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Dielectric relaxation studies in 5CB nematic liquid crystal at 9 GHz under the influence of external magnetic field using microwave cavity spectrometer

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Abstract. Resonance width, shift in resonance frequency, relaxation time and activation energy of 5CB nematic liquid crystal are measured using microwave cavity technique under the influence of an external magnetic field at 9 GHz and at different temperatures. The dielectric response in liquid crystal at different temperatures and the effects of applied magnetic field on transition temperatures are studied in the present work. The technique needs a small quantity (< 0.001 cm^3) of the sample and provides fruitful information about the macroscopic structure of the liquid crystal.

Keywords. Dielectric relaxation; phase transition; liquid crystals; microwaves.

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1. Introduction

Liquid crystals are materials with many unique physical, optical and electro-optical properties. New compounds and mixtures of liquid crystals are chemically stable with low absorption and very large optical anisotropy. They are easily oriented at boundaries and also easily reoriented by electric or magnetic fields. Therefore, these are important optical materials for numerous applications in modern optoelectronics [1]. Nematic liquid crystals are fluid phases found by anisometric molecules which, though free to rotate as in ordinary liquids, are preferentially aligned along a common axis, called director. The dielectric properties play an important role [2–6] in understanding the macroscopic structure of the matter. The dielectric measurements by the resonance techniques have higher accuracy than measurements by the transmission techniques especially for the dielectric loss. Therefore, the resonance techniques are still widely used. Various resonance techniques to conduct dielectric properties measurement and accuracy of microwave cavity perturbation technique have been described elsewhere [7,8]. This technique had been used by various researchers for

studying dielectric response of biomolecules such as water [9], ionic and aqueous solutions [10,11], organic liquids [12] and various liquid crystals [13–16]. Haung Ming *et al* [17] used this technique for measuring the moisture content of sulphide mineral concentrates. Further, it has also been used to study dielectric response of various solid-state materials [18,19] as it provides the results with accuracy especially at low perturbation. Liquid crystals are of considerable interest because of their technological importance [20] as well as for the interaction responsible for the mesophases. The dielectric data help in understanding the molecular structure of compounds and thus it is used as a tool for fundamental research. The dielectric technique [5,6] is important to study the characteristics of materials for many industrial, scientific and medical applications at microwave frequencies.

In this paper the experimental study of 5CB nematic liquid crystal at 9 GHz with two different values of magnetic field is carried out. The observed data of width of resonance profile (Δw) and shift of resonance frequency (Δf) are analysed to measure the relaxation time using Debye's single relaxation time mechanism [2]. The free activation energy (ΔG) has been computed using the data of relaxation time at different temperatures to monitor the phase transition temperatures.

2. Experimental procedure

The 5CB nematic liquid crystal chosen was procured from B.D.H. Limited, UK. This liquid crystal has two phenyl rings with alkyl group having five carbon atoms on one end and a C–N group on the other. It has no central group. Such a liquid crystal is useful from the point of view of chemical and photochemical stability and it can be used in display devices due to its stable thermal character. The fundamental concept of the perturbation technique is that the presence of a *small* piece of the dielectric sample in the resonant cavity will cause a shift in resonance frequency and decrease in the quality factor of the cavity. 'Small' here means that the volume of the sample is much smaller than the volume of the cavity. It is also assumed that the presence of the specimen to the change in the overall geometrical configuration of the electromagnetic field must be very small. The complex permittivity of the specimen can then be calculated from the changes of resonance frequency and the quality factor of the cavity. A cylindrical cavity is adopted as the equations for the fields are easier to derive for simple geometries [21]. A reflex Klystron was used to generate a signal frequency near the cavity resonance by setting voltage and mechanical tuning. A block diagram of microwave cavity spectrometer is shown in figure 1. The detailed description of microwave cavity spectrometer is given elsewhere [7–9].

After setting voltage and mechanical tuning, a signal is generated by a reflex Klystron having frequency near cavity resonance. Saw-tooth ramp voltage was obtained by Tektronix Model 561A oscilloscope. To produce an AC signal of that frequency at the detector, a chopper signal of 31 kHz is impressed simultaneously upon the Klystron repeller electrode. The arrangement allows for the synchronized sweep of frequency with the voltage scan of the oscilloscope. Moreover, 31 kHz signal enables high gain tuned amplifier to be employed to detect the signal reaching the RF detector. The second derivative of the signal was displayed on one channel of the oscilloscope. A signal from the Klystron was sent to the marker mixture along with harmonics of the frequency standard. A tuned radioreceiver (Hammurlund Model HQ 180A) was used to compare these two signals. Two markers

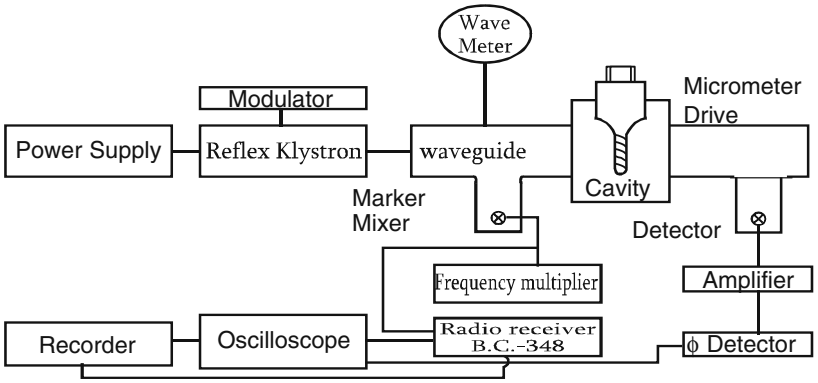

Figure 1. Block diagram of microwave cavity spectrometer used in the present study.

$(f_k - nf_s)$ and $(nf_s - f_k)$, where f_k is the Klystron frequency and nf_s is the proper harmonic of the standard signal source, were generated and displayed on the second channel of the oscilloscope. The separation of these markers was set to 4 MHz. A cylindrical microwave cavity is shown in figure 2 with an internal diameter of 4.9 cm and a quality factor $Q_L \approx 5000$ operating in the TM_{010} mode served as the test cavity.

Temperature control of the sample was achieved by flushing chilled or steam-heated air around the resonant cavity with the help of copper pipe heat exchanger placed in a Dewar flask or in boiling water. The rate of flow was adjusted as required to maintain thermal equilibrium between the sample and the cavity. The cavity was thermally insulated and shielded to maintain temperature stability. The temperature of the sample was measured by a thermocouple lead using an analog voltmeter. The measurements of the resonance frequency, shift and width were taken before and after putting the sample in the cavity. The

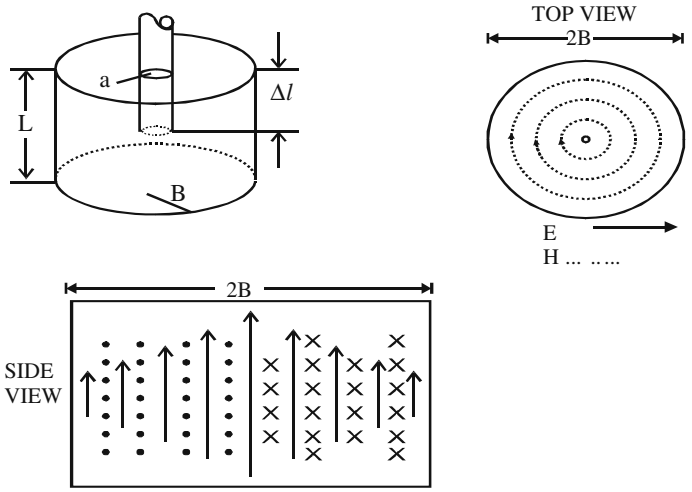

Figure 2. A sketch of the resonance cavity with assumed fields in TM_{010} mode pattern.

temperature was recorded at appropriate steps over a desired temperature range from 288 to 308 K.

Slater's perturbation equations [3,4] was used to find the dielectric permittivity (ε'), dielectric loss (ε''), relaxation time (τ) and activation energy (ΔG) by analysing the measured data of relative shift of the resonant frequency (Δf), relative width of the resonant profile (Δw) and the amplitude of the resonance profile (Δh). The frequency of the applied field was fixed at 9 GHz for both sample and the cavity operated in the TM_{010} mode.

These data, Δf and Δw, were analysed using Slater's perturbation equations for finding dielectric permittivity (ε') and dielectric loss (ε'') using the following equations:

$$\frac{\Delta f}{f_0} = -\left[\frac{(\varepsilon' - 1)}{2}\right] F(E) \tag{1}$$

and

$$\Delta\left(\frac{1}{Q}\right) = \frac{\Delta w}{f_0} = \varepsilon'' F(E), \tag{2}$$

where f_0 is the cavity resonance frequency. The form factor $F(E)$ represents the field interacting with the sample in the cavity and the energy stored per cycle in it, which is expressed by

$$F(E) = \frac{\int_v E_s \cdot E_0 \, dv}{\int_V E \cdot E_0 \, dV}. \tag{3}$$

Here E represents the electric field applied to the cavity, E_0 is the unperturbed component of the electric field in the cavity without the sample, E_s is the field interaction with the sample, v is the volume of the sample and V is the volume of the cavity. The microwave cavity spectrometer is appropriate to determine relaxation time when the form factor is annulled. Dividing eq. (2) by eq. (1) we get the loss tangent.

The expression for the loss tangent ($\tan\delta$) is given as

$$\tan\delta = \left(\frac{\varepsilon''}{\varepsilon'}\right) = \frac{\Delta w}{2\Delta f}. \tag{4}$$

The relaxation time (τ) using Debye's mechanism of relaxation time is

$$\tau = \left(\frac{1}{\omega}\right) \times \frac{\Delta w}{2\Delta f}, \tag{5}$$

where $\omega (= 2\pi f_0,\ f_0 = 9\ \text{GHz})$ is the angular frequency.

These frequency domain measurements need a new frequency each time. However, we have done experiments at 9 GHz frequency.

The activation energy (ΔG) is determined using the equation

$$\Delta G = 2.303 RT \log\left(\frac{\tau k T}{h}\right), \tag{6}$$

where R is the molar gas constant, h is the Plank's constant, k is the Boltzmann constant, T is the absolute temperature and τ is the relaxation time derived from eq. (5).

3. Results and discussions

The variation of Δf, Δw, τ and ΔG with temperature is shown in figures 3, 4, 5 and 6, respectively, for magnetic fields $B_1 = 190$ G and $B_2 = 652$ G. It is evident that the temperature variation of the shift, width, relaxation time and free energy of activation are nonlinear. This nonlinearity arises due to interactions. The width of the resonance profile, relaxation time and free energy of activation show changes in nonlinearity with increase in temperature. Δw and Δf increase by increasing the temperature in the crystal (C) to nematic (N) regions with an abrupt decrease during C–N and N–I (isotropic) phase transition. The transition temperature for the change of phase from C to N is 297 K and that for N to I is 307.6 K, which are in good agreement with literature values. But this is not so sharp, may be due to the smaller variation of the activation energy from C to N and N to I. The other discontinuities and nonlinearites in the plots may be due to higher-order transitions. The amplitude of the resonance profile is increased as a result of the reduced damping at higher temperatures. It is interesting to note that there is a good correlation between the variation

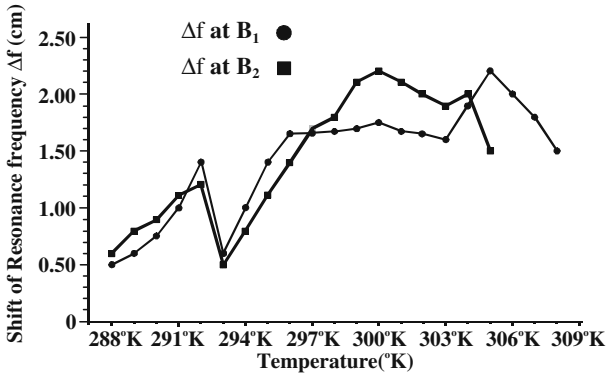

Figure 3. Plot of temperature vs. shift of resonance frequency: $B_1 = 190$ G, $B_2 = 652$ G.

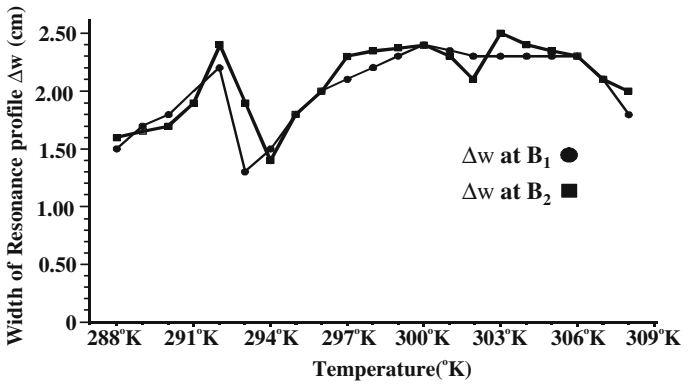

Figure 4. Plot of temperature vs. width of resonance profile: $B_1 = 190$ G, $B_2 = 652$ G.

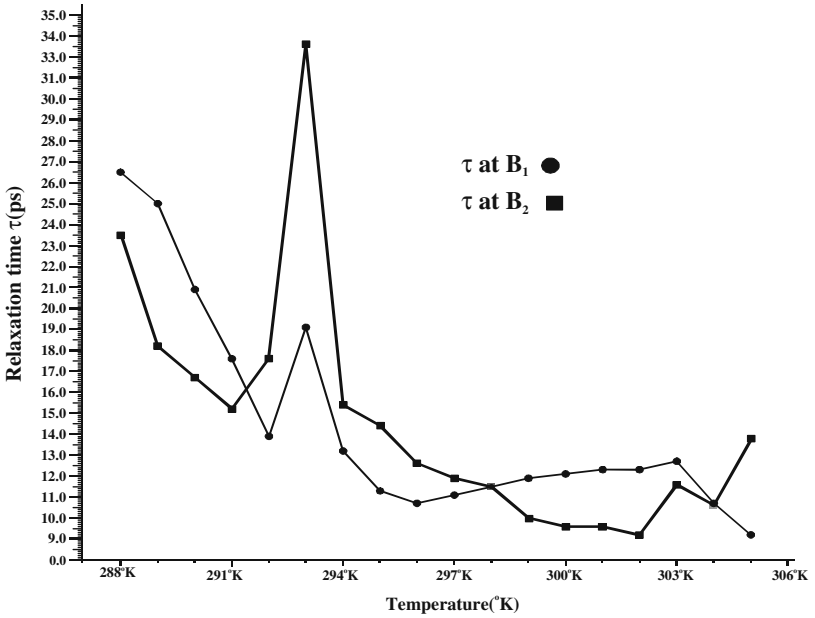

Figure 5. Plot of temperature vs. relaxation time: $B_1 = 190$ G, $B_2 = 652$ G.

of the shift and the amplitude of the resonance profile making it evident that polarizability changes by applying the electric field. Johri and Roberts [22] also found nonlinear variations of these parameters for water systems and interpreted this in terms of the associated behaviour of the hydrogen bonding in water. As the magnetic field increases, the relaxation time and the activation energy increase abruptly at 293 K. This increase may be due to the presence of higher conducting atoms in the sample liquid crystal.

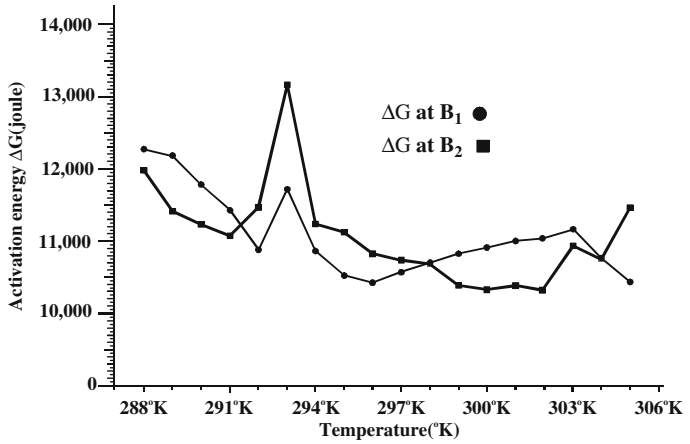

Figure 6. Plot of temperature vs. activation energy: $B_1 = 190$ G, $B_2 = 652$ G.

4. Conclusions

From the present experimental investigations and detailed analysis of the data of the 5CB liquid crystal, we can conclude that the microwave cavity spectrometer is the best to monitor phase transition in liquid crystals and relative change in dielectric response, and it requires very small quantity of sample ($<10^{-9}$ m^3). Further, unlike the dielectric resonance techniques where computer programs are required to solve complicated characteristic equations, the calculation for the complex permittivity of the perturbation technique is relatively simple and does not require such programs. The nematic liquid crystal chosen for this study, that is 5CB, is sensitive to perturb the cavity and the magnetic field, and it influences the phase transition mechanism significantly. This liquid crystal is potentially useful at a higher frequency range. The temperature dependence of the dielectric response gives valuable information about the phase transition occurring in this system. It is worth mentioning that the cavity perturbation technique gives relative measurement of resonance width, shift and the absolute values of ε' and ε'', which are possible if $F(E)$ could be measured or predicted by any theoretical model which includes all molecular parameters and their anisotropies. A complete phase transition mechanism on the microscopic scale is possible only if perturbations can be observed by proving the molecular system for a wider range of frequencies.

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