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Image Dynamics of Glucose dispersed *4*-Cyano-*4'*-Pentyl Biphenyl Liquid Crystal: Thermal & Spectroscopic Approach

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Abstract:
To recheck the biosensor nature of 4-cyano-4'-pentyl biphenyl (5CB) mesogen, glucose was directly dispersed in it. A novel approach was suggested to check the biosenitivity of 5CB. Image analysis of pure 5CB & glucose dispersed 5CB (5CBG) mesogens were done through MATLAB software and various optical parameters were measured. These image dynamics have revealed the biosensor nature of 5CB liquid crystal. The phase transition temperatures of both the samples were measured through Polarizing optical microscope, Differential scanning caloriemeter and Image analysis techniques. Variations in the lattice parameters of 5CB due to the presence of glucose were obtained through Powder XRD. All the studies significantly proved that 5CB is a best suitable biosensor material.
Keywords: 4-Cyano-4'-Pentylbiphenyl; Glucose; Absorption Coefficient; Birefringence; Phase Retardation;

INTRODUCTION:

As Science is always updating to improve existing technology, Liquid Crystal materials are also found applications not only in display devices, but also in biosensor devices. Biosensors using liquid crystal materials for sensing the biomolecules are working efficiently and found vast applications in medical field [1-7]. As liquid crystal molecules are very sensitive and respond rapidly in terms of orientation mechanism, the biochemical events occurring at the interface can be viewed easily through polarizing optical microscopy. Glucose biosensor plays major role in diabetes testing. However, there exists good number of research articles that dealt with biosensors and especially to detect glucose through immobilization. Glucose oxidase is the enzyme that would be immobilized initially and varying its concentration in a solvent made the enzyme to exhibit different properties which can be observed through various techniques like change in steady state UV fluorescence [8], cyclic voltammetry measurements [9], development TEM grids using mixed polymer brushes of PAA-b-LCP and QP4VP-b-LCP and observing the textural changes through polarizing optical microscopy [10-11].

The easiest and low cost biosensor was achieved through liquid crystal to detect various biomaterials like glucose, haemoglobin, serum proteins [12]. 4-cyano-4'-pentylbiphenyl (5CB) is the basic liquid crystal that exists in room temperature with nematic phase and was used by good number of researchers for biosensor applications. Going through all the above literature made us to observe the direct mixing of glucose into the lattice of 5CB. The present study reports the variation of 5CB lattice parameters and its image dynamics in the presence of glucose. Glucose doped 5CB (5CBG) was characterized through Polarizing Optical Microscopy, Differential Scanning Caloriemetry, Powder X-Ray Diffraction and MATLAB software. Remarkable changes were observed in XRD and image analysis studies. Results were analyzed systematically through various physical phenomena.

MATERIALS AND METHODS:

In order to characterize the biosensor activity of a liquid crystal, we have purchased 4-cyano-4'-pentylbiphenyl (5CB) from Merck, China and Glucose powder from Baidyanath Company, New Delhi. Both the materials were 99% pure. Powder glucose was then dispersed in the liquid state of 5CB with 5 wt%. The mixer of 5CB and glucose was placed under constant stirring for 3 hours to achieve uniformity. Thus, 5CBG was prepared and made into a glass slide using a glass cover slip such that the thickness of the sample remains 0.17 mm. The slide form of 5CBG was studied under Polarizing Optical Microscopy (POM) from Meopta Instruments in crossed polarisers' position. Textural images of 5CBG were recorded using Sony Digital Camera with 16 MP resolution for every 1^0C rise in temperature. All the images were analyzed for dynamics using MATLAB software. Image analysis was taken for heating mode of observations starting from 25^0C ending to 60^0C. Transition temperatures of the prepared sample (5CBG) were once again confirmed through Differential Scanning Caloriemeter (DSC) of Perkin Elmer Instruments from RRI, Bangalore. DSC scanning was done from 15^0C to 70^0C at a rate of 5^0C/min. For particle size calculations the sample was sent for XRD through which crystallinity of the sample was judged. Finally, all the observations of 5CBG through POM, DSC, XRD and MATLAB were correlated with pure 5CB.

RESULTS AND DISCUSSIONS:

Glucose doped 5CB liquid crystal slide was kept under the eyepiece of crossed polarisers of Polarizing Optical Microscope. Sample was heated from 25^0C and images of the textures were captured for every 1^0C. Astonishingly, the threaded nematic phase of 5CB was changed to marbled nematic phase in 5CBG and phase transition temperatures were decreased to some extent. Marbled nematic phase of 5CBG was shown in Figure 1 and transition temperatures were tabulated in Table 1.

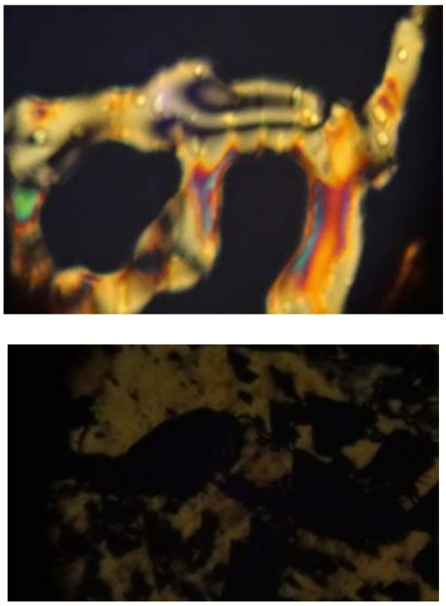

Figure 1. Threaded & Marbled nematic textures of 5CB & 5CBG.

Table 1. Phase transition temperatures of 5CB & 5CBG through POM.

Sample	T_{CN}	T_{NI}	T_{IN}	T_{NC}
5CB	25	37	36	24
5CBG	22	34	33	22

Differential Scanning Caloriemeter (DSC) studies of 5CB and 5CBG samples were helped us to calculate specific heat (C_p) and enthalpy (ΔH) of both samples which are tabulated in Table 2.

Table 2. Phase transition temperatures of 5CB & 5CBG through DSC.

Sample	T_{CN}	T_{NI}	T_{IN}	T_{NC}	C_P	ΔH
5CB	24.39	37.44	23.67	34.26	0.4657	35.93
5CBG	22.59	36.84	22.09	33.21	0.5107	23.95

It is observed from Table 2, that transition temperatures of 5CBG were decreased to some extent compared to pure 5CB. In addition, enthalpy of 5CBG decreased which indicates the formation of this liquid crystal occurred with low heat. Also, it is observed that 5CBG shows high specific heat compared to pure 5CB. These changes in the thermal properties of 5CB revealed that agglomeration of glucose molecules was occurred in the lattice of 5CB, which has resulted to exhibit marble texture and high specific heat. This feature again strengthens the biosenstive nature of liquid crystal. Thus, the dopant molecules have disturbed the lattice structure of 5CB. To study the structural properties, powder XRD was taken to both 5CB and 5CBG samples. Here, remarkable and additional peak was observed in the XRD spectra of 5CBG as shown in Figure 2, which confirms the dopant existence and its influence in the host lattice. The parameters measured from XRD spectra are tabulated in Table 3. Formula used to calculate these parameters are

Bragg's Law:

$$2d\sin\theta_B = n\lambda \quad (1)$$

Scherrer Formula:

$$<t> = \frac{0.94\lambda}{FWHM\ \cos\theta B} \quad (2)$$

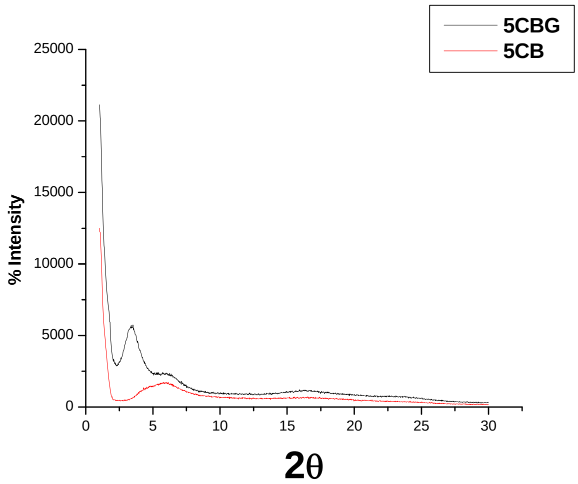

Figure 2. XRD spectra of 5CB & 5CBG.

Table 3. Lattice calculations of 5CB & 5CBG through XRD.

Sample	Θ_B (in degrees)	FWHM (in degrees)	<t> (in A^0)	d (in A^0)
5CB	2.9395	2.961	28.04	15.03
5CBG	1.7318	1.235	67.36	25.51

From Table 3, it is observed that Bragg reflection related to host lattice (5CB) was shifted to lower angles and in turn this increased the interplanar spacing and particle size (<t>). Thus, glucose doping made the 5CB lattice so sensitive to X-ray diffraction.

Finally, a novel approach to know the biosenstive nature of 5CB was done using MATLAB 2013 Software. Recorded images of 5CB and 5CBG were analysed through various codes of MATLAB, in which each digital image was first converted into a matrix of pixel size and mathematical calculations were performed to the matrix. The parameters calculated with this method were [13-18], Mean, Variance, Skewness, Kurtosis, Entropy, absorption coefficient, birefringence, phase retardation and order parameter using the formulae

i) Mean

$$\mu = \frac{1}{N}\sum_{i=1}^{m}\sum_{j=1}^{n} I(i,j) \quad (3)$$

ii) Variance

$$Var = \frac{\left(\sum_{i=1}^{m}\sum_{j=1}^{n} I(i,j) - \mu\right)^2}{N-1} \quad (4)$$

iii) Skewness

$$Skewness = \frac{\left(\sum_{i=1}^{m}\sum_{j=1}^{n} I(i,j) - \mu\right)^3}{(N-1) * S^3} \quad (5)$$

iv) Kurtosis

$$Kurtosis = \frac{\left(\sum_{i=1}^{m}\sum_{j=1}^{n} I(i,j) - \mu\right)^4}{(N-1) * S^4} \quad (6)$$

v) Entropy

$$Entrpoy = -\sum_{i=1}^{m}\sum_{j=1}^{n} P_{(i,j)} \log(P_{(i,j)}) \quad (7)$$

vi) Absorption Coefficient

$$AC = \frac{1}{d}\log\left[\frac{I_0}{I}\right] \quad (8)$$

vii) Birefringence

$$\Delta n = \frac{\lambda}{\pi d}\sin^{-1}\left(\sqrt{\frac{I}{I_0}}\right) \quad (9)$$

viii) Phase Retardation

$$\delta = \frac{2\pi d \Delta n}{\lambda} \quad (10)$$

ix) Order Parameter

$$S = \frac{\Delta n}{(\Delta n)_0} \quad (11)$$

All the above parameters were calculated to each image from 25^{0}C – 50^{0}C for 5CB and 25^{0}C – 60^{0}C for 5CBG. They were shown following behaviour with temperature as shown in Figure 3(i) – 3(xii).

Astonishingly, all the image dynamics of 5CBG observed from Figure 3(ii), 3(iv), 3(vi), 3(viii), 3(x) & 3(xii) were decreased to remarkable amount and at 35^{0}C, whatever style followed by 5CB was completely reversed by 5CBG. In addition, extra peaks were identified in 5CBG to indicate the new phase transitions due to the presence of glucose molecules. Especially, 5CBG shows decrease in entropy values to indicate the orderliness of lattice structure as crystallinity was increased in it. Also, absorption coefficient values were decreased compared to 5CB that strengthens the transmission and scattering properties of the prepared sample to the optical energy. However, 5CBG exhibited decrease in birefringence values when compared to pure 5CB. Finally, Order Parameter (S) of the pure 5CB is around 0.03 in its complete nematic phase and that of glucose dispersed 5CB has got 0.11. Hence, order parameter was increased, that is, same nematic phase with more orderly arrangement of molecules towards the director was achieved in the dispersed sample, which can help to prepare a biosensor in future. This confirms that due to the presence of biomolecules liquid crystal nature was decreased. Phase retardation values of 5CBG showed slight decrement which represents the strength of molecular coupling.

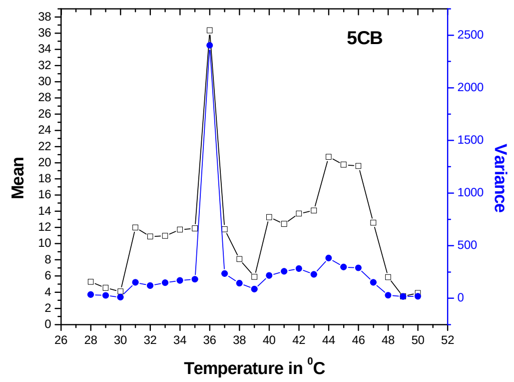

Figure 3(i). Mean & Variance of 5CB.

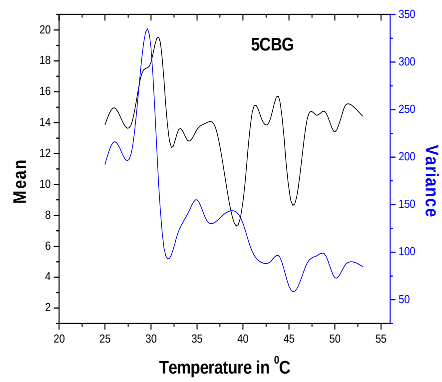

Figure 3(ii). Mean & Variance of 5CBG.

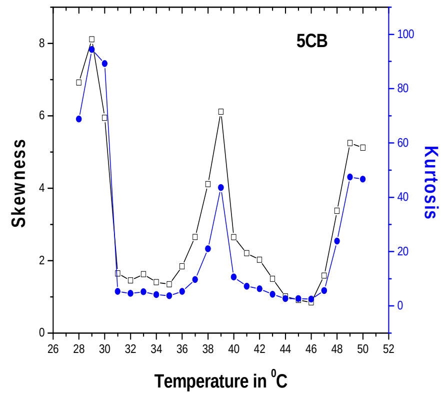

Figure 3(iii). Skewness & Kurtosis of 5CB.

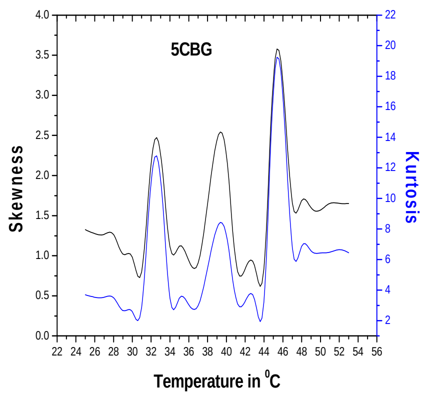

Figure 3(iv). Skewness & Kurtosis of 5CBG.

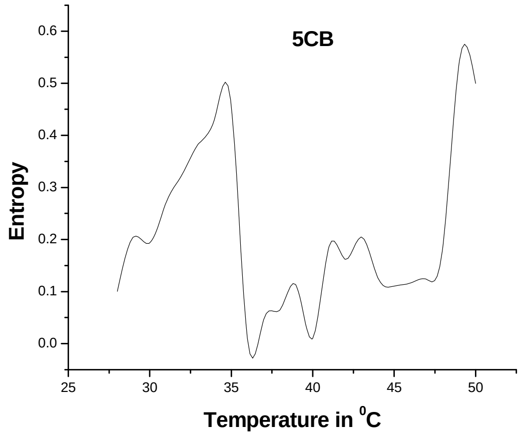

Figure 3(v). Entropy of 5CB.

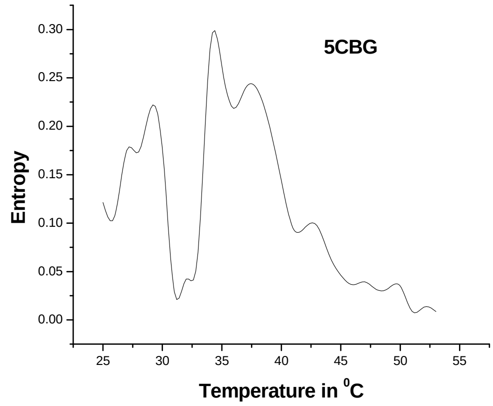

Figure 3(vi). Entropy of 5CBG.

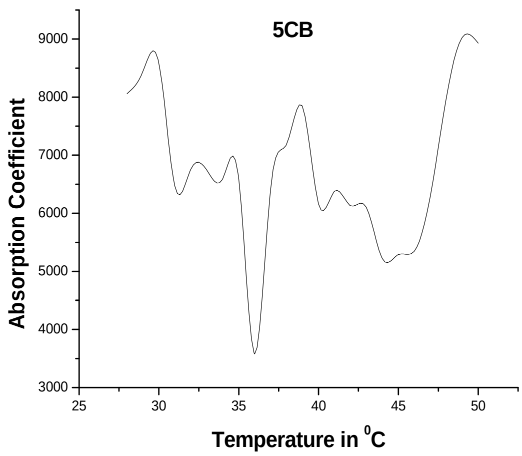

Figure 3(vii). Absorption coefficient of 5CB.

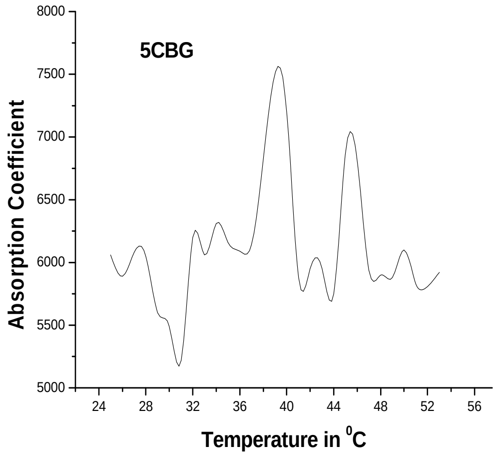

Figure 3(viii). Absorption coefficient of 5CBG.

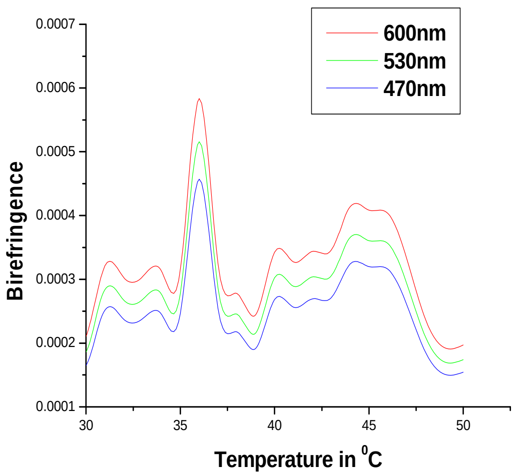

Figure 3(ix). Birefringence of 5CB.

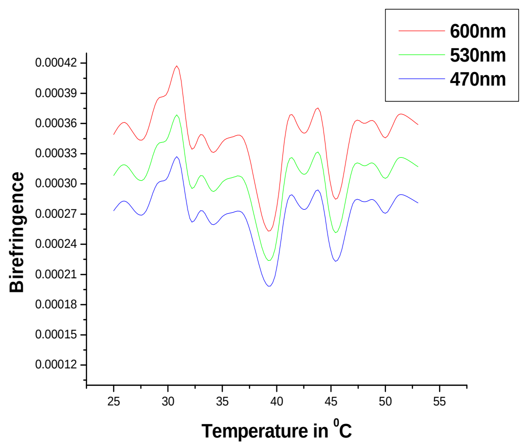

Figure 3(x). Birefringence of 5CBG.

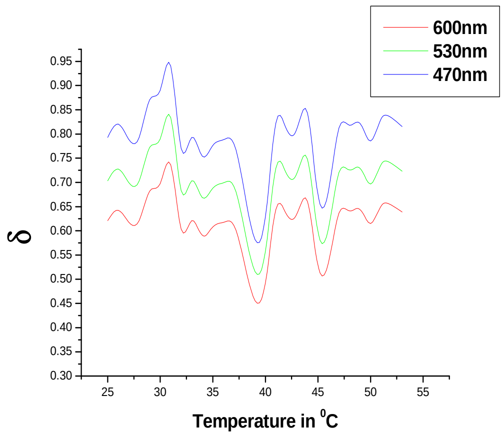

Figure 3(xii). Phase retardation of 5CBG.

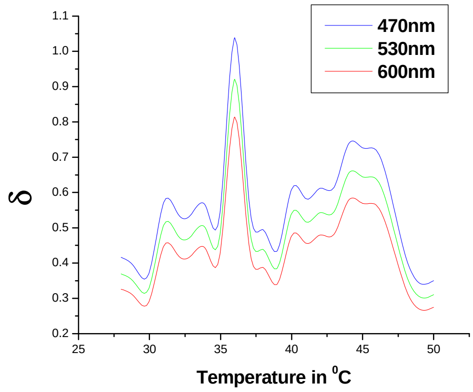

Figure 3(xiii). Order Parameter of 5CB.

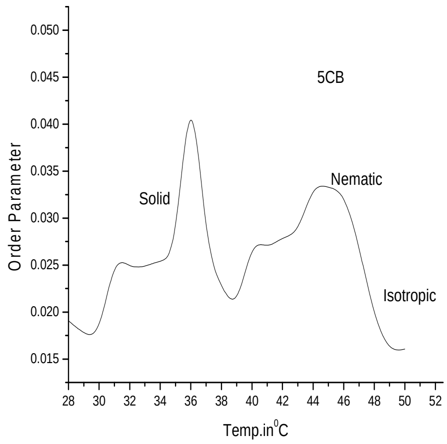

Figure 3(xi). Phase retardation of 5CB.

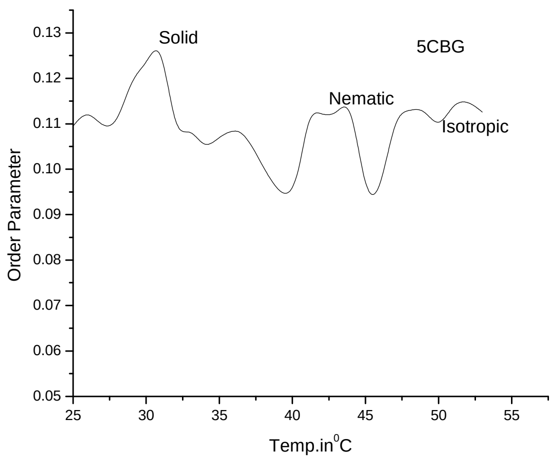

Figure 3(xiv). Order Parameter of 5CBG.

CONCLUSIONS:

The prepared liquid crystal sample was 5CBG. All the observations of 5CBG were correlated with pure 5CB. Prepared sample was characterised through thermal, spectral and image analysis techniques. We can confidently conclude that glucose completely reduces the thermal and optical properties of 5CB lattice which was decreased its LC nature and increased its crystallinity. However, LC phase duration remains almost same for the temperature range in both the samples. Increase in particle size and order parameter, decrease in absorption coefficient, entropy, birefringence, phase retardation reveal the biosensitivity of 5CB. Hence, through all these observations, we have supported that 5CB is a best suitable biosensor material. Especially, the proposed method for biosensor activity recheck is Image analysis through MatLab.

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