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THERMAL, FERROELECTRIC AND DIELECTRIC STUDIES ON TWO DISTINCT STRUCTURAL ISOMERS: A FIELD INDUCED TRANSITION IN PSEUDO-NEMATIC PHASE

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Thermal, Ferroelectric and Dielectric Studies on Two Distinct Structural Isomers: A Field Induced Transition in Pseudo-Nematic Phase

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The thermal and ferroelectric properties of a novel series comprising of two distinct ferroelectric structural isomers, (S)-1,4 – diphenylene bis (2-chloro-3- (4 – dodecyloxyphenyl)-propionate) (DCDP-1) and (S)-1,3 – diphenylene bis (2-chloro-3- (4 – dodecyloxyphenyl)-propionate) (DCDP-2), are studied by polarizing thermal microscopy (TM), differential scanning calorimetry (DSC), spontaneous polarization (P_S), response times (τ), viscosity (η) and dielectric techniques. The thermal studies imply these isomers exhibit altogether different thermal and phase behaviour. The comparative ferroelectric investigations infer an increment in the magnitude of P_S(three fold) and a wide ferroelectric thermal range in DCDP-2 compound. Further, the detailed ferroelectric studies on DCDP-2 compound reveal the occurrence of spontaneous polarization, for the first time, in the threaded nematic like phase (here after this phase is referred as pseudo-nematic). A detailed investigation confirming the existence of a field-induced transition (FiT) from pseudo-nematic to nematic phase is achieved through spontaneous polarization and dielectric studies.

Keywords: DCDP-1; DCDP-2; FiT; pseudo-nematic

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INTRODUCTION

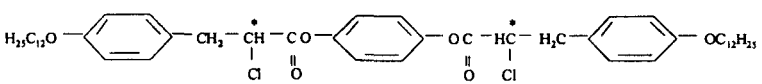
It is well known that the origin of chirality in a liquid crystalline molecule is induced by the incorporation of an asymmetric centre [1]. The impact of the chirality on the phase behaviour and macroscopic structure can be envisaged either by substituting an electronegative atom at the asymmetric carbon or by increasing the additional transverse dipoles along the long molecular axis [2]. On the otherhand, the recent trend of obtaining ferroelectricity in achiral mesogens can be achieved through the incorporation of C_{2v} molecular symmetry [3-7]. Furthermore, the advent of this molecular symmetry on the ferroelectric properties of achiral molecules is also realized by the high magnitude of spontaneous polarization and microsecond response times. Such a polar symmetry, however, promotes a unique way of forming helix, which influences many physical and thermal properties of the tilted mesophases. It is well known [2] that the configuration of the helicoidal structure can be altered by the variation of thermoelectric conditions (temperature and/or applied field) which inturn effects the molecular alignment giving rise to the origin of new phases. Watanabe et.al recently reported [6] the influence of applied field on the phase behaviour of achiral molecules and they proposed a frustrated structure to the ferroelectric smectic phases.

In fact, due to their structural dissimilarities in the helicoidal configuration of molecular layers, no comparative studies have been made to correlate the conventional FLC's having linear structure with

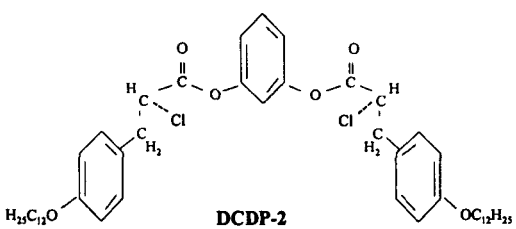
that of ferroelectric materials possessing C_{2v} symmetry (bow shaped) in the direction of their the ferroelectric properties. In order to study the impact of such configurational dissimilarities in the helicoidal structures of FLC materials belonging to the above categories, the design of molecule plays a crucial role which intern facilitates to compare their significant mesogenic properties associated with the tilted phases. In other words, a proper correlation between the thermal and ferroelectric properties is possible only when the molecule possesses a structural isomerism which constitutes two distinct molecular symmetries (linear and bow shaped molecules). Nevertheless, this structural anomaly also facilitates to elucidate the role of asymmetric carbons and bent molecules possessing C_{2v} symmetry and vice versa.

The desired structural isomeric configurations (linear and bow) of FLC materials under present investigation, DCDP-1 and DCDP-2 are obtained by designing their molecular skeletons in such a way that: (a) two chlorine atoms are introduced at each asymmetric center of L-tyrosine by the nucleophilic substitution of amino group with the retention of asymmetric configuration (b) the esterification of hydroxyl groups of the central phenyl moieties at 1,3- and 1,4 – positions. In continuation of our work on chiral [8-12] and achiral [13] materials. the present communication deals with the experimental investigations on two ferroelectric isomers, DCDP-1 and DCDP-2 (Fig. 1) using polarizing thermal microscopy (TM), differential scanning calorimetry (DSC), spontaneous polarization (P_S), response times (τ), viscosity (η)

and dielectric techniques. Moreover, we made a successful attempt to establish the occurrence of a new ferroelectric pseudo-nematic phase accompanied by a field induced transition (FiT) in DCDP-2 compound through a proper correlation of the experimental results on P_S and dielectric studies.



DCDP-1



DCDP-2

FIGURE 1 Molecular structures of DCDP-1 and DCDP-2

EXPERIMENTAL

Techniques

Optical textural observations are made with Olympus DX-50 polarizing microscope equipped with DP10 CCD display while the temperature is monitored and controlled by an Instec milli Kelvin temperature

controller interfaced to a PC with an accuracy of ± 0.01 K. DSC thermograms are recorded on a DSC-7 Perkin Elmer with a scan rate of 5^0C/min in the cooling runs. For the measurement of ferroelectric and dielectric properties the compound is filled in a polyimide buffed 10μ spacer cell (Device Tech., USA) by capillary action in its isotropic state. Spontaneous polarization is measured by field reversal method [14] with an automated set-up and the resulting polarizing current profiles are recorded on a 500 MHz digital storage oscilloscope (Hewlett Packard- 54610B). The profiles are digitized and analyzed using a GPIB 54657A, which is interfaced to an Intel Pentium computer system. Response times are simultaneously measured [15] with spontaneous measurements. Dielectric studies are carried out on a Hewlett Packard 4192A low-frequency impedance analyzer using Instec STC 200C temperature controller equipped with HS 400 hot stage.

The detailed synthetic procedure of compounds, DCDP-1 and DCDP-2 along with their structural confirmation by spectral techniques are reported elsewhere [12].

RESULTS AND DISCUSSION

Thermal studies

The phase variants and their transition temperatures are determined [16] from the characteristic textural observations using polarizing microscope.

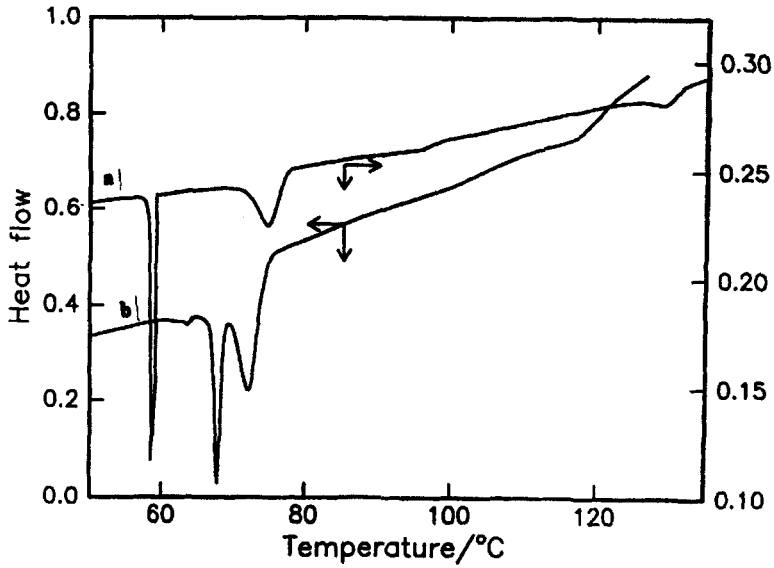


FIGURE 2 DSC thermogram of (a) DCDP-1 and (b) DCDP-2

On cooling the isotropic liquid, the DCDP-1 compound exhibits threaded nematic texture in chiral nematic (N*) phase, focal conic fan texture (yellow colour) in smectic-A phase, appearance of transition bars with concentric striations across these fans in smectic-C* phase, broken focal conic texture in smectic-F* and smooth multicoloured mosaic texture in smectic-G phase. However, the textural observations of DCDP-2 molecule imply a threaded nematic (N*), schlieren marble smectic-C*, broken focal conic texture in smectic-F* and smooth

mosaic in smectic-G phases. The phase sequence variant of the present compounds are represented as:

DCDP-1: Iso. – N* - SmA – SmC* - SmF* - SmG – Cryst.

DCDP-2: Iso. – N* – SmC* - SmF* - SmG – Cryst.

TABLE 1 Transition temperatures of DCDP-1 and DCDP-2 compounds obtained by different techniques

	Technique	Iso–N*	N*-A	A-C*	C*-F*	F*-G	G-K
DCDP-1	TM	130.4	95.2	81.6	76.2	68.5	60.7
	DSC	129.2	94.9	[*]	74.8	[*]	58.9
		(7.2)	(4.0)		(14.9)		(75.6)
	P_s	---	---	77.0	75.5	---	---
	Dielectrics	130.4	97.8	81.7	76.2	66.6	60.9
		Iso–N*	N*-C*		C*-F*	F*-G	G-K
DCDP-2	TM	116.5	104.6		103.1	97.8	84.5
	DSC	116.9	100.1		[*]	[*]	78.2
		(8.6)	(1.5)				(22.2)
	P_s	114.7	105.1		97.5	96.2	---
	Dielectrics	116.2	103.8		101.8	96.9	83.6

Temperatures are given in degree centigrade and enthalpies are given in parentheses

The corresponding transition temperatures of P_s show gradient due to the variation in the scan rate

[*] DSC peaks are not well resolved

Pseudo-nematic phase

Since the formation of helix in the chiral smectic layers involves tilt orientational ordering in a direction normal to the layers, the spontaneous polarization is also rotated in a spiraling manner in the same direction. If such a helix is unwound by means of applied field, the corresponding smectic layers will become ferroelectric[2]. However, in the case of a chiral nematic layers, a twisted structure associated with the side-to-side interaction of the molecules leads to the formation of the helix, which is entirely different from the helicoidal structure of smectic layers [2], and this could be the only possible reason for the non-ferroelectric nature of this optically active phase. Interestingly, in the present study, for the first time, chiral nematic phase of DCDP-2 molecule is found to exhibit spontaneous polarization with a magnitude of ~40 nC/cm ^{2} and hence, we designate this phase as pseudo – nematic. These results along with the influence of field on the existence of ferroelectric nature of pseudo-nematic phase will be discussed in the forthcoming sections.

Spontaneous polarization

Polarizing current profiles, obtained by field reversal method, at different temperatures are analyzed to obtain the magnitude of spontaneous polarization. The temperature variation of the spontaneous polarization of DCDP-1 and DCDP-2 compounds are illustrated in Figure 3.

In the DCDP-1 mesogen the onset of smectic C* is observed at 77.3^{0}C with P_s = 9 nC/cm^2. The spontaneous polarization increases with decreasing temperature and attains a saturated value of 32 nC/cm^2 at 75.5^{0}C. On further decrease of temperature the value of spontaneous polarization starts to decrease indicating the onset of smectic F* phase. In the entire thermal span of smectic C*, the proportional increase of spontaneous polarization with temperature indicates the stabilization of this phase.

The spontaneous polarization studies with the simultaneous optical observations of DCDP-2 molecule reveal the presence of pseudo-nematic (threaded nematic texture) and smectic-C*(schlieren) phases (Fig.3). The onset of P_s in pseudo-nematic phase starts at 114 ^{0}C and attains a saturated value of 40nC/cm^2 at 105^{0}C. The transition from pseudo-nematic to smectic-C* is manifested by a kink in the P_s (Fig.3). In the smectic-C* thermal range (105 – 97.5^{0}C), the magnitude of P_s rises from 40 to 95 nC/cm^2 before it starts to decrease indicating the onset of smectic- F* phase (broken focal conic fan texture).

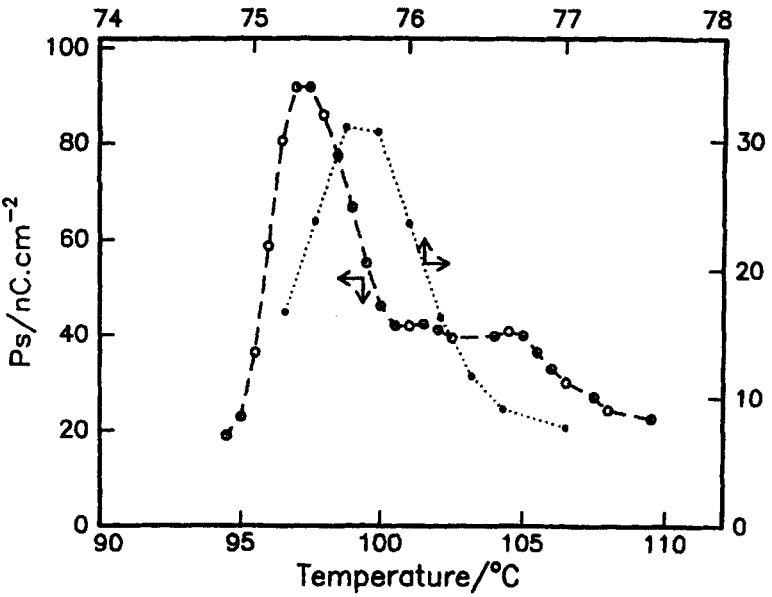


FIGURE 3 The temperature variation of spontaneous polarization (10 Hz ± 120 V): solid circles represent P_s of DCDP-1; hollow circles represent P_s of DCDP-2

Field Induced Transition (FiT) in Pseudo-Nematic Phase

A quantitative approach is made to study the effect of applied electric field on polarizing current profile in the phase. Figure 4 illustrates the

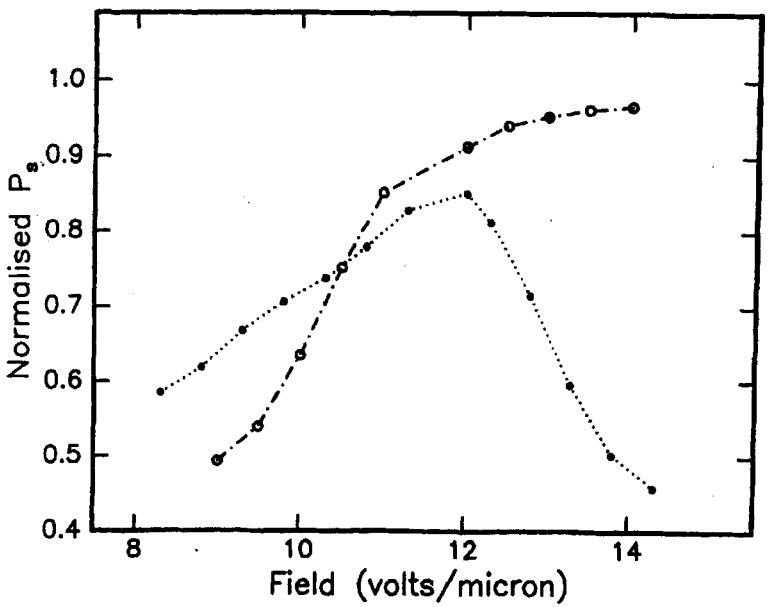


FIGURE 4 Variation of field vs normalized P_s: hollow circles represent P_s of DCDP-1; solid circles represent P_s of DCDP-2

influence of the field on normalized P_s at 110°C for DCDP-2 (pseudo-nematic) compound. The usual trend of increment in the magnitude of P_s with field is observed until the field reaches a critical value of 12 V/micron. A noteworthy feature of this study implies that on further increase of field beyond this critical value, the magnitude of

spontaneous polarization starts to decrease and finally annihilates when the applied field is 14 V/micron and also found that this polarizing profile re-emerges with the withdrawal of the critical field. Moreover, it is noticed that the magnitude of response times remains unaltered in the entire thermal span of pseudo-nematic phase even after the applied field crosses the critical value of 12 V/micron. On the basis of this experimental evidence, it is reasonable to attribute this phenomenon to a field induced transition (FiT) from a pseudo-nematic phase similar to that observed in its isomer (DCDP-1). Further, this FiT persists in the entire thermal range of pseudo-nematic phase. However, a similar experimental study in the successive C* phase suggests that this field has no such influence on the polarizing current profile. In other words, in the entire thermal range of C* the normalized P_s increases with field and attains a saturated value at 12 volts per micron, as expected in conventional FLC's [8,9]. Further increase of field beyond the critical value has no effect on the saturated value of the normalized P_s. It is interesting to recall our previous results on an AFLC system [10] where an applied field with a critical value of 2V/micron is required to generate a field induced transition from tristable antiferroelectric to one of the bistable ferroelectric phases. The possible explanation towards the occurrence of FiT in the pseudo-nematic phase can be substantiated with the molecular contributions arising due to the polar symmetry (C_{2v}) which intern influence its tilt orientational ordering normal to the layers. The FiT from a pseudo-nematic to chiral nematic phase is also

evidenced from the fact that: a similar experimental observation of influence of applied field on the switching behaviour in traditional chiral nematic (DCDP-1) and pseudo-nematic (DCDP-2) implies an identical switching behaviour in both the phases.

Response Times and Viscosity

Temperature variation of response times (τ) of both DCDP-1 and DCDP-2 compounds are illustrated in Figure 5. A linear variation of response times with decreasing temperature is obtained for the DCDP-1 compound with a maximum value of 80 μ seconds at 75.5 °C. The response times fall sharply with the onset of smectic F* phase. In the DCDP-2 compound, the response times increase with decreasing temperature and attains a saturated value of 170 μ seconds at 100 °C. The decrease of temperature shows the steep fall of the response time indicating the onset of smectic F* phase.

The effective tortional viscosity η is computed (without considering the switching mechanism) from the response times τ

$$\tau = \eta/P_s.E$$

where E is the applied field. The variation of η with the temperature for both DCDP-1 and DCDP-2 compounds are illustrated in figure-6. A glance at this suggests the high viscous nature of smectic C* phase of DCDP-2 when compared to DCDP-2 when compared to DCDP-1. This high viscous nature can be attributed to the bow configuration of the

molecule achieved through the inducement of the C_{2v} symmetry. Moreover, this result is in good agreement with the earlier reported work of Niori *et al* [4,5].

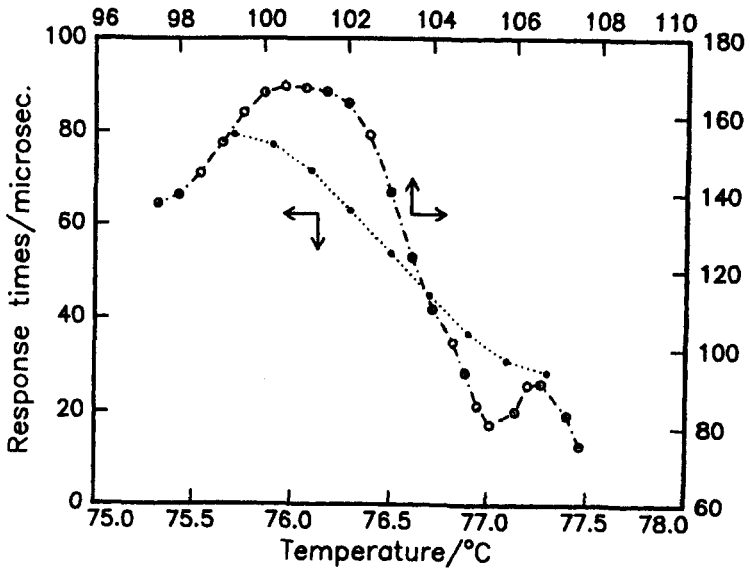


FIGURE 5 Variation of response times: solid circles represent DCDP-1; hollow circles represent DCDP-2

Dielectric Measurements

Transition temperatures in the thermotropic liquid crystals are determined either by thermal microscopy or by differential scanning

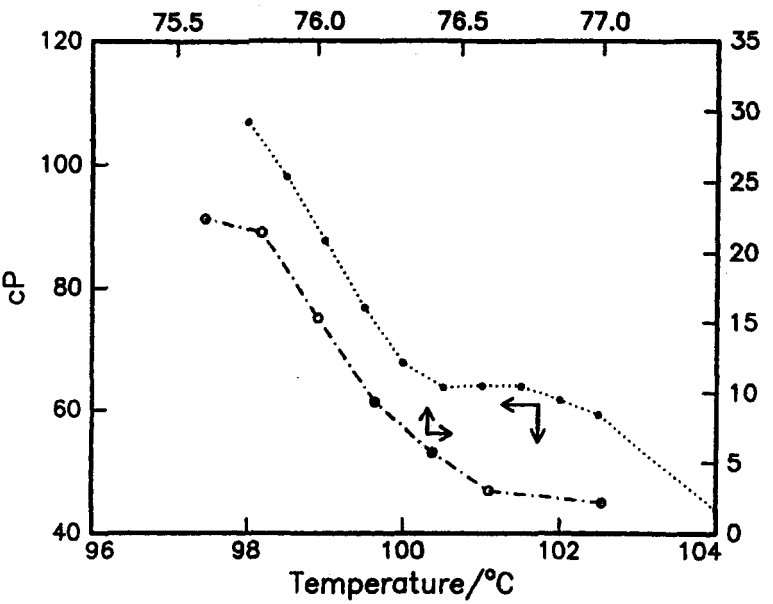


FIGURE 6 Variation of viscosity : hollow circles represent DCDP-1; solid circles represent DCDP-2

calorimetry (DSC). Some phase transitions involve only subtle paramorphatic changes and require an experienced eye to detect them, while DSC can detect only first transitions involving large enthalpy

values. Transition temperatures which can not be resolved either by thermal microscopy or DSC such as second order transitions can also be detected by the temperature variation of dielectric constant.

The dielectric measurements have been performed in pseudonematic and smectic C* phases with the layers aligned perpendicular to the glass plates. The cell is used is polyimide buffed 10 m spacer (Display Tech., USA). It is calibrated using standard substance to calculate the stray capacitance, C_s. The empty cell capacitance, C_o is measured and found to be frequency independent in the frequency range from 1 Hz to 2 MHz. Sample orientation is obtained by slowly cooling the sample from the isotropic phase in the magnetic field. A bias of 3.5 volts per micron is applied in the direction perpendicular to the helical axis to suppress the helix. The real and imaginary ε' and ε'' of the complex dielectric constant are calculated using relations

$$\varepsilon' = (C - C_s)/(C_o - C_s)$$

$$\varepsilon'' = 1/[2\pi f (C_o - C_s)]$$

Where C is the capacitance of the cell when filled with liquid crystal and f is the frequency of measuring field. At low frequency the measured dielectric absorption has been an additional contribution from the ions; the non-liquid crystalline contributions are eliminated by a standard procedure [18]. For each temperature the dielectric absorption, ε' is corrected for the ionic contributions.

The temperature variation of permittivity, (ε′) and dielectric loss (ε″) are determined at a frequency of 10 kHz in different smectic phases of DCDP-1 and DCDP-2 compounds and are depicted as figures 7 and 8, respectively. From the dielectric spectra of DCDP-1 a peak observed at 130.4^{0}C is attributed to the isotropic to nematic transition. The region

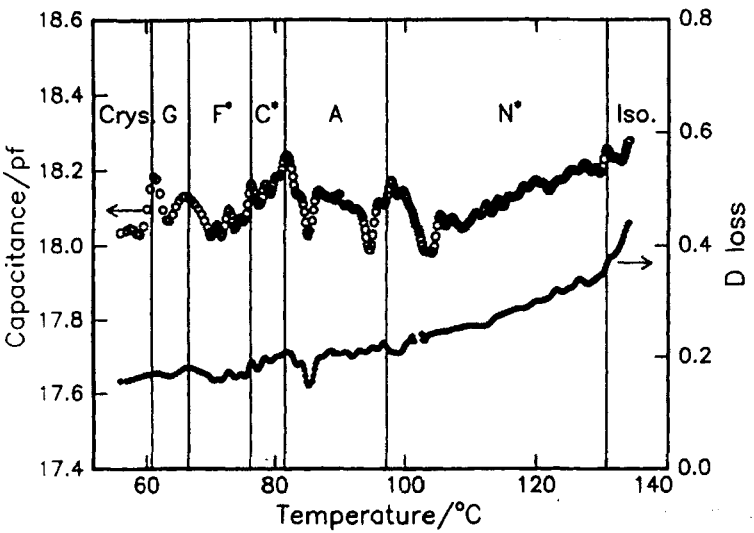


FIGURE 7 Dielectric spectra of DCDP-1 mesogen: hollow Circles represent permittivity variation; solid circles denote dielectric loss

between 130.4 and 97.8^{0}C remained almost linear indicating the stabilization of this nematic phase. A peak at 97.8^{0}C indicates a transition from nematic smectic A phase. In the entire thermal range of

smectic-A, the permittivity is unaltered. Few dips in the spectra may be due to the subtle paramorphitic change in this phase. On further cooling smectic A transforms to smectic C* phase which is reflected as a peak at 81.7^{0}C. Smectic C* phase as a narrow thermal range and it culminates at 76.2^{0}C with the onset of the smectic F* phase which is observed in the dielectric spectra as a low intense peak. The onset of smectic G is attributed to broad peak at 66.6^{0}C. Finally a sharp peak at 60.9^{0}C indicates crystallization of the sample. Similar variations of identified in the dielectric loss spectra of this compound.

The dielectric spectra of DCDP-2 mesogen are illustrated in figure-7. In the permittivity profile, a sharp peak at 116.2^{0}C indicates the isotropic to pseudonematic transition. Small peaks at 112.0 and 108.0^{0}C correspond to the paramorphitic changes in the nematic phase. This pseudo-nematic phase culminates at 103.8 ^{0}C giving rise to the smectic C* phase, which is reflected in dielectric spectra as a well resolved sharp peak. The narrow thermal range (103.8 to 101.8^{0}C) of smectic C* phase is observed between two resolved sharp peaks. The phase transition from smectic F* to smectic G is observed at 96.9^{0}C in the form of broad peak. The peak at 83.6^{0}C is attributed to crystallization of the compound. Similar anomalies are observed in the dielectric loss spectrum also. Further, the phase transition temperatures obtained by this technique are in concurrence with those obtained from TM, DSC and spontaneous polarization studies (Table 1).

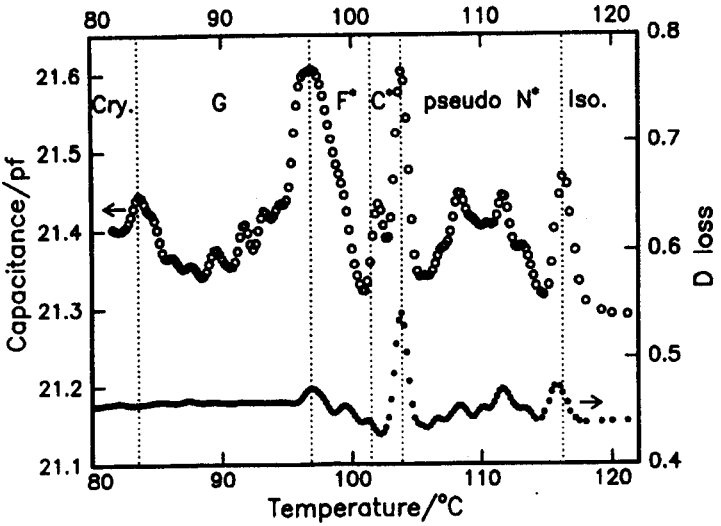


FIGURE 8 Dielectric spectra of DCDP-2 mesogen: hollow circles represent permittivity variation; solid circles illustrate dielectric loss

Goldstone mode in pseudo-nematic and smectic C* phases

In its relaxed state, the smectic C* structure is helicoidal, and can be described [19] in terms of one dimensional model, giving the azimuthal angle φ as a function of z along the helix axis, $\varphi = qz$, where q ($2\pi/Z$) is the wave-vector of the helix and Z is the helical pitch. The local polarization vector P (z) also traces out a helix in the z-direction:

$$P = (-P \sin \varphi, P \cos \varphi, O) \qquad [1]$$

For the local direction n we have

$$N = (\sin \theta \cos \varphi, \sin \varphi, \cos \theta) \quad [2]$$

where θ is the angle between the director and the smectic layer normal, coinciding with the z-axis. If P from equation [1] is projected on to a circle with φ ranging over a full period, the isotropic distribution of P gives no average polarization along y-axis. However, an electric field along 'y' will make the P-distribution anisotropic yielding a net P_y component. It is this component responsible for the origin of the Goldstone mode contribution to the dielectric constant. When the helical structure in the C* phase is deformed and allowed to relax, it does so with a characteristic relaxation time τ.

The two fluctuation modes in this chiral smectic liquid crystals are the Goldstone mode associated with the fluctuation of azimuthal angle (φ) around the helical axis and the soft mode described as the fluctuation of the molecular tilt (θ).

The dielectric relaxation measurements are performed in both pseudo-nematic and smectic C* phases with the layers aligned perpendicular to the glass plate. Sample orientation is obtained by slowly cooling the sample from the isotropic to the smectic C* phase in the magnetic field. The dielectric strength of the Goldstone mode and the corresponding relaxation frequency are obtained as a function of

temperature using the Cole-cole plots. The relaxation mechanism of the Goldstone mode very nearly follows a Debye behaviour (Fig.9). The temperature dependence of relaxation frequency of the Goldstone mode in pseudo-nematic phase is illustrated in the Figure 10. As expected in the case of the conventional FLC's [20,21], the dielectric strength of DCDP-2 due to the Goldstone mode decreases and its relaxation frequency increases with the application of bias fields.

The frequency spectrum over different temperatures reveals that there are two relaxation mechanisms relating to the Goldstone mode in pseudo-nematic and smectic C* phases at 18 kHz and 9 MHz, respectively. It is observed that both the relaxations frequencies shift to the lower side with the decrease of temperature and are classified as Goldstone mode as they are suppressed with the applied dc bias field.

The relaxation frequency is obtained as a function of temperature using Cole-Cole plots. Figure-10 illustrates the plot of logarithm of the relaxation frequency versus the inverse of the absolute temperature in pseudo-nematic and smectic C* phase. This Arrhenius plot yields the activation energies of 0.34 eV in pseudo-nematic and 0.51 eV in smectic C* which are in quantitative agreement with the reported data [22] for other ferroelectric substances. A glance at figure 10 shows a linear variation of relaxation frequencies in pseudo-nematic and smectic

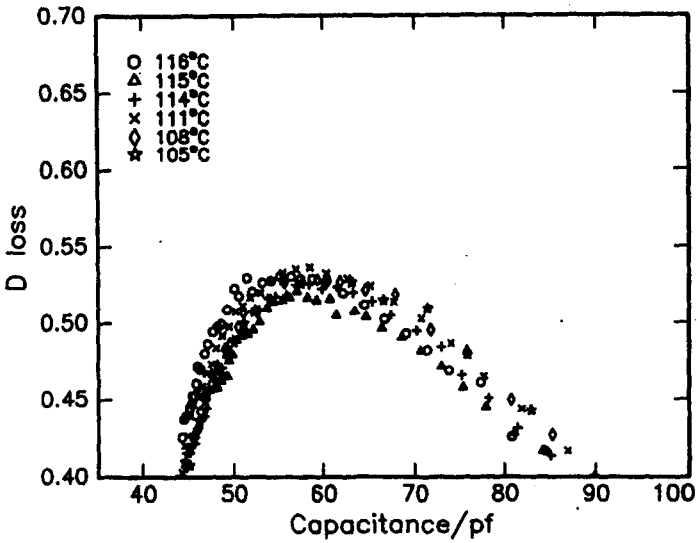


FIGURE 9 Cole-cole plot at different temperature in pseudo-nematic phase of DCDP-2

C* phases, which clearly illustrate two distinct dipolar mechanisms, operate throughout the entire thermal ranges of both the phases. Further, it is worth mentioning that magnitudes of the activation energies derived from the trend in the variation of the slopes of relaxation frequencies (Fig. 10) clearly imply a phase transition from pseudo-nematic to smectic C*.

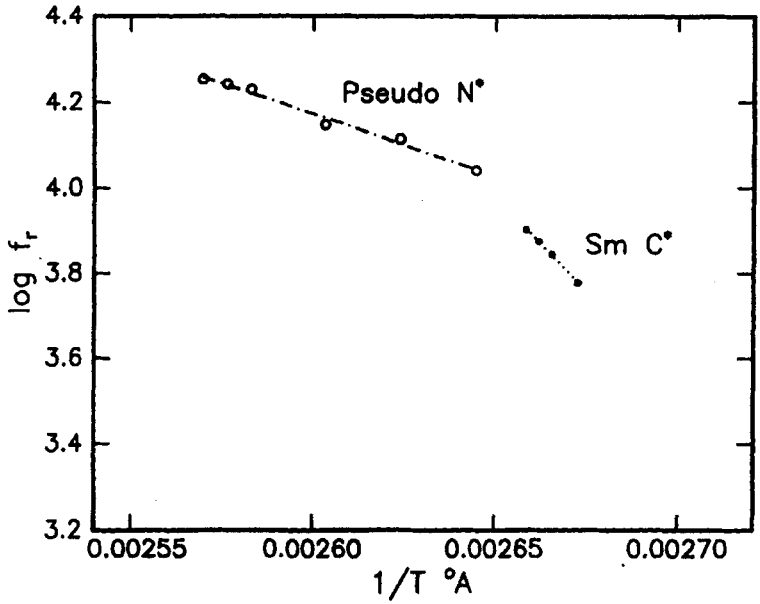


FIGURE 10 Arrhenius plot in pseudo-nematic and smectic C* Phases of DCDP-2

SUMMARY

The salient features from the thermal and ferroelectric investigations on the present ferroelectric isomers are

1. the presence of different phase variant viz., N* - A, A – C* (DCDP-1) and pseudo-N* - C* (DCDP-2)

2. DCDP-1 exhibits a wide liquid crystalline range when compared to its isomer and interestingly, DCDP-2 shows wide ferroelectric thermal range
3. the existence of a ferroelectric pseudo-nematic phase and a field induced transition from ferroelectric pseudo-nematic to nematic phase in DCDP-2
4. The experimental results on the thermal and ferroelectric properties of these isomers suggest a striking influence of the incorporation of polar symmetry in the present linear molecule which inturn promoted, in particular, remarkable ferroelectric behaviour in the bent molecule. The relatively high magnitude of Ps in DCDP-2 can best be accounted on the basis of the different dipolar orientations in the molecular layers due to induced polar symmetry. Nevertheless, the presence of two asymmetric centers coupled with the polar symmetry constitute an important role on the enhancement of the magnitude of spontaneous polarization. A possible explanation towards the role of these two chiral centers can be evinced from the fact that the electron polarization at each asymmetric carbon, achieved by the substitution of highly electronegative chlorine atom, seems to work coherently in the vicinity of elongated delocalized electron cloud. This situation leads to the formation of such an electron cloud between the two asymmetric centers of DCDP-2 serves substantial evidence towards the field-dependent ferroelectric behaviour of the pseudo-nematic phase.

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