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High performance dual frequency liquid crystal compounds

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Some high birefringence ester liquid crystal compounds with cyanate or isothiocyanate polar groups were investigated, the relaxation frequency of which is in the kilo-Hertz region. These compounds are useful for formulating dual-frequency liquid crystal (DFLC) mixtures. The dielectric relaxation and electro-optical properties of these compounds were characterised. Potential applications of these compounds for DFLC mixtures to meet different requirements are discussed.

Keywords: dual-frequency liquid crystal; dielectric relaxation; electro-optical property; birefringence

1. Introduction

Nematic liquid crystals (LCs) have been widely used for amplitude and phase modulations. A critical requirement is response time. The response time of a LC device is determined by cell gap, viscoelastic coefficient, anchoring energy and operating voltage (*1*). Low viscosity high birefringence LCs (*2*) enable a thin cell gap to be employed for achieving fast response time. Driving methods, such as overdrive and undershoot (*3, 4*), have been utilised to accelerate the LC dynamics. However, only the turn-on process can be electrically boosted by applying a high voltage pulse. In order to electrically accelerate the decay process, dual-frequency liquid crystals (DFLCs) have proven useful (*5, 6*). Various electro-optical devices using DFLCs have been developed, e.g. displays (*7, 8*), variable optical attenuators (*9, 10*), spatial light modulators (*11, 12*), tunable polarization filters (*13*), and adaptive optics (*14, 15*).

The dielectric anisotropy ($\Delta\varepsilon$) of a DFLC material can be positive or negative depending on the frequency of the electric field. Therefore, DFLC materials can be used in homogeneous alignment or vertical alignment (VA). For a VA DFLC cell, the turn-on and turn-off time are determined by the LC material properties and applied voltage. The LC dynamics is governed by the balance of electric torque, elastic torque and viscous torque (*1*). It is not easy to obtain analytical solutions. However, under the following assumptions, i.e. strong anchoring boundary conditions, negligible backflow and inertial effects, small reorientation angle and single elastic constant, the LC director's response time can be expressed as follows (*16*):

$$\tau_{on} \sim \frac{\tau_o}{(V_h/V_{th,h})^2 - 1}, \tag{1}$$

$$\tau_{off} \sim \frac{\tau_o}{(V_l/V_{th,l})^2 - 1}, \tag{2}$$

where

$$\tau_0 = \gamma_1 d^2 / K_{33}\pi^2. \tag{3}$$

In Equations (1) and (2), V_h and V_l are the driving voltages for turning-on and turning-off the LC cell, $V_{th,h}$ and $V_{th,l}$ are the threshold voltages of the DFLC at high frequency (as a negative $\Delta\varepsilon$ liquid crystal) and low frequency (as a positive $\Delta\varepsilon$ liquid crystal), respectively, and in Equation (3), γ_1 is the rotational viscosity, K_{33} is the bend elastic constant and d is the cell gap. These equations provide useful guidelines for achieving a faster response time. For instance, to obtain 2π phase modulation, we need $d\Delta n = \lambda$. A higher birefringence can thereby effectively reduce the response time through the use of a thinner cell gap. Similarly, a smaller viscoelastic coefficient is also helpful to achieve a shorter response time. A large dielectric anisotropy would result in a lower threshold voltage, and thereby help to boost the voltage switching ratio (V/V_{th}), which is helpful for achieving fast response time, as revealed by Equations (1) and (2).

DFLC mixtures are composed of two types of LC compounds: the first type has a positive dielectric anisotropy at low frequencies (called positive compounds) but its $\Delta\varepsilon$ decreases with an increase of

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driving frequency because of dielectric relaxation; the second type of compound has a negative dielectric anisotropy (called negative compounds) and its $\Delta\varepsilon$ remains almost constant when the driving frequency is below the MHz range. Positive compounds endow a DFLC mixture with frequency-dependent dielectric anisotropy and positive $\Delta\varepsilon$ at low frequencies, whereas negative compounds give the mixture a large but negative $\Delta\varepsilon$ at high frequencies. The crossover frequency, where the dielectric anisotropy changes sign, is mainly determined by the dielectric relaxation of the positive compounds. The birefringence and viscoelastic coefficient of a DFLC mixture will also be affected by the positive compounds. Therefore, a thorough understanding of the positive compounds is necessary in order to formulate high performance DFLC mixtures.

In this paper, we systematically investigated some positive compounds with low dielectric relaxation frequencies. We doped these compounds in a negative LC mixture and measured the frequency-dependent dielectric anisotropy at a series of temperatures. The birefringence and the effect of these compounds on viscoelastic coefficient were also studied. Our investigation revealed that four-ring single ester cyano (CN) compounds exhibit the lowest relaxation frequency while possessing a large dielectric anisotropy. Thus, they can be utilised to formulate DFLC mixtures operating at an elevated temperature in order to benefit from the reduced viscoelastic coefficient. Three-ring single ester isothiocyanate (NCS) compounds exhibit a low relaxation frequency, small viscoelastic coefficient and high birefringence. These compounds can be employed in high performance DFLC mixtures operating at room temperature. Three-ring double ester CN compounds exhibit a large dielectric anisotropy and relaxation frequency similar to that of three-ring single ester NCS compounds; however, their birefringence is relatively low and viscoelastic coefficient is relatively large. These compounds can be used in DFLC mixtures to achieve large dielectric anisotropy. Three-ring single ester CN compounds exhibit a large dielectric anisotropy and a moderate birefringence, but their relaxation frequencies are the highest among the compounds we compared. These compounds can be used in DFLC mixtures that require a large dielectric anisotropy and high birefringence.

2. Experiments and results

Table 1 lists the molecular structures of the nine positive compounds we investigated. These compounds are grouped into four categories according to their number of rings, number of ester groups and polar groups: three-ring single ester cyanates; three-ring double-ester cyanates; three-ring single ester isothiocyanates; four-ring single ester cyanates. Most of these compounds have lateral fluoro substitutions in order to lower their melting temperatures. In Table 1, we also give abbreviations for each compound, in which P stands for a phenyl ring, C for a cyclohexane ring, E for ester and 3F for a fluoro group at the 3-position.

Mesomorphic properties

DFLC mixtures are intended to be used in their nematic phase, preferably near room temperature. Therefore, the mesomorphic properties, such as melting point and fusion enthalpy, of these compounds will determine the concentration of each compound to be used in the DFLC mixture and the mixture's mesomorphic properties. We used a differential scanning calorimeter (DSC, TA-100) to measure the phase transition temperatures and melting enthalpy of these compounds. The results are listed in Table 2.

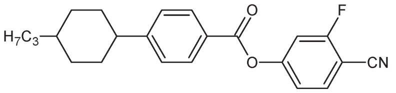
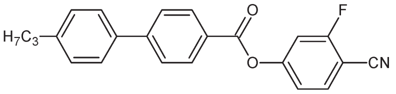
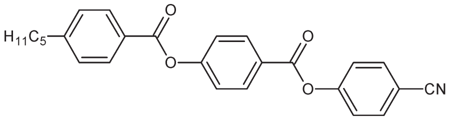
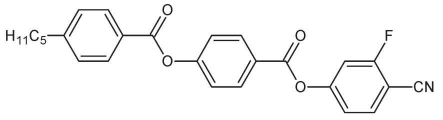
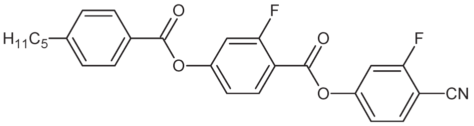
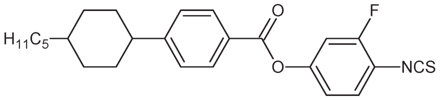
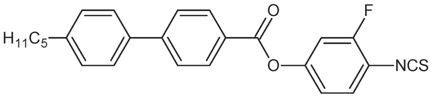
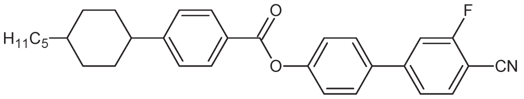
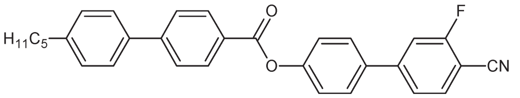
All these compounds show a fairly high crystal-to-nematic transition temperature. The mesomorphic properties of the double ester compounds indicate that the lateral fluoro substitutions help to decrease the melting temperature. The large melting enthalpy of some compounds will definitely limit their solubility in the DFLC mixtures. Therefore, to formulate a DFLC mixture with low melting temperature we employed several positive compounds with different core structures in a negative $\Delta\varepsilon$ LC host.

Dielectric relaxation at various temperatures

We studied the dielectric relaxation of the positive compounds in a negative $\Delta\varepsilon$ LC host, designated as an N-2 mixture. N-2 is a negative $\Delta\varepsilon$ LC mixture consisting of laterally difluorinated tolanes, commercially available from SliChem (China). Its birefringence is 0.15 (at $\lambda = 589\,\text{nm}$ and 20°C) and its dielectric anisotropy is -4.1 (at 1 kHz and 20°C). To study dielectric properties, we doped 10% of each single positive compound into N-2 and measured the mixture's dielectric constants as a function of frequency. The solubility of PEPEP-5CN is poor because of its high melting point and large fusion enthalpy. Therefore, we prepared a binary mixture using 46 wt% PEPEP-5CN and 54 wt% of its homologue PEPEP-3CN, and doped 10 wt% of the binary mixture in N-2.

A doped mixture was filled in two cells with vertical alignment and homogeneous alignment. The

Table 1. Molecular structures and abbreviations of the positive compounds studied.

Group	Abbreviation	Structure
3-ring single ester CN	CPEP(3F)-3CN	
	PPEP(3F)-3CN	
3-ring double ester CN	PEPEP-5CN	
	PEPEP(3F)-5CN	
	PEP(3F)EP(3F)-5CN	
3-ring single ester NCS	CPEP(3F)-5NCS	
	PPEP(3F)-5NCS	
4-ring single ester CN	CPEPP(3F)-5CN	
	PPEPP(3F)-5CN	

dielectric permittivity along ($\varepsilon_{\|}$) and perpendicular ($\varepsilon_{\perp}$) to the principal molecular axis was obtained by comparing the measured capacitance of the cells before and after filling (*17*). The dielectric relaxation of these cells was measured at a series of temperatures. The dielectric properties of pure N-2 were measured the same way. The $\varepsilon_{\|}$ and $\varepsilon_{\perp}$ of N-2 remain constant when the driving frequency is below 1 MHz. Then we calculated the permittivity as a function of driving frequency of each compound by extrapolation.

Table 2. Phase transition temperatures and heat fusion enthalpies of the positive compounds studied.

Compound	T_m /°C	T_c /°C	ΔH /kcal mol^{-1}
CPEP(3F)-3CN	100	201	4.7
PPEP(3F)-3CN	96	201	5.5
PEPEP-5CN	122	240	7.3
PEPEP(3F)-5CN	100	214	6.3
PEP(3F)EP(3F)-5CN	89	202	6.7
CPEP(3F)-5NCS	86	219	5.0
PPEP(3F)-5NCS[a]	88	220	3.2
CPEPP(3F)-5CN	105	361	6.9
PPEPP(3F)-5CN	91	346	5.9

[a] PPEP(3F)-5NCS has a smectic phase; the smectic to nematic transition temperature is 175°C.

Figures 1(a) and 1(b) show the frequency-dependent $\varepsilon_{\|}$ and $\varepsilon_{\perp}$, respectively, of CPEP(3F)-5NCS at various temperatures. We used the Debye equation,

$$\varepsilon_{\|}(f) = \varepsilon(\infty) + \frac{\varepsilon(0) - \varepsilon(\infty)}{1 + (f/f_c)^2}, \qquad (4)$$

to fit the relaxation frequencies at each temperature and then derived the activation energy of these compounds in N-2. In Equation (4), $\varepsilon(0)$ is the permittivity at dc voltage, $\varepsilon(\infty)$ is the permittivity in the high frequency limit, f is the frequency of the driving voltage and f_c is the relaxation frequency. When we compared the dielectric relaxation of different compounds, we doped 10% of the single compound with 90% of N-2 (negative $\Delta\varepsilon$ host).

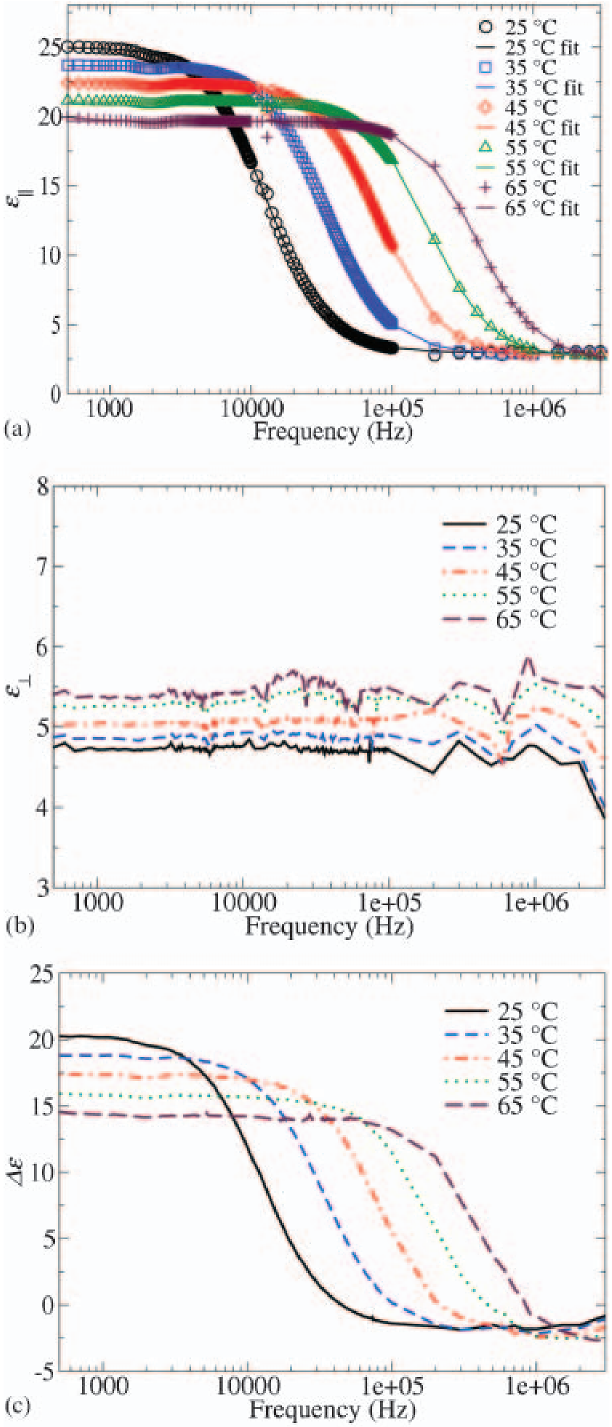


Figure 1. Dielectric permittivity of CPEP(3F)5NCS as a function of driving frequency at a series of temperatures along the director direction (a) and perpendicular to the director direction (b); dielectric anisotropy of CPEP(3F)5NCS as a function of driving frequency at a series of temperatures (c).

Therefore, the Debye equation is still applicable. In the case of PEPEP-3CN and PEPEP-5CN, their relaxation behaviour is also very close because of similar core structure. As a result, the Debye equation still provides a nice fit. As can be seen in Figure 1(b), $\varepsilon_{\perp}$ remains almost unchanged when the driving frequency is below 1 MHz. The dielectric anisotropy of CPEP(3F)-5NCS as a function of frequency is shown in figure 1c.

As can be seen in Figure 1c, the dielectric relaxation of CPEP(3F)-5NCS is temperature dependent. As the temperature increases, $\varepsilon_{\parallel}$ decreases in the low-frequency region, whereas $\varepsilon_{\perp}$ increases for all the frequencies measured. The relaxation frequency increases with temperature according to the following equation (*6*):

$$f_c = A\exp(-E/kT), \qquad (6)$$

where A is a material constant, k is the Boltzmann constant, E is the activation energy and T is the temperature in Kelvin. The dielectric relaxation properties and temperature dependence of the other positive compounds were similar to those of CPEP(3F)-5NCS.

The relaxation frequency of the positive compounds largely determines the crossover frequency and thereby the operating conditions of the DFLC mixture. A low crossover frequency is preferred because it decreases the operational frequency. If the crossover frequency is too high, the required high frequency to drive the DFLC may fall into the intermediate region where the dielectric relaxation is not yet finished. In this situation, the dielectric heating effect (*17, 18*) may take place which, in turn, shifts the crossover frequency. Moreover, to generate high-frequency voltages would require a more sophisticated circuit design.

Figure 2 shows the relaxation frequency of the positive compounds at different temperatures. The rigid core structure plays the most important role in determining the relaxation frequency. The four-ring single ester CN compounds exhibit the lowest relaxation frequency in N-2 host. The three-ring single ester NCS compounds and the three-ring double ester CN compounds exhibit similar dielectric relaxation frequencies in the measurement temperature range. Their relaxation frequencies are higher than those of CPEPP(3F)-5CN and PPEPP(3F)-5CN. The three-ring single ester CN compounds have the highest relaxation frequencies.

Table 3 lists the measured dielectric anisotropy of the positive compounds at low and high frequency limits at 25°C. Also included are the relaxation frequencies at 25°C and the activation energy of these compounds. The three-ring single ester NCS compounds have the smallest dielectric anisotropy, whereas the CN compounds exhibit a much higher

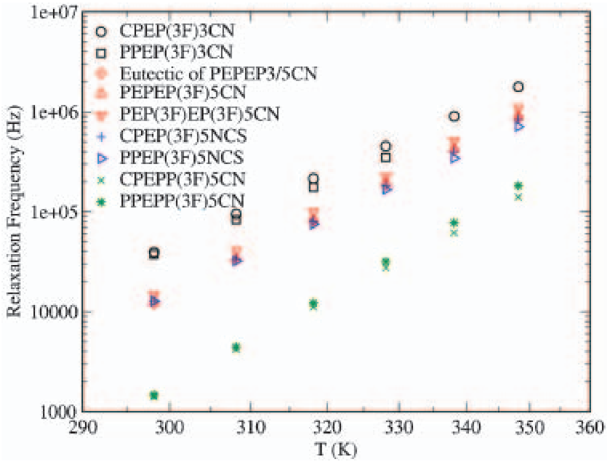


Figure 2. Relaxation frequencies as a function of temperature of the positive compounds. Note that the relaxation frequency is in logarithmic scale, and the temperature is Kelvin and in reciprocal scale.

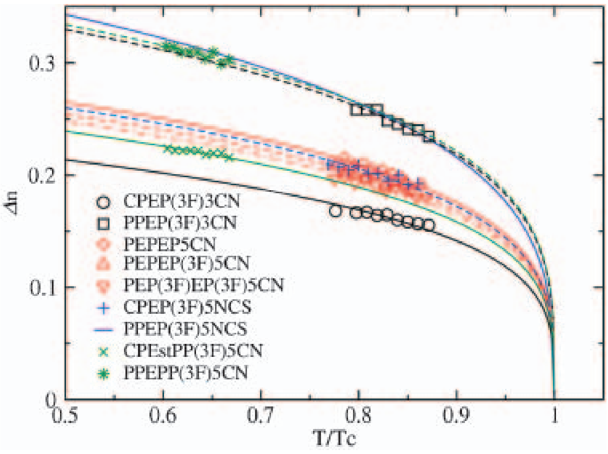


Figure 3. The optical birefringence of the positive compounds as a function of reduced temperature, T/T_c. T and T_c are both thermal dynamic temperatures.

dielectric anisotropy. This is because CN has a stronger dipole moment than NCS. The three-ring double ester CN compounds have a larger Δε than other compounds. The fluoro substitution not only lowers the melting point but also enhances the dielectric anisotropy. The trade-off is the higher rotational viscosity.

Electro-optical properties

The birefringence and viscoelastic coefficient play important roles in determining the response time of a DFLC cell, as shown in Equations (1)–(3). We measured the birefringence of the positive compounds in their individual nematic phase, despite their high melting point. The single compound was heated to a nematic temperature and then filled into an ITO (indium tin oxide) glass cell with antiparallel rubbings. The temperature-dependent birefringence of each compound was measured using a He–Ne laser ($\lambda = 633$ nm). Because of its high smectic to nematic transition temperature, the birefringence of PPEP(3F)-5NCS was derived from the guest–host method. We doped 25% of PPEP(3F)-5NCS into E44 (Merck) and measured the birefringence of the mixture at various temperatures. The birefringence of E44 at these temperatures was also measured. Comparing the results at the same reduced temperatures (T/T_c, where T_c is the clearing point of the mixture), the birefringence of PPEP(3F)-5NCS can be derived.

Figure 3 shows Δn as a function of reduced temperature for these compounds. The birefringence depends mostly on the π-electron conjugation length (*19, 20*). Therefore, compounds with a cyclohexane ring exhibit a lower birefringence than similar compounds with a phenyl ring. The isothiocyanate compounds exhibit a higher birefringence than the corresponding cyano compounds. When functioning as a component in a nematic mixture with clearing point 140°C, the birefringence exhibited by a single compound is listed in Table 4.

Table 3. Measured dielectric relaxation properties, relaxation frequencies (f_c), and activation energies (E) of the positive compounds.

Compound	Δε(0), 25°C	Δε(∞), 25°C	f_c /kHz, 25 °C	E /meV
CPEP(3F)-3CN	41.5	−4.3	39.2	677
PPEP(3F)-3CN	43.5	−5.5	37.4	623
PEPEP-3/5CN binary	45.0	−4.4	12.2	759
PEPEP(3F)-5CN	56.5	−6.3	12.7	761
PEP(3F)EP(3F)-5CN	69.1	−4.0	14.8	776
CPEP(3F)-5NCS	20.3	−1.7	12.9	749
PPEP(3F)-5NCS	21.4	−2.6	12.8	720
CPEPP(3F)-5CN	33.6	−3.5	1.4	817
PPEPP(3F)-5CN	35.6	−5.3	1.5	865

Table 4. Birefringence, Δn, of the positive compounds at 25°C when functioning in a nematic mixture with clearing point 140°C; γ_1/K_{11} of the E44 doped with 10% of the compounds studied at 25°C. Also included for comparison is E44, a Merck mixture with high Δn.

Compound	Δn at 25°C	γ_1/K_{11} /ms µm^{-2} (25°C)
CPEP(3F)-3CN	0.184	28.7
PPEP(3F)-3CN	0.284	29.8
PEPEP-5CN	0.228	32.3
PEPEP(3F)-5CN	0.219	32.1
PEP(3F)EP(3F)-5CN	0.213	28.9
CPEP(3F)-5NCS	0.224	23.5
PPEP(3F)-5NCS	0.290	24.6
CPEPP(3F)-5CN	0.206	24.7
PPEPP(3F)-5CN	0.288	31.4
E44	0.242	23.1

Table 5. Dielectric relaxation parameters and electro-optical properties of DFLC-A and DFLC-B at a series of temperatures.

	DFLC-A				DFLC-B			
T /°C	25	35	45	55	25	35	45	55
Δn (at 633 nm)	0.174	0.166	0.160	0.159	0.179	0.177	0.178	0.173
γ_1/K_{11} /ms μm^{-2}	40.95	20.73	10.98	8.42	28.1	15.87	9.17	7.62
FoM /μm^2 s^{-1}	0.74	1.33	2.33	3.01	1.14	1.98	3.45	3.94
f_c /kHz[a]	0.87	2.6	7.3	18.8	11.3	29.3	67.9	147.5
$\Delta\varepsilon(0)$	3.72	3.53	3.35	3.28	4.37	4.04	3.66	3.35
$\Delta\varepsilon(\infty)$	−4.50	−4.20	−3.87	−3.52	−3.68	−3.46	−3.27	−3.04

[a] f_c is the crossover frequency.

To evaluate the viscoelastic coefficient of these positive compounds, we doped 10% of each compound in E44 (Merck) and measured the viscoelastic coefficient of the doped mixture. Results are presented in Table 4. The viscoelastic coefficient of E44 is also listed for comparison. With the introduction of the positive compounds, the viscoelastic coefficient of the mixture increases. The rotational viscosity depends on the moment of inertia of the LC compound, molecular conformation and intermolecular interactions (*21*). The NCS compounds and CPEPP(3F)-5CN result in the least increase to γ_1/K_{11}, whereas PPEPP(3F)-5CN and the double ester compounds cause a significant increase. A large viscoelastic coefficient leads to a slow response time.

Performance in DFLC mixtures

We formulated two DFLC mixtures to demonstrate the performance of the most promising positive compounds in DFLC mixtures. DFLC-A consists of 80% N-2 and 20% binary mixture of CPEPP(3F)-5CN and PPEPP(3F)-5CN. DFLC-B consists 70% N-2 and 30% binary mixture of CPEP(3F)-5NCS and PPEP(3F)-5NCS. The dielectric relaxation and electro-optical properties of these mixtures were characterised at a series of temperatures and the results are presented in Table 5 and Figure 4.

As can be seen in Table 5, DFLC-B exhibits higher birefringence and lower viscoelastic coefficient than DFLC-A. We attribute the higher birefringence of DFLC-B to the higher concentration of large birefringence positive compounds. The lower viscoelastic coefficient of DFLC-B originates from the lower viscoelastic coefficient of the single ester NCS compounds. Therefore, devices employing DFLC-B can respond faster than similar devices employing DFLC-A when operating at the same temperature. However, at elevated temperatures, the crossover frequency of DFLC-B is so high that the frequency required to drive the mixture as a negative $\Delta\varepsilon$ liquid crystal is too high for practical applications. Therefore, DFLC-B is more suitable for operating at room temperature. On the other hand, Figure 4 shows that the crossover frequency of DFLC-A is substantially lower than that of DFLC-B. At some elevated temperatures, the crossover frequency of

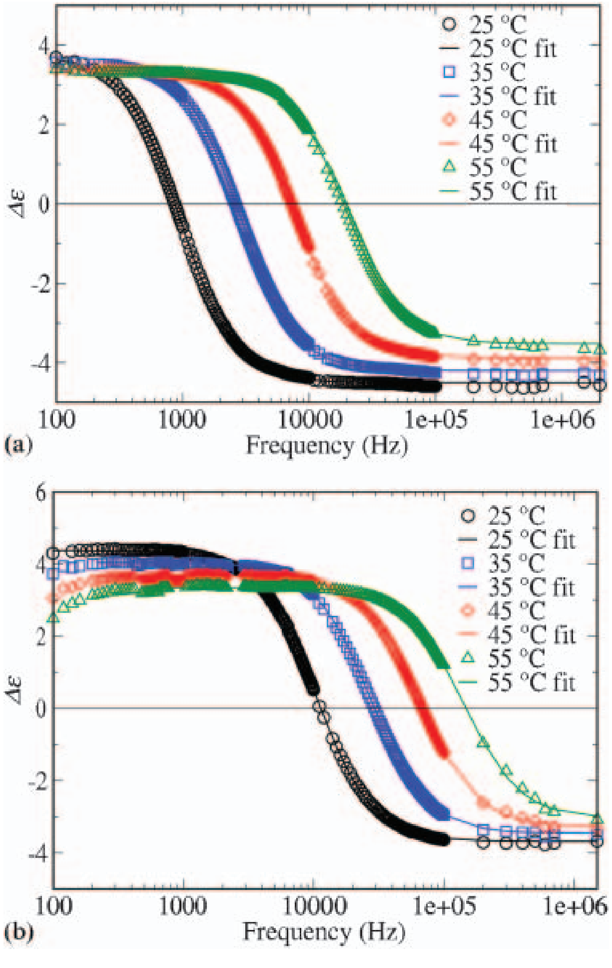


Figure 4. Dielectric relaxation of DFLC-A (a) and DFLC-B (b) at a series of temperatures. The symbols are experimental data and the lines are fittings to the Debye equation. The two positive compounds in each mixture exhibit similar relaxation frequencies in N-2. Therefore, the Debye equation with single relaxation frequency still works well. Note that the fitting starts at higher frequency when the temperature is higher to avoid effects from ionic impurities.

DFLC-A is still not very high, and the high driving frequency required to address the mixture as negative $\Delta\varepsilon$ liquid crystal is still acceptable. As a result, DFLC-B can be operated at elevated temperatures to benefit from the reduced viscoelastic coefficient. If we set the high driving frequency and thereby the crossover frequency at a certain value, DFLC-A can be operated at a higher temperature than DFLC-B. In this case, the figure of merit, $\mathrm{FoM} \equiv K_{33}(\Delta n)^2/\gamma_1$ (*22*), of DFLC-A will be higher than that of DFLC-B owing to the greatly reduced viscoelastic coefficient at a higher operating temperature. Therefore, DFLC-B is more suitable for devices operating at elevated temperatures.

3. Conclusion

We have investigated the dielectric relaxation and electro-optical properties of some low relaxation frequency positive compounds for DFLCs. The high melting temperature and relatively large melting enthalpy of most compounds indicate that adoption of homologues and compounds with varied core structure are necessary to achieve a desirable nematic range. In all these compounds, the three-ring single ester NCS compounds exhibit a low relaxation frequency, high birefringence and small viscoelastic coefficient, which make these compounds suitable for a high performance DFLC mixture operating at room temperature. The four-ring single ester CN compounds exhibit a very low relaxation frequency, large dielectric anisotropy and high birefringence. These compounds can be utilised to formulate DFLC mixtures operating at elevated temperatures. The viscoelastic coefficient is greatly reduced at higher temperature, resulting in a much shorter response time. However, the crossover frequency increases as the temperature increases. Thus, we need to design a low crossover frequency ($f_c \sim 1$ kHz) DFLC at room temperature so that we can operate the DFLC at an elevated temperature while still keeping a relatively low crossover frequency, say $f_c < 5$ kHz). Operating a DFLC at an elevated temperature would greatly reduce the viscoelastic coefficient. The three-ring double ester compounds have a relaxation frequency similar to that of three-ring single ester NCS compounds, but have a relatively low birefringence and large viscoelastic coefficient. These compounds are useful for the DFLC mixtures that require a large dielectric anisotropy and low crossover frequency. The three-ring single ester CN compounds have a large dielectric anisotropy and high birefringence, but their viscoelastic coefficient is relatively large. As a result, these compounds are more suitable for DFLC mixtures operating at room temperature to increase the low frequency $\Delta\varepsilon$ and help to improve mesomorphic properties.

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