C<sub>45</sub>H<sub>78</sub>O<sub>2</sub>; Melting point: 48<sup>0</sup>C.

Sample B: Name of the sample: Cholesteryl chloride (Cholesteric phase)

Molecular formula: C<sub>27</sub>H<sub>45</sub>C<sub>11</sub>; Melting point: 96<sup>0</sup>C.

Mixtures of the samples were prepared in the different proportions.

5A + 5B: 50% of sample A & 50% of sample B

4A + 6B: 40% of sample A & 60% of sample B

3A + 7B: 30% of sample A & 70% of sample B

2A + 8B: 20% of sample A & 80% of sample B

The two samples were first weighed accurately using analytical microbalance in the required proportions. Then the mixture was stirred enough to ensure thorough and complete mixing. A small amount of this homogeneous mixture in powder form was sandwiched between two cover slips and the sides were sealed by non conducting tape. When the sample slide is heated, at a particular temperature the sample melts and spreads uniformly between the bottom and top cover slip.

## IV. EXPERIMENTAL TECHNIQUES

The essential tools used in present study are Differential scanning calorimetry (DSC), Fourier Transform Infrared Spectroscopy (FTIR), Ultraviolet Visible (UV) Spectroscopy and ordinary refractive indices using multiwavelength Abbe refractometer

1 Differential scanning calorimetry (DSC)

DSC is a thermoanalytical technique in which the difference in the amount of heat required to increase the temperature of the sample and reference is measured as a function of temperature. Both the sample and reference are maintained

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nearly at the same temperature throughout the experiment. The temperature of the sample holder increases linearly as a function of time. Even small changes in thermodynamic quantities at transitions between mesophases can be detected by DSC. The result of a DSC experiment is a curve of heat flux versus temperature or versus time. Most liquid crystal to liquid crystal transitions are discontinuous. Such transitions are revealed by larger peaks.

## 2 Fourier Transform Infrared Spectroscopy (FTIR)

FTIR is a powerful tool for identifying types of chemical bonds in a molecule by producing an infrared absorption spectrum that is like a molecular fingerprint. The FTIR spectroscopy detects the vibration characteristics of chemical functional groups in the sample.[10] When infrared light interacts with matter, chemical bonds will stretch, contract and bend. The chemical functional group absorbs IR radiation in a specific wave number range. It is a chemical analytical technique that measures the infrared intensity vs wavelength of light. The IR is divided into three regions i.e. far infrared ( 4 – 400 cm<sup>-1</sup> ), mid infrared ( 400 – 4000 cm<sup>-1</sup> ) and near infrared (4000 – 14000 cm<sup>-1</sup> ) It can be applied to the analysis of types of chemical bonds i.e. functional groups. IR spectroscopy works because chemical bonds have specific frequencies at which they vibrate corresponding to energy levels. From the recorded FTIR spectra various functional groups of different samples can be identified.

## 3 Ultraviolet Visible (UV) Spectroscopy

UV spectroscopy is an accurate and powerful procedure to analyze a substance. It measures the absorption, transmission and emission of ultraviolet and visible light by matter. Absorption of ultraviolet or visible light causes electron to move from lower to higher energy levels. Because the spectrum of an atom or molecule depends on its electron density level, it is useful for identifying unknown substances.

## 4 Determination of pitch from RI

Liquid crystals are birefringent in nature. When the light enters liquid crystals it splits into an ordinary ray and an extraordinary ray. The ordinary ray moves faster than the extraordinary ray. The cholesteric liquid crystals consist of thin birefringent layers normal to the optic axis and each one is turned through a small angle with respect to its neighbours. Because of this turn each linear component of the light experiences a change in refractive index while passing from one layer to the next. The changes will be an increase for the fast and a decrease for the slow component. By measuring the refractive index of ordinary ray ( $n_o$ ) for the given wavelength of light ( $\lambda$ ), pitch (P) of the Cholesteric liquid crystal and the mixtures can be calculated using the relation,

$$\lambda = P n_o \dots\dots\dots \text{Equation 2}$$

If d is the spacing between thin birefringent layers of the cholesteric liquid crystals then

$$\lambda = 2 d n_o \dots\dots\dots \text{Equation 3}$$

V. RESULTS AND DISCUSSION

1 Differential scanning calorimetry (DSC)

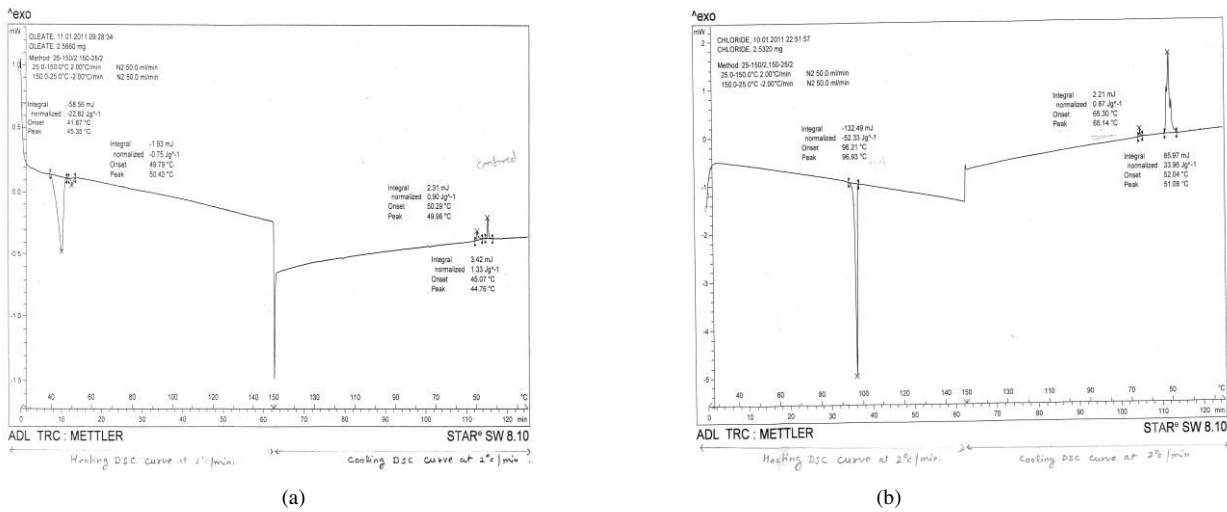


Fig.1. DSC Thermograph of (a) sample A (b) sample B

The fig 1. (a) and 1. (b) show the DSC thermographs for pure Cholesteryl Oleate and pure Cholesteryl Chloride respectively. The fig 1.a indicates the peaks at 45.35<sup>o</sup> C,50.43<sup>o</sup> C while heating and at 49.96<sup>o</sup> C,44.76<sup>o</sup> C while cooling. These are the phase transition temperatures for Cholesteryl Oleate. Similarly from fig 1.b 96.95<sup>o</sup> C,65.14<sup>o</sup> C,51.08<sup>o</sup> C are the phase transition temperatures(PTT) for Cholesteryl Chloride. Similarly the phase transition temperatures for all the mixtures are measured from their respective DSC thermographs. The phase transition temperatures for A,B and their mixtures are presented table 1.The phase transition temperatures which are different than the pure Cholesteryl Oleate or pure Cholesteryl Chloride are indicated by the \* on the top of the value. Also there is an increase in the value of heat absorbed for the mixtures.

Table 1

Sample A		Sample B		Sample5A+5 B		Sample4A+6B		Sample 3A+7B		Sample2A+8 B	
Peak °C	Heat Jg <sup>-1</sup>	Peak °C	Heat Jg <sup>-1</sup>	Peak °C	Heat Jg <sup>-1</sup>	Peak °C	Heat Jg <sup>-1</sup>	Peak °C	Heat Jg <sup>-1</sup>	Peak °C	Heat Jg <sup>-1</sup>
Heating											
45.35	-22.82	96.95	-52.33	40.15*	-4.18	38.19*	-5.55	39.78*	-4.90	39.68*	-1.17
50.43	-0.75			47.94*	-0.28	48.02*	-0.36	47.88*	-0.2479	47.81*	-0.12
				95.35*	-0.31	93.07*	-2.66	91.83*	-6.05	96.77	-18.77
Cooling											
49.96	0.90	65.14	0.87	50.57	0.82	49.53	0.14	54.93*	0.92	58.30*	0.87
44.76	1.33	51.08	33.96							40*	13.27

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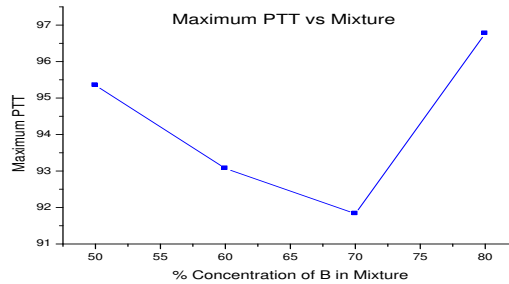


Fig.2 Maximum PTT in °C Vs Concentration of B in Mixture (Maximum PTT: A = 50.43°C, B = 96.95°C)

Fig.2 indicates the values of maximum PTTs for all the mixtures. From the figure it is clear that the maximum phase transition temperature reaches minimum for the mixture 3A+7B. In the mixture of Cholesteryl Oleate and Cholesteryl Chloride, Chloride imparts optical activity to Oleate. The helix undergoes distortion thereby changing the pitch, resulting in the phase change. The maximum PTT for 5A+5B, 4A+6B, 3A+7B, 2A+8B is less than that for the sample B. The maximum PTT for Sample B is 96.95°C which reduces to 91.83°C in the mixture 3A+7B.

## 2 Fourier Transform Infra-red Spectroscopy (FTIR)

The fig.3 (a) and 3(b) show the FTIR graphs for pure Cholesteryl Oleate and the mixture 2A+8B respectively. From similar such FTIR graphs for sample B, 5A+5B, 4A+6B and 3A+7B, the values of the wavelengths with intensities for A, B, 5A+5B, 4A+6B, 3A+7B, 2A+8B are measured.

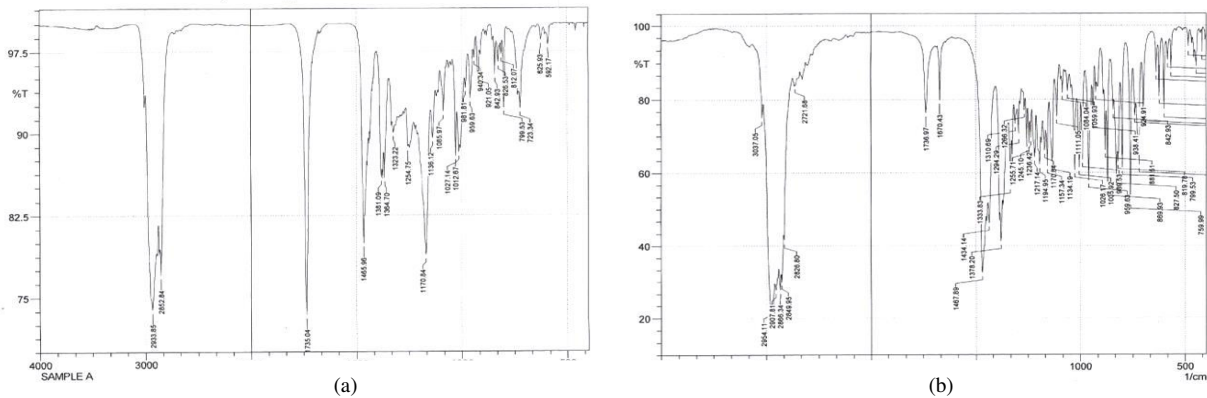


Fig.3: FTIR of (a) sample A, (b) sample 2A+8B

The wavelengths for different mixtures correspond to the wavelengths found either in sample A or in sample B or in both. The wavelengths which are found in all the mixtures are presented in table 2. The corresponding functional groups are identified and are also presented in the same table.

Table 2.: Wavelength, Intensity and Functional group of A, B, 5A+5B, 4A+6B, 3A+7B, 2A+8B

	Wavelength cm <sup>-1</sup>	Intensity for A	Intensity for B	Intensity for 5A+5B	Intensity for 4A+6B	Intensity for 3A+7B	Intensity for 2A+8B	Functional group
1	622.07		89.87980	90.62116	83.95854	75.13262	80.77960	Alkyl Halide
2	698.26		86.36297	86.43396	79.49562	68.64833	75.91160	
3	799.53	92.28832	76.36168	75.96584	67.87760	52.91806	61.44698	

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4	827.5		73.10736	78.79862	68.98070	54.37551	59.04991	Alkene
5	842.93	95.31636	–	85.65219	78.79334	68.54327	76.85237	
6	869.93		65.83800	80.22726	69.30085	54.71814	55.69348	
7	881.51		78.11814	84.63236	74.89840	63.05371	66.08648	
8	924.91		93.00774	87.74038	82.42069	73.01078	81.80713	
9	959.63	93.14064	–	74.58066	66.10171	50.64335	57.88395	Alcohol
10	1005.92		79.82349	72.45523	66.02169	50.63188	61.38835	
11	1111.05		89.84302	83.09068	77.40827	65.78659	75.28553	
12	1170.84	78.866666	89.11001	63.24916	63.03365	46.88258	65.99710	
13	1217.14		81.58184	74.73960	68.23200	54.28905	64.14933	
14	1245.1		86.06778	74.48194	69.35154	55.73544	67.94371	Ester
15	1255.71		85.93209	74.73650	68.98513	55.71534	67.02671	
16	1294.29		86.78953	78.93704	72.75366	60.34057	69.98025	
17	1333.83		74.74161	72.50887	64.36438	49.37273	56.36189	
18	1378.2		58.44308	58.65336	52.07526	34.20830	41.11425	
								Alkyl Halide

From Table 2 we can conclude that though the wavelengths are common, the intensities of absorption for the mixtures are different. This is because the sample molecules selectively absorb radiation of specific wavelengths which causes the change in the dipole moment. Consequently, the vibrational energy levels of sample molecules transfer from ground state to excited state. The frequency of the absorption peak is determined by the vibrational energy gap. The number of absorption peaks is related to the number of vibrational freedom of the molecule. The intensity of absorption peaks is related to the change of dipole moment and the possibility of the transition of energy levels. So the less value of the intensity of absorption indicates the stability in the structure. The mixture 3A+7B has less value of the intensity of absorption. This indicates that the mixture 3A+7B has more stability amongst all the mixtures of A and B.

When a liquid crystal molecule absorbs IR, its chemical bonds vibrate, the bonds can stretch, contract and bend. From the FTIR graphs for sample A, B, 5A+5B, 4A+6B and 3A+7B, 2A+8B the values of the stretching in the wavelengths are measured and indicated in table 3. The corresponding bonds are also given in the same table.

Table 3: Stretching between wavelengths and bond present in 5A+5B, 4A+6B, 3A+7B, 2A+8B

Sample	Stretching between wavelengths cm <sup>-1</sup>	Bond present
5A+5B	1735.04 cm <sup>-1</sup> and 2851.33 cm <sup>-1</sup>	C=C
4A+6B	1735.04 cm <sup>-1</sup> and 2850.91 cm <sup>-1</sup>	C=C
3A+7B	1735.04 cm <sup>-1</sup> and 2851.88 cm <sup>-1</sup>	C=C
2A+8B	1736.97 cm <sup>-1</sup> and 2849.95 cm <sup>-1</sup>	C=C

From table 3 we can conclude that the bond present in mixtures 5A+5B, 4A+6B, 3A+7B and 2A+8B is C=C.

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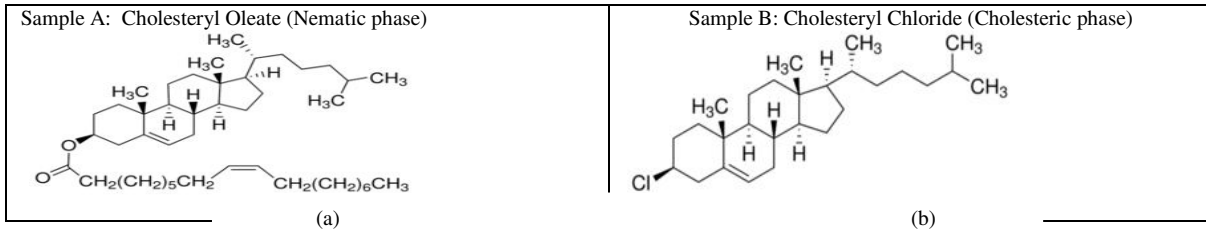


Fig4: Bond line formulae for (a) A, (b) B

The bond line formula for A as shown in Fig4 (a) indicates the presence of double bond(C=C),Alkenes and Easter(moity).Similarly the bond line formula for B indicates the presence of Halide(Chloride) and double bond(C=C). The double bond(C=C) is present in both the samples A and B which is also present in the mixtures as indicated in Table3.This confirms the miscibility of the two liquid crystals under study.

### 3 Ultraviolet – Visible (UV) Spectroscopy

The fig5 (a) and 5(b) show the UV for pure Cholesteryl Oleate and the mixture 5A+5B respectively.

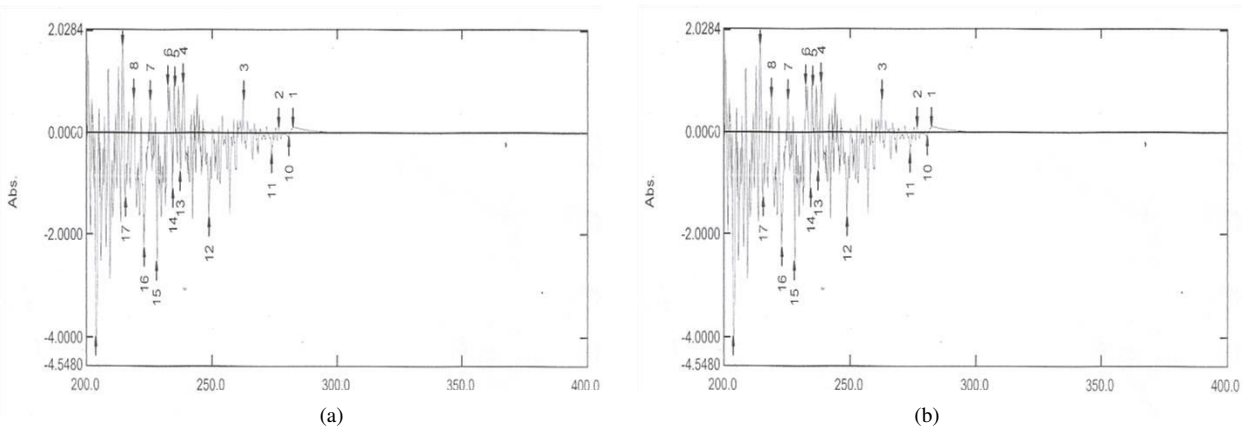


Fig.5: UV of sample (a) A, (b) 5A+5B

The measurements of the absorbance peak and the corresponding wavelength range from UVs of A, B, 5A+5B,4A+6B, 3A+7B and 2A+8B are presented in table 4.

Table 4: Absorbance peak and Wavelength for A, B, 5A+5B, 4A+6B, 3A+7B, 2A+8B (λ: Wavelength, Abs: Absorbance)

Sample A		Sample B		Sample 5A+5B		Sample 4A+6B		Sample 3A+7B		Sample 2A+8B	
λ	Abs	λ	Abs	λ	Abs	λ	Abs	λ	Abs	λ	Abs
282.2	0.1133	276.8	0.3401	279.8	0.2623	283	0.3497	282.2	0.2114	282.8	0.1376
276.4	0.1179	256	0.5596	264.6	0.7092	279.4	0.1924	274.4	0.4022	265	0.4974
262.4	0.6642	244.6	1.0548	245.8	0.5092	276.2	0.1985	265.6	0.4429	253.4	0.6523
238.6	1.0006	240.4	1.9052	243	0.5905	266.4	0.5728	249.4	0.5349	246	1.1259
235	0.9257	229.8	1.1094	233	1.2798	261	0.2984	235.8	1.2412	240.6	0.5023
232.4	0.9540			220.2	1.7083	256.6	0.0865	221.	0.9436	233	0.6452
225.2	0.6710			208.4	1.7689	254.4	0.2718	205	0.7672	214.8	1.0292

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218.8	0.6801					250.2	0.5359			209.2	0.6941
214.2	1.7340					245.4	0.8767			206.6	1.4804
						241.6	1.1084			202.8	0.2238
						225.8	1.6348				

Table 5 gives the wavelength range and the wavelength of maximum absorption for A, B, 5A+5B, 4A+6B, 3A+7B and 2A+8B.

Table 5: Wavelength range and longest wavelength for A, B, 5A+5B, 4A+6B, 3A+7B, 2A+8B

	Wavelength range of absorption (nm)	Longest wavelength of absorption (nm)
A	214.2 - 282.2	214.2
B	229.8 - 276.8	240.4
5A+5B	208.4 - 279.8	208.4
4A+6B	225.8 - 283	225.8
3A+7B	205 - 282.2	235.8
2a+8B	202.8 - 282.8	206.6

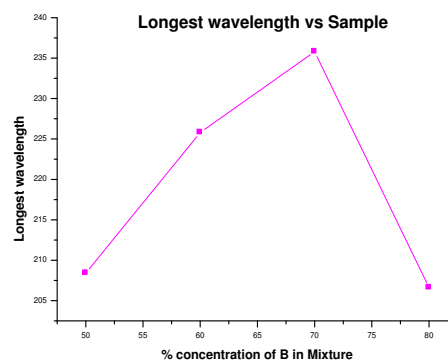


Fig.6: Longest Wavelength vs Concentration of B in Mixture

When light passes through the sample, energy from the light is used to promote an electron from a bonding or non-bonding orbital into one of the empty anti-bonding orbitals. Since the wavelength range of absorption for all samples is between 200nm to 300nm, the molecule must contain either pi- bonds or atoms with non-bonding orbital. Similarly highly delocalized systems in a molecule are often responsible for absorbing light in the visible region. The absorption over a range of wavelengths rather than at fixed one suggests the continuous change in the gaps between the orbitals. This is because of the rotations and vibrations in the molecule are continuously changing the energies of the orbitals. This greater delocalization lowers the energy gap between the highest occupied molecular orbital and the lowest unoccupied pi -anti-bonding orbital. Because of which it needs less energy to promote an electron and so a longer wavelength of light is absorbed. Fig.6 indicates that the mixture 3A+7B absorbs the longer wavelength of light amongst all the mixtures. So the mixture 3A+7B will utilize less energy from the source.

#### 4 Determination of pitch from RI

The values of pitch (P nm) and the spacing between thin birefringent layers (d nm) of the cholesteric liquid crystal B and mixtures 5A+5B, 4A+6B, 3A+7B, 2A+8B at temperature 30°C are calculated from equation 2 and equation 3. They are presented in the table 6.

Table 6: Pitch of B, 5A+5B, 4A+6B, 3A+7B, 2A+8B (P= pitch, λ=wavelength of the light used.)

λnm →		404.7	435.8	486.1	546.1	587.6	589.3	632.8	656.3	706.5
B	P	264.626	285.883	320.209	361.017	389.17	390.324	419.832	435.776	469.704
	d	132.31	142.942	160.105	180.509	194.59	195.162	209.916	217.888	234.852
5A+5B	P	264.075	285.512	320.045	361.168	389.588	390.749	420.387	436.323	470.652
	d	132.038	142.756	160.023	180.584	194.794	195.375	210.194	218.162	235.326
4A+6B	P	263.789	285.239	319.775	360.917	389.314	390.474	420.094	436.036	470.276
	d	131.895	142.62	160.388	180.459	194.657	195.237	210.047	218.018	235.138



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3A+7B	P	263.589	284.917	319.433	360.486	388.876	390.04	419.768	435.767	469.747
	d	131.795	142.459	160.217	180.243	194.438	195.02	209.884	217.884	234.824
2A+8B	P	266.133	287.497	321.886	362.821	391.097	392.255	421.83	437.787	471.919
	d	133.067	143.749	160.943	181.411	195.549	196.128	210.92	218.894	235.960

The calculated values of pitch for all the mixtures match with the value of the pitch of cholesteric liquid crystal (sample B). For all the wavelengths, the value of pitch is minimum for the mixture 3A+7B. Also; the distance between the two successive layers of the mixture 3A+7B is minimum amongst all the values.

## VI. CONCLUSION

We studied the miscibility of Nematic liquid crystal with Cholesteric liquid crystal, with increasing concentration of Cholesteryl Chloride in the mixtures. The transition temperatures obtained by Differential scanning calorimetry (DSC) were found to change depending on the proportion of the constituents. Lower transition temperatures were obtained when Cholesteric chloride is mixed with Cholesteryl Oleate in 30%+70% proportion indicating stabilizing effect of pitch of Cholesteric Chloride in the mixture. The Fourier Transform Infra-red Spectroscopy (FTIR) and Ultraviolet – Visible (UV) Spectroscopy techniques confirmed the miscibility of the two liquid crystals. The stability of the mixture 3A+7B was also confirmed by these techniques. The pitch and the spacing between thin birefringent layers is reduced in the mixture 3A+7B. So we can conclude that among all four mixtures studied by us, the mixture of Cholesteryl Oleate and Cholesteryl Chloride in 30%+70% proportion is more stable. Because of its low phase transition temperatures and less requirement of energy, the mixture of Cholesteric chloride with Cholesteryl oleate in 30%+70% proportion holds promise in the optical as well as non-optical applications of liquid crystals.

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