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Diluters' effects on high Δn and low-viscosity negative $\Delta\varepsilon$ terphenyl liquid crystals

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Lateral difluoro terphenyls exhibit a high birefringence, fairly low viscosity and modest dielectric anisotropy, but their alignment in a vertical alignment (VA) cell often deteriorates at elevated temperatures if their concentration is high. We have studied the effects of six diluters for lowering the viscosity while stabilizing the VA of the terphenyl host mixture at elevated temperatures. The pros and cons of each diluter are analysed.

Keywords: high birefringence; fluorinated terphenyl; liquid crystals; vertical alignment

1. Introduction

It took almost 30 years from the discovery of the vertical alignment (VA) mode in the early 1970s (see (*1*)) to a fully commercial product by the end of the past decade (*2*). A multi-domain VA mode liquid crystal display (LCD) produced commercially for the first time by Fujitsu, exhibited an unprecedented contrast ratio compared with other technologies available at that time. Further development of the VA mode was marked by advanced super VA (ASV) mode (*3*) and patterned VA (PVA) also referred to as a type of multidomain VA (MVA) (*4*). At present, the VA mode stands for an excellent contrast ratio (*5*). The VA mode also dominates the reflective liquid crystal on silicon (LCoS) projectors market (*6*). Although the excellent contrast ratio makes VA a preferred choice for display applications, especially large format LCD TVs, the response time of the negative dielectric anisotropy remains a technical challenge.

A uniform surface alignment is a prerequisite for producing a high-quality LCD. Both organic (polyimide) and inorganic (SiO_x) alignment layers have been used for aligning negative $\Delta\varepsilon$ liquid crystal (LC) mixtures. For large-screen direct-view LCDs, buffed polyimide is a favourable choice because of its low cost and manufacturability. For projection LCDs, SiO_x inorganic alignment layers are more robust than buffed polyimide to high-power arc lamp illumination (*6*). Depending on the type of polyimide and buffing conditions, the anchoring energy could vary from strong to moderate. Although the switching time of VA modes is relatively fast, further improvement is still required, especially for greyscale switching. The response time of existing $-\Delta\varepsilon$ mixtures may not fully support the emerging 240 Hz refresh rate or colour sequential operation. To address the LCD response time one should consider birefringence and rotational viscosity effects. With this motivation we explore laterally fluorinated terphenyl LCs.

2. Experimental details

Several techniques were used to characterize the physical properties of the single terphenyl compounds and mixtures. Differential scanning calorimetry (DSC; TA Instrument Model Q-100) was used to determine the phase transition temperatures. Results were obtained from 3–6 mg samples in the heating and cooling cycles at a scanning rate of 2°C min^{-1}. The birefringence was derived from phase retardation measurement of 5-μm homeotropic cells (pretilt angle ~87°) using a He–Ne laser (λ = 633 nm) (*7*). At a given temperature, the phase retardation is related to the cell gap d, birefringence Δn and wavelength λ as

$$\delta = 2\pi d\Delta n/\lambda. \quad (1)$$

Each compound performance was compared based on the figure-of-merit defined as (*8*)

$$FoM = K_{33}\Delta n^2/\gamma_1, \quad (2)$$

where K_{33} is the bend elastic constant and γ_1 is the rotational viscosity. The temperature-dependent birefringence of a LC can bse described as follows:

$$\Delta n = \Delta n_0 S, \quad (3)$$

$$S = (1 - T/T_c)^{\beta}, \quad (4)$$

where Δn_0 is the birefringence at T = 0 K, S is the order parameter, β is a material constant and T_c is the

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clearing temperature of the LC. Here, Δn_0 and β were obtained by fitting the experimental data using Equations (3) and (4). Once these two parameters are determined, the LC birefringence at any temperature can be extrapolated. Using Equation (2) and knowing that $K_{33} \sim S^2$ and $\gamma_1 \sim S \cdot \exp(E/kT)$, FoM can be expressed as follows (*9*):

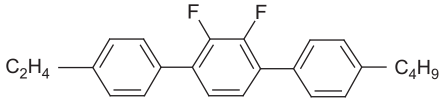
$$FoM = a(\Delta n_0)^2 \left(1 - \frac{T}{T_c}\right)^{3\beta} \exp\left(\frac{-E}{\kappa T}\right), \qquad (5)$$

where a is a proportionality constant. Figure of Merit (FoM) is commonly used to compare the performance of a LC compound or mixture because it is independent of the cell gap employed. The dielectric and elastic constants of the mixtures were measured using the capacitance method (*9*, *10*) of a single homeotropic cell, using a computer controlled LCAS II (LC-Vision) instrument.

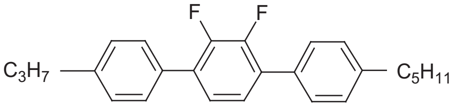
3. Results and discussion

3.1 All terphenyl host mixture

Laterally fluorinated terphenyl compounds exhibit a high birefringence and relatively low rotational viscosity (*11–15*). Therefore, they may be good candidates for improving the LC response time through cell gap reduction while no penalty will be paid in rotational viscosity. Despite the merit of utilizing laterally fluorinated terphenyl compounds a severe drawback of difficult alignment needs to be addressed. A 4-alkyl-4”alkyl-2’,3’-difluoro-[1,1’;4’,1”]-terphenyl as a general structure was chosen for its low viscosity. Two homologues (structures I and II) were studied:



Structure I,



Structure II.

These two homologues are pure nematics and their melting and clearing temperatures (in °C) are [56, 100] and [55, 123], respectively. To lower the melting temperature, we formulated a binary eutectic mixture which consists of 35:65 ratio by weight of the -24 (4”-butyl-4-ethyl-2’,3’-difluoro-[1,1’4’,1”]terphenyl) and -35 (4”-pentyl-4-propyl-2’,3’-difluoro-[1,1’4’,1”]terphenyl) homologues as a host mixture for our further study on the low molecular mass dopant compounds, the diluters. The nematic range of the host mixture extends from 24.4 to 116°C, with a strong super-cooling effect down to around 4.6°C which allows us to conduct measurements at room temperature (approximately 23°C).

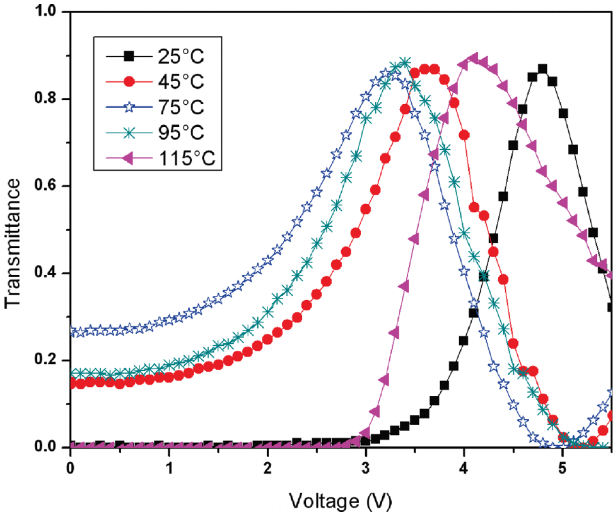


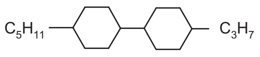
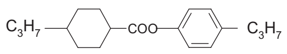
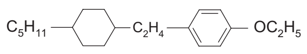
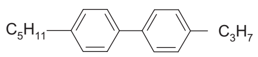
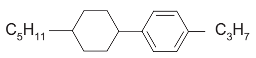
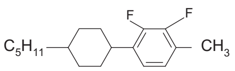
Figure 1. Voltage-dependent transmittance of the terphenyl host mixture at different temperatures.

Figure 1 shows the voltage-dependent transmittance (VT) curve of the terphenyl binary mixture at different temperatures. The light leakage at V = 0 becomes noticeable as the temperature increases, and becomes worst at 75°C. After that, the light leakage drops and returns 0 at 115°C. We assume that the surface tension of the binary mixture on polyimide decreases when the temperature increases, but increases when the temperature is close to the clearing point (*16*). From the van der Waals interaction model of LC molecules and the polyimide layer (*17*), surface tension theory can explain the LC orientation: substrates with surface energy lower than the LC surface tension cause homeotropic alignment; otherwise alignment is parallel to the substrate plane, following any long-range order (e.g. grooves) present on the surface (*18*). We assume that at high temperature, when light leakage is not zero at V = 0, the LC directors have a high pretilt angle as compared with normal VA. In order to improve alignment of the highly birefringent terphenyl host mixture, a low molecular mass component has to be doped. Therefore we studied the performance of six different compounds classified as ‘diluters’ to the high birefringence terphenyl host.

3.2 Diluter effect on all terphenyl host mixture

Table 1 lists the six diluter compounds used in our experiment. It is critically important for the mixture formulation to preserve the high clearing temperature so that the physical properties are less sensitive to the temperature fluctuations. Therefore, we carefully

Table 1. Diluter compounds structures and physical properties.

No	Compound	Δn	T_c^1(°C)	T_c^2 (°C)
1	(*19*)  CC53	0.0399	97.8	72.8
2	(*20*)  CEstP33	0.0620	34.0	33.6
3	(*21*)  CEtP5O2	0.0791	46.2	37.4
4	(*20*)  PP53	0.1384	49.1	15.9
5	(*22*)  CP53	0.0330	–5.6	–1.0
6	(*23*)  CPFF51	0.0318	16.0	–10.1

T_C^1 is diluter clearing point temperature as the measured by DSC method for 100% concentration.
T_C^2 are the values extrapolated from the guest-host system with terphenyl.
Δn are extrapolated from 20wt% guest-host measured by Abbe refractrometer.

evaluated each diluter's effects on the clearing point temperature of the terphenyl host mixture. Table 1 also lists the clearing point of each diluter measured by DSC (T_C^1) for its 100% concentration, the clearing point extrapolated from the guest–host systems with binary terphenyl mixture based on 20% molar concentration (T_C^2). In all six cases, the T_C^2 extrapolated from the system with terphenyl host are lower than directly measured T_C^1 (DSC data). This leads us to a conclusion that when low molecular mass dopant is mixed with relatively large molecular mass terphenyl host, it affects the clearing point of the system more than predicted based on a molar ratio of the components. We observed that the linear T_C add-up relationship does not apply in the reported cases. A similar conclusion can be made if the directly measured clearing point temperature T_C^1 is compared with the available literature data for different host systems (*19–23*). Although in most cases, the extrapolated T_C^2 values are close to the one obtained from ZLI-type host mixtures (*19–23*), a significant discrepancy was observed in the case of compounds 4 and 6. This implies to the non-additive behaviour with our host, i.e. the T_C of a mixture doped with compounds 4 or 6 could not be projected directly.

Figure 2 shows the clearing point temperature change of the guest-host systems as the diluter's concentration increases. From Figure 2, we find that compound 1 has the least effect. Even at 30 wt%, T_C only drops by 18°C. In contrast, compound 6 destabilized the nematic phase most effectively among the six diluters investigated; at 30 wt% the T_C of the mixture drops by 48°C. Therefore, we shortly conclude that compound 1 is the most attractive for mixture formulation if the clearing temperature standpoint is considered and terphenyl mixture is a host medium. The clearing point of the guest–host system decreases almost linearly with increased amount of diluter.

Figure 3 depicts the diluter's concentration-dependent birefringence. Similarly to T_C, birefringence decreases linearly with increasing concentration of a diluter. In this category, the most conjugated diluter, compound 4, stands out as the structure with the least effect on the system's birefringence. If the choice is purely based on birefringence, biphenyl-core-based diluter is favoured. Although the birefringence of compound 1 is small due to the absence of unsaturated bonds, its effect on Δn is still moderate because of its weak effect on T_C. Rotational viscosity γ_1 plays a crucial role in LCD response time. The response time of a nematic LC device is linearly proportional to γ_1. The rotational viscosity of an aligned LC depends on

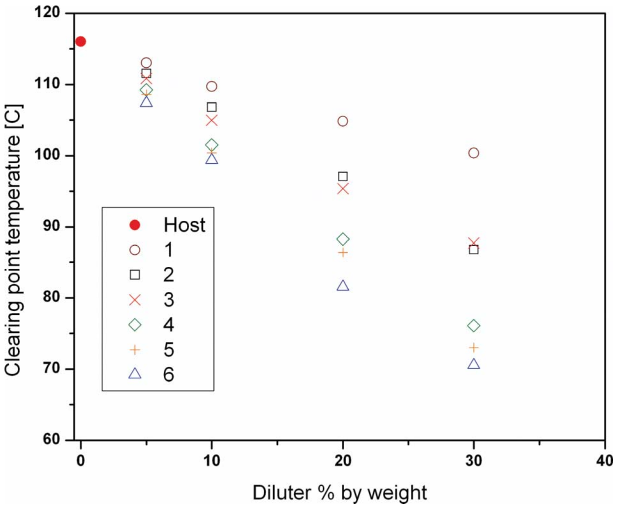


Figure 2. Measured clearing point versus diluter concentration.

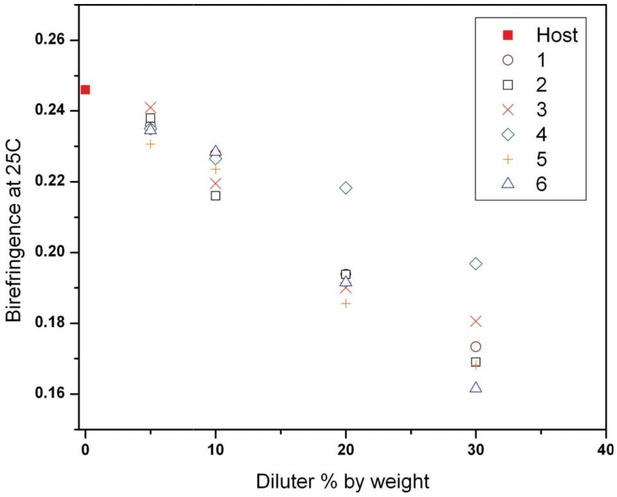


Figure 3. Birefringence versus diluter concentration.

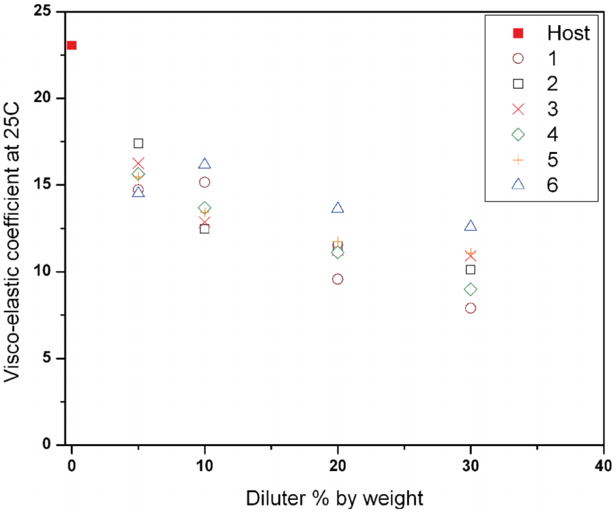


Figure 4. Viscoelastic coefficient versus diluter concentration.

the detailed molecular constituents structure, moment of inertia, activation energy and temperature.

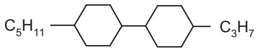
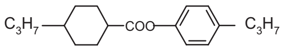
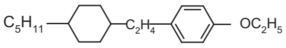
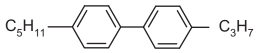
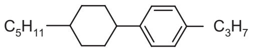
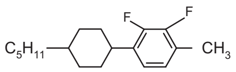
Figure 4 shows the dopant-percentage-dependent viscoelastic coefficient (γ_1/K_{33}). Rotational viscosity decreases sharply with temperature increase. Meanwhile, K_{33} decreases at the same time. Although both parameters shows similar tendency, elastic constant decreases at a slower pace right until a few degrees before clearing point. Measured for different concentration of each dopant, viscoelastic coefficient shows tendency to saturate above a certain amount of dopant is included into the mixture. Compound 1 causes the biggest decrease in γ_1/K_{33} among the investigated structures, followed by biphenyl-based compound 4. With no surprise, compound 6 shows the highest values of γ_1/K_{33} due to the lateral fluorination heavily impacting rotational viscosity. All other compounds show similar values of γ_1/K_{33}. FoM results from birefringence and the viscoelastic coefficient allow us to compare the performance of the different LC mixtures regardless of the optimal cell gap.

Table 2 shows dielectric anisotropy, threshold voltage, bend elastic constant K_{33} and rotational viscosity measured for guest–host systems with 30 wt% diluter. Data was collected at room temperature conditions. Among the structures investigated, compounds 1 and 3 show relatively high clearing point, therefore, we observed a relatively large K_{33}, which results in low viscoelastic coefficient; compounds 4 and 5 have low melting points; and compound 6 has the smallest threshold voltage. Figure 5 shows dopant-concentration-dependent FoM compared at 25°C. As the diluter concentration increases, it becomes evident that FoM favours birefringence because FoM depends on $(\Delta n)^2$ (Equation (2)). From the FoM values shown in Figure 5, when concentration exceeds 20 wt%, compound 4 is the most favourable for mixture formulations if the FoM standpoint is considered. The drawback in this case is a severe destabilization of the nematic phase resulting in a lower T_C. FoM values of the system with biphenyl compound 4 is followed closely by the system with bicyclohexyl-based compound 1. Bicyclohexyl dopant only slightly affects T_C, significantly improves viscoelastic coefficient γ_1/K_{33} but falls short in the birefringence category.

3.3 Molecular modelling of a diluter component

Molecular modelling has been proven to help in understanding the phenomena observed experimentally. Therefore, we developed a model of investigated diluter component using HyperChem (v.7) single PC software. A semi-empirical method of modified neglect of diatomic overlap was used with Polak–Ribiere geometry optimization algorithm. As a result, a single molecule was calculated *in vacuo* to its possibly lowest energy gradient, which suggests the most likely configuration of the molecule. Calculation was conducted until the total energy gradient of 1×10^{-5} kcal mol^{-1} Å^{-1} calculated as a root-mean-square value. The energy gradient is the rate of change (first derivative) of total energy with respect to displacement of each atom in the *x*, *y* and *z* directions. Table 3 lists diluter compounds with calculated dipole moments, mean polarizability values, and the length of the molecule. Based on dipole moment values we divide diluter compounds into two categories: the first with distinct dipole moment and the second with dipole moment

Table 2. Physical properties of the guest–host systems with 30 wt% diluters dopped into the terphenyl host.

Number	Compound	$\Delta\varepsilon$	V_{th}	K_{33}	γ_1 (mPas)
1	(*19*)  CC53	−1.2	3.898	16.4	129.4
2	(*20*)  CEstP33	–	–	–	–
3	(*21*)  CEtP5O2	−1.36	3.787	17.5	191.1
4	(*20*)  PP53	−1.07	3.083	9.13	82.0
5	(*22*)  CP53	−1.06	3.57	12.1	134.1
6	(*23*)  CPFF51	−2.03	2.328	9.86	124.1

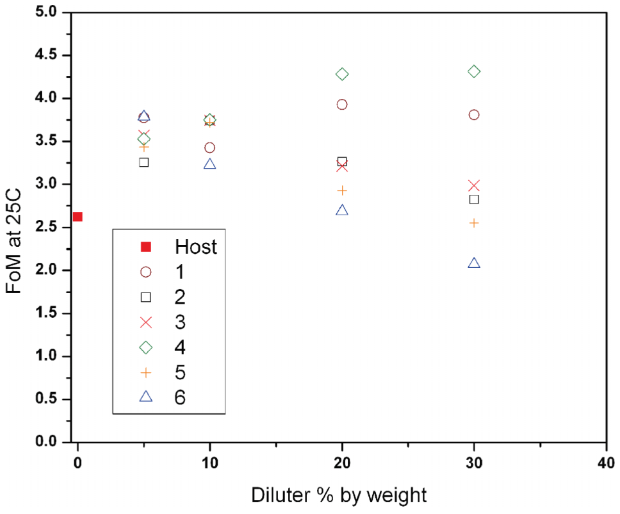


Figure 5. FoM versus diluter concentration.

Table 3. Molecular modelling results for the investigated diluter compounds. An exemplary terphenyl compound is included for comparison.

Compound	Dipol moment (μ)	Mean polarizability (a.u.)	Length (Å)	Conjugation length (Å)
CC53	0.049	154.3	16.1	–
CEstP33	1.826	163.8	15.2	6.7
CEtP5O2	1.248	176.5	18.8	6.6
PP53	0.054	176.7	17.1	10.2
CP53	0.054	163.5	16.6	5.9
CPFF51	3.110	150.0	14.6	6.0
PPP24*	3.326	224.9	18.6	14.6

*PPP24 stands for 2’,3’-difluoro-4-ethyl-4”butyl-[1,1’;4’,1”]-terphenyl host mixture’s compound.

value almost zero. Compounds 1, 4 and 5 do not have a molecular constituent enabling a strong dipole. A calculated isosurface of electrostatic potential of these structures is uniform and rather neutral, as shown in Figure 6(a). The introduction of elements with high electronegativity results in significant disturbance in electrostatic potential. Shown as an example,

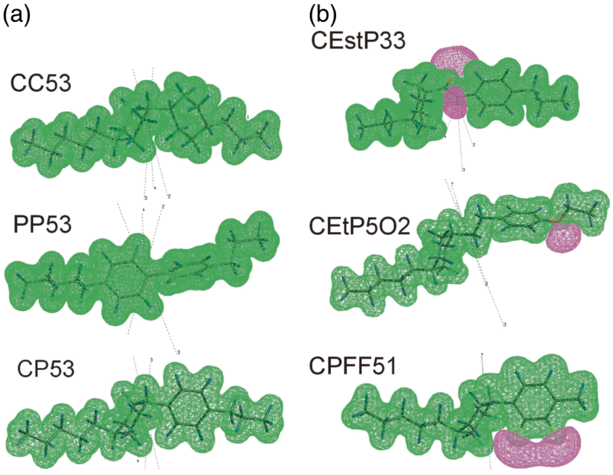


Figure 6. A calculated isosurface of electrostatic potential for six investigated diluter compounds.

compound 6 is modelled containing two laterally substituted fluorine atoms into the phenyl ring's positions 2 and 3. Electronegativity of fluorine is known to be four and is the highest among elements of the periodic table. As carbon's electronegativity is 2.5 and oxygen's is 3.5, both of which are lower than that of fluorine, we observe distinct negative charge accumulating within the space associated with lateral fluorine, as shown in Figure 6(b).

The large dipole moment calculated for compounds 2, 3 and 6 is a natural consequence of the pull effect of electrons; see Table 3. Considering a LC mixture threshold and operational voltage we may place more value on a diluter compound with large dipole moment perpendicular to the long molecular axis, such as compound 6. In such a case, the overall dielectric anisotropy of the mixture should not be affected much as long as the clearing point of the mixture remains relatively high, which is shown in Table 2. Although the dipole moment of compound 6 is relatively large, even a small amount of this diluter affects the clearing point temperature of the mixture the most among investigated structures (see Figure 1). Therefore, the merit of a strong dipole is diminished significantly. In contrast, the nearly neutral compound 1 shows relatively small effect on the clearing point temperature of the mixture. The drawback of using compound 1 as a dopant is the negative effect on the mixture's dielectric and optical anisotropy. The classical Lorentz–Lorenz equation (see (*24*)) correlates the refractive index of an isotropic media with molecular polarizability in the optical frequencies. We calculated the mean polarizability for modelled structures which follows the π-electron conjugation length of the structures. Compounds with longer π-electron conjugation show higher polarizability and birefringence as a macroscopic property (*25–27*). Considering polarizability and resulting birefringence, compound 4 will be the preferable choice as its π-electron conjugation length is the longest among diluters investigated while its overall length is the mid-range. The viscoelastic coefficient of compound 4 is relatively low which results in high overall value of FoM. However, again a critical drawback for the biphenyl core diluter compound is the relatively low clearing point temperature. Therefore it may not be used in excess amounts as a dopant to the host mixture.

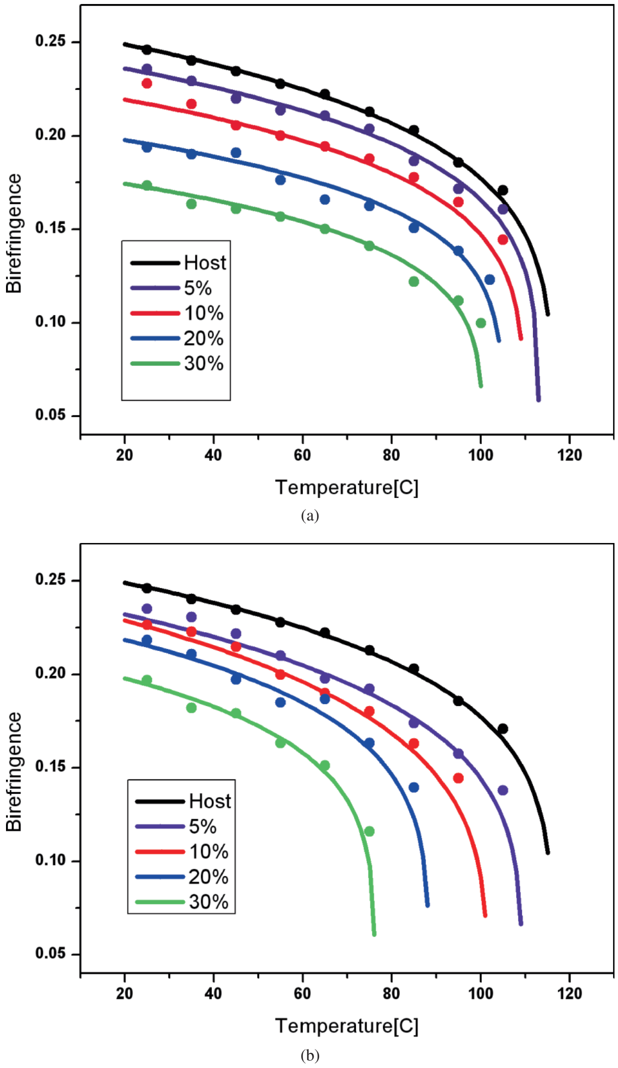
Considering all of the collected data, we conclude at this point that compounds 1 and 4 could be the most suitable dopants for high Δn, negative $\Delta\varepsilon$ terphenyl-based mixtures. Therefore, we present their temperature-dependent birefringence, viscoelastic and FoM in detail.

4. Temperature effect on bicyclohexyl and biphenyl doped systems

Temperature has a major influence on LCD performance. For handheld displays, the ambient temperature could vary significantly, depending on the geographic locations. For a projection display, the lamp-induced thermal effect could heat up the LCD panel to 50°C or higher. As the temperature increases, the birefringence, dielectric anisotropy, elastic constants and viscosity all drop, but at different rates. In a notebook or desktop computer, the LC operation temperature is limited to 35°C due to the low-power backlight employed. Therefore it is critically important to know the exact behaviour of the dopant component through the entire operational temperature range.

Figure 7 shows a comparison of the temperature-dependent birefringence for stepwise increased amounts of compound 1 (Figure 7(a)) and compound 4 (Figure 7(b)) doped into the terphenyl host. Birefringence measured for binary terphenyl host mixture is included to visualize the effect of a dopant added. As mentioned before, the guest–host's birefringence decreases linearly as the diluter's concentration increases. Therefore, every additional amount of a diluter added decreases birefringence at a constant rate. As the birefringence of the compound 4 is relatively high, the guest–host birefringence remains high even with a 30 wt% dopant added. However, this conclusion will only be valid if room temperature range is considered (Figure 7(a) and (b)). If the comparison is made for birefringence of the guest–host systems at elevated temperature of 60°C, the values become even as the T_C effect comes into play.

Figure 8 shows the temperature-dependent viscoelastic coefficient of compound 1 (Figure 8(a)) and

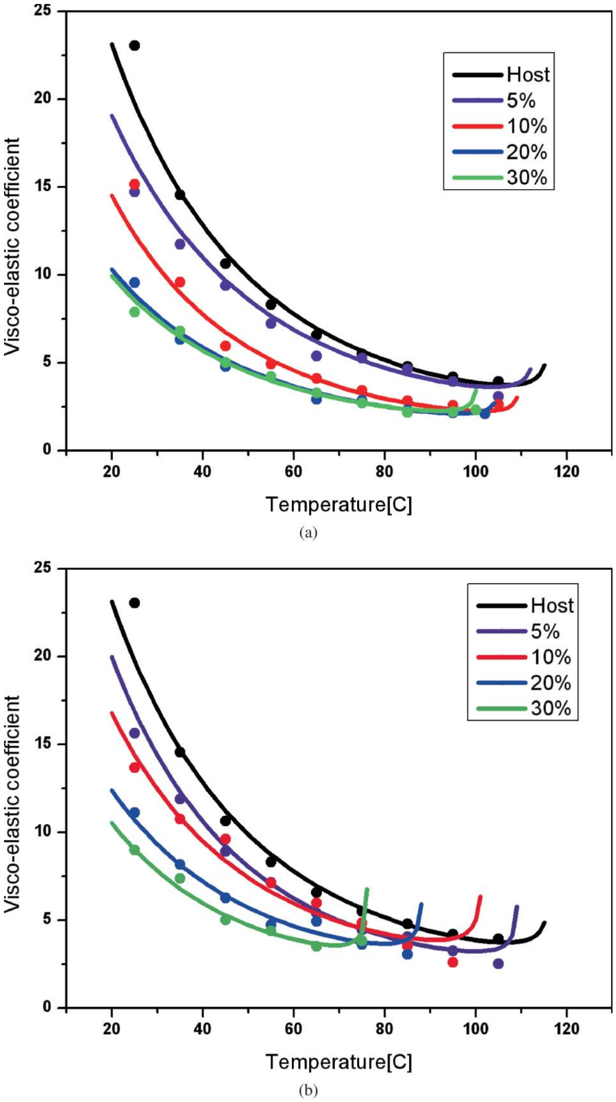


(a)

(b)

Figure 7. Temperature-dependent birefringence of (a) compound 1 and (b) compound 4.

compound 4 (Figure 8(b)). The value of γ_1/K_{33} decreases as the dopant concentration increases. We noticed that for both dopants the viscoelastic coefficient does not change much between 20% and 30% of the dopant amount added. This suggests that systems are saturated with a dopant component and a further increase of guest concentration will only affect the birefringence and clearing point temperature. A decrease of the viscoelastic coefficient further results in FoM values, as Figure 9 shows. Despite the significant structural difference, both compounds show a similar trend if FoM is considered. The highest FoM was measured for 10 and 20 wt% concentrations of diluter components. Although both diluters show similar strength in terphenyl host, for compound 1, at 10% and 20%, the FoM are larger than the host; for compound 4, at 20%, FoM is the largest, but it is still lower than the FoM of the host because of its low clearing point. Despite high birefringence, in both cases, 5% doped mixtures suffer from relatively high viscoelastic coefficient. Therefore, its overall performance expressed by FoM is worse than mixtures with higher concentration of a diluter component. On the other hand, the amount of diluter doped into terphenyl host should exceed 20 wt% as the mixtures with 30 wt% diluter show poor FoM performance resulting from a loss of birefringence and lower T_C, which is especially evident for the case of biphenyl compound 4.



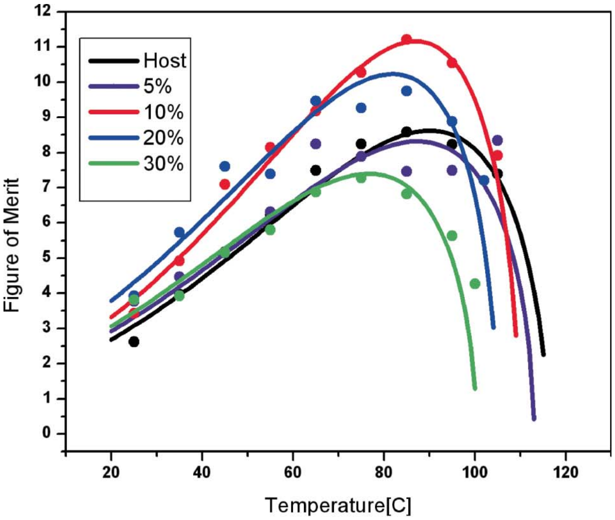
(a)

(b)

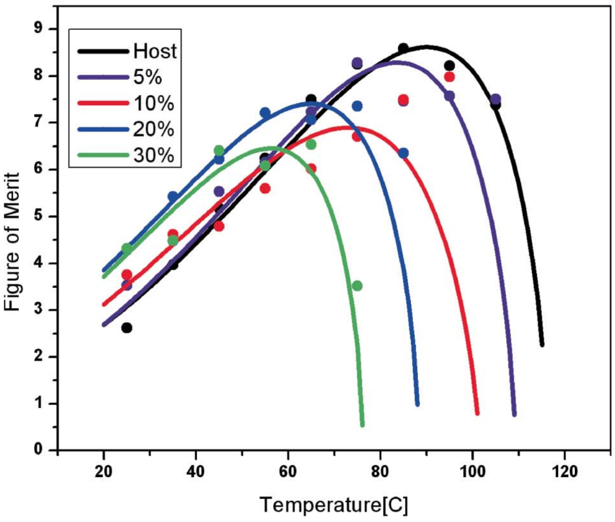
Figure 8. Temperature-dependent viscoelastic coefficient of (a) compound 1 and (b) compound 4.

5. Conclusion

We have reported a detailed study of the performance of low molecular weight dopants if mixed into negative dielectric anisotropy terphenyl-based host



(a)



(b)

Figure 9. Temperature-dependent FoM of (a) compound 1 and (b) compound 4.

mixture. A 4-alkyl-4”alkyl-2’,3’-difluoro-[1,1’;4’,1”]-terphenyl as a general structure was chosen for its relatively low viscosity and high birefringence attractive for display applications. Our results indicate that viscoelastic coefficient is not a linear function of the amount of diluter compound added. Therefore, there is a distinct range of concentration beyond which switching performance of a doped LC mixture would not change while the clearing point temperature will degrade gradually. A second important conclusion resulting from our experiment is that practically any amount of diluter dopant improves the molecular alignment property. These results will have an impact on the LC material formulation in order to optimize the clearing point temperature, birefringence and viscosity of the negative $\Delta\varepsilon$ mixtures from the response time viewpoint. As the fast response time LCs are always in focus, our materials are helpful for reducing the motion picture image blurs. Moreover, LC formulations for colour sequential displays, in which colour filters can be eliminated so that the optical efficiency and device resolution can be tripled, may also be based on the results of the reported experiment.

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