Enhancing Birefringence by Doping Fluorinated Phenyltolanes

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Abstract—We have designed, synthesized, and evaluated the physical properties of some high birefringence (Δn) isothiocyanato phenyltolane and quaterphenyl liquid crystals. These compounds exhibit $\Delta n \sim 0.48 - 0.52$ at room temperature and wavelength $\lambda = 633$ nm. Laterally substituted short alkyl chains and fluorine atom eliminate smectic phase and lower the melting temperature. The moderate melting temperature and very high clearing temperature make those compounds attractive for eutectic mixture formulation. These compounds can be used as dopants for color-sequential liquid crystal displays or eutectic mixtures for laser beam steering.

Index Terms—Eutectic mixtures, high birefringence, liquid crystals, phenyltolane isothiocyanates, quaterphenyl isothiocyanates.

I. INTRODUCTION

TIGH birefringence (Δn) liquid crystals (LCs) support a wide range of applications such as flat panel display [1], laser beam steering [2], and tunable-focus lens [3]. For these devices, high Δn improves the response time of the LC devices through cell gap (d) reduction. This advantage is especially important for color-sequential liquid crystal displays (LCDs) using blinking backlight [4] or primary color (RGB) light-emitting diodes (LEDs) [5] and laser beam steering [6]. To steer a near infrared ($\lambda = 1.55 \,\mu m$) laser beam using an optical phased array, high birefringence ($\Delta n \sim 0.4$) enables a thin ($d \sim 4 \,\mu$ m) LC layer to be used while keeping the required 2π phase change. The use of thin LC layer leads to a reasonably fast response time. In the RGB LED-backlit color-sequential LCDs, the pigment color filters can be eliminated which not only reduces the LCD cost but also triples the device resolution. However, to avoid color break the LC response time (gray to gray) should be kept below 5 ms. Let us use a $1.5-\mu m$ twisted nematic LC cell as an example. To satisfy Gooch-Tarry first minimum condition [7], the required LC should have $\Delta n \sim 0.3$.

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To achieve high birefringence, linearly conjugated molecules are the preferred candidates. Conjugation length can be extended by multiple bonds or aromatic rings in the rigid core. Three problems are associated with highly conjugated LC compounds are high melting temperature, increased viscosity and reduced UV stability. To overcome high melting temperature, we could form eutectic mixtures. The increased viscosity is inherent to all the highly conjugated compounds. However, isothiocyanato (NCS) compounds are less viscous than the cyano (CN) ones except that the NCS compounds tend to exhibit smectic phases [8]. Highly conjugated LC materials typically have absorption tails in the long UV region [9]. The phenyl ring structures are much more stable than triple bond and double bond linking groups. In the laser beam steering at $\lambda = 1.55 \ \mu m$, a UV/visible filter can be placed in front of the optical phased array to remove the harmful UV irradiation. In LED-lit color sequential LCDs, there is no UV content in the RGB LEDs. As a result, the concern for UV-induced LC photostability for high Δn LCs is greatly relieved.

In this paper, we present some high birefringence ($\Delta n \sim 0.5$) isothiocyanato phenyltolane and quaterphenyl compounds and mixtures for the abovementioned applications. These compounds can be used as dopants for enhancing the Δn of existing commercial LC mixtures for color-sequential LCDs or they can be used themselves as eutectic mixtures for laser beam steering.

II. EXPERIMENT

Several measurement techniques were used to measure the physical properties of the single compounds and mixtures. A 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 with rate 2 °C/min. The electro-optic properties of the LC compounds and mixtures were measured using 5 and 8 μ m homogenous cells with indium-tin-oxide (ITO) electrodes coated in the inner sides of the glass substrates. A thin polyimide layer was overcoated on ITO and buffed in antiparallel directions to produce a small pretilt angle (~ 2°). A linearly polarized He–Ne laser with $\lambda = 633$ nm was used as the light sources for the electro-optic measurements. Experimental setup and technique are detailed in [10].

The phase retardation (δ) of the homogeneous cells was measured with a LabVIEW data acquisition system. The LC birefringence at wavelength λ and temperature T was then obtained from the following equation [10]:

$$\delta(V, T, \lambda) = 2\pi d\Delta n(V, T, \lambda) / \lambda \tag{1}$$

TABLE I BIPHENYL-TOLANE BASED COMPOUNDS



III. SINGLE COMPOUNDS—FLUORINATED PHENYL TOLANE BASED ISOTHIOCYANATES

Table I lists the molecular structures and phase transition temperatures of five isothiocyanato phenyltolane LCs investigated. Even with lateral fluorine substitution(s), the melting temperatures of these compounds are still relatively high. The monofluoro substituted compounds 1 and 2 exhibit a wide nematic range, but their melting temperatures are quite high ($\sim 146 \,^{\circ}$ C). Therefore, their solubility in eutectic mixtures would be limited to less than 5%. To further lower the melting temperature and increase the solubility we proposed 3,5-difluorosubstituted isothiocyanates in the neighborhood of the NCS terminal group. Compound 3 is an example of such structure. Indeed, the melting point drops to 65°C for the C4 homologue. Moreover, the smectic phase is destabilized and it exists only in the monotropic form. Next step was to further fluorinate phenyl tolane rigid core. Two different examples are proposed here. Similar to compound 3, compound 4 has difluoro-substituted ring with terminal group. In addition it has additional fluorine laterally substituted in the 3'-position of the tolane part of the rigid core. It does not lower the melting temperature in comparison to compound 3 but the smectic phase is completely suppressed. Another proposed modification of the phenyl tolane rigid core is manifested by compound 5. In this case, there are 2', 3'-difluoro substitutions in the middle ring of the compound and only one fluoro substitution in the neighborhood of the NCS group. This compound shows a relatively low melting temperature and an enantiotropic smectic A phase with transition to nematic phase at 88 °C. As Table I shows, multiple lateral fluorinations lower the melting point of the presented compounds. The tradeoffs are twofold: their viscosity is increased and birefringence reduced. As a result, its overall figure-of-merit (FoM) is reduced quite noticeably. Finding the least viscous modification of the phenyl tolane rigid core could be itself a challenge.

Most of device applications are performed at room temperature (~ 23 °C). Thus, we would like to know the physical properties of these high melting compounds at room temperature. Two methods are commonly employed to extrapolate the electro-optical properties of these single compounds at room temperature: 1) guest-host system and 2) fitting extrapolation from elevated temperature results. In the guest-host system, about 10 wt% of the guest compound to be investigated is dissolved in a room-temperature LC host mixture. By knowing the properties of the host mixture, the properties of the guest compound at room temperature can be estimated. In the second method, we measure the properties of the compound at several temperatures in its nematic phase and then fit these data with theories, and finally extrapolate the results to room temperature. In this study, we focus on the birefringence (Δn) , visco-elastic coefficient (γ_1/K_{11}) and FoM defined as FoM = $K_{11}(\Delta n)^2/\gamma_1$. For example, the birefringence of an LC can be described by the following equation:

$$\Delta n = \Delta n_o (1 - T/T_c)^\beta \tag{2}$$

where Δn_o and β are fitting parameters: Δn_o being the birefringence at T = 0 and β is a material constant, and T_c is the LC clearing temperature. By fitting experimental data with (2), we can obtain Δn_o and β . Once these two parameters are determined, the birefringence of the LC at room temperature can be extrapolated. In this study, we use the second method.

In experiment, we measured the temperature-dependent birefringence and visco-elastic coefficient and then calculated the FoM of the five phenyltolane isothiocyanates listed in Table I. Results are shown in Figs. 1(a)–(c), respectively. With no surprise, both single fluorinated phenyltolane isothiocyanates have the highest birefringence among the five compounds investigated. Two reasons stand for this when we consider rod-like rigid compounds. More linear and fewer lateral substitution the structure is, the higher birefringence it shows. The second reason is their higher T_c . From (2), a higher T_c is helpful to enhance birefringence, provided the molecular structure is similar.

The extrapolated Δn value at 25°C is 0.590 and 0.562 for compounds 1 and 2, respectively. Compound 2 has fluorine substitution in the 2nd position of the phenyl ring with NCS terminal group. Its clearing temperature is about 7 degrees lower than that of compound 1 in which the fluoro substitution is at the 3rd position of the phenyl ring. Another factor affecting birefringence is molecular packing. By comparing the simulated electrostatic charges on the surface of the molecules, the fluorine in the 2nd position has higher negative charge than that in the 3rd position. This suggests that the fluorine in the 2nd position is likely to drag out more π -electron clouds from the conjugated core than if the fluorine is substituted in the 3rd position of the same phenyl ring.

Additional lateral fluorination in the 2-position of the middle phenyl ring, compound 4, further decreases the clearing point so that its birefringence is lowered. However, it does not lower melting point too noticeably for the investigated rigid core. It effectively destabilizes smectic phase and severely lowers the



Fig. 1. Temperature dependent (a) birefringence, (b) visco-elastic coefficient, and (c) FoM of fluorinated biphenyl-tolanes. Dots are experimental results and lines are fittings.

clearing temperatures. The extrapolated birefringence for compound 5 is 0.470 at 25° C, which is the lowest among the five phenyltolanes investigated. The last compound in the phenyltolane group is trifluorinated compound 5. Its difference with compound 4 is the place of one fluoro substitution. In compound 5, the phenyl ring with NCS group has single fluoro substitution in 3-position while the middle phenyl ring has two fluoro groups in the 2', 3'-positions. To our surprise, the birefringence of this compound is similar to that of difluorinated compound 3 rather than trifluorinated homologue (compound 4). Similar to compounds 3 and 4, compound 5 also exhibits a smectic to nematic transition at 88.2°C which is relatively low. Its clearing temperature is very high, similar to those of compounds 1 and 2. It seems like single fluorination of the phenyl ring with terminal NCS group is more favorable for high clearing temperature.

Through the free relaxation time measurement from a homogeneous cell, we are able to calculate the visco-elastic coefficient of the LC compounds. Results are depicted in Fig. 1(b). Along with our prediction the compounds with fewer lateral substitutions indeed exhibit a lower visco-elastic coefficient. From Fig. 1(b), the following sequence is observed: 4 > 5 > 3 > 2 > 1 at room temperature. As the temperature increases, the visco-elastic coefficient declines quickly. As T > 110 °C, the difference between the five compounds becomes relatively small. With the exception for the triple fluorinated compound 4, all other investigated compounds maintain almost the same value of γ_1/K_{11} with slightly lower value for the single fluorinated structures (compounds 1 and 2).

Fig. 1(c) shows the temperature dependent FoM calculated from the data presented in Fig. (1a) and (b). Compounds 1 and 2 have the highest FoM among the five compounds studied. Their FoM reaches 250 μ m²/s and 230 μ m²/s at the optimal operating temperature of 171 °C and 177 °C, respectively, for compounds 1 and 2. On the contrary, the maximum FoM of compound 4 is ~ 40 μ m²/s, which is ~6× smaller than those of compounds 1 and 2.

It is impractical to operate any LC device at such a high elevated temperature. The desired operating temperature is room temperature. Thus, we extrapolate FoM to $T \sim 25$ °C. At low temperature, visco-elastic coefficient increases significantly and FoM decreases to 50 μ m²/s and 40 μ m²/s, respectively, for compounds 1 and 2. These extrapolated results indicate that the single fluorinated phenyltolane isothiocyanates are attractive for those applications where high birefringence is preferred. Triple fluorinated phenyltolane (compound 4) has the lowest FoM among the five compounds studied. High viscosity is a severe drawback of this structure, otherwise, it shows pure nematic phase.

IV. SINGLE COMPOUNDS—FLUORINATED QUATERPHENYL BASED ISOTHIOCYANATES

Unsaturated bonds rich in π -electrons are less stable than a phenyl ring. Thus, we decided to get rid of the triple bond and replace it with a phenyl ring incorporated into the rigid core of the molecule. As expected, the phase transition temperatures are significantly increased. Table II lists the molecular structures of the investigated quaterphenyls and their phase transition temperatures. All three quaterphenyls have clearing point well above 300 °C which is extremely high for LC materials. Thus, decomposition occurred during the transition to isotropic state. Similar to phenyltolane rigid core, the most effective way for decreasing transition temperatures appears to be 3,5-difluoro substitutions in the phenyl ring with the NCS terminal group. Compound 6 has the lowest melting point (113.5 °C) and its smectic to nematic transition (226.8 °C) is also the lowest among the three quaterphenyl compounds studied. Trifluorinated compound 7 shows 10 deg. higher melting temperature and also smectic phase stable at higher temperatures than compound 6. With no substitution at the phenyl ring with polar terminal group, compound 8 has the highest phase transition temperatures among the three quaterphenyl molecular systems investigated.

Due to very high smectic to nematic transition temperatures we were unable to measure the electro-optical properties of the quaterphenyl NCS compounds. Also their high melting enthalpy results in poor solubility. By using either our high birefringence





 TABLE III

 MIXING PATTERN OF PRESENTED GUEST-HOST SYSTEMS

	UCF-1	BL038
EU-1	HB-1	HB-2
EU-2	HB-3	HB-4

host mixture or Merck's BL038 we were still unable to obtain reliable Δn value. We will continue to search a suitable host mixture in order to characterize this series of high birefringence compounds. A limited usability as a dopant medium is predicted as a consequence.

V. EUTECTIC MIXTURES

It is commonly known that only several single compounds possess liquid crystalline properties at room temperature. Thus, eutectic mixtures are commonly practiced. The purposes of our experiment are twofold: 1) to evaluate the electro-optical performance of different phenyltolane isothiocyanates and 2) to formulate high birefringence mixtures with low melting temperatures. Two host mixtures were chosen as benchmarks for comparisons. The first one is our in-house high birefringence mixture UCF-1 and the second is a commercially available Merck high birefringence mixture BL038. Detailed data for both host mixtures can be found in Table IV.

There is a significant difference in the birefringence and viscosity of the host mixtures. It is especially well expressed by the FoM value which at 25 °C was measured to be 11.6 μ m²/s and 2.2 μ m²/s, respectively, for UCF-1 and BL038. This difference originates from birefringence and visco-elastic coefficient. UCF-1 has about 50% higher Δn and lower viscosity than BL038. The clearing point of UCF-1 is comparable to that of BL038. These two mixtures were doped by two different eutectic compositions. The first eutectic mixture, designated as EU-1, contains compounds 1–4, and the second eutectic mixture (EU-2) contains compounds 1–3 and 5. This way we formed four new high birefringence mixtures. Table III shows the mixing patterns. Two mixtures are based on UCF-1, designated as HB-01 and HB-3, and two based on BL038

TABLE IV Physical Properties of UCF-Base Mixtures

Mixture	UCF-1	BL038	HB-1	HB-2	HB-3	HB-4
Т _{иф} [°С]	<-30	<-20	<0	<0	<0	-4
T, [°C]	100	100	130	128	138	136
V _{th} [V _{nas}]	1.6	1.6	1.8	1.7	1.8	1.7
$\epsilon_{\rm II}$	20.9	19.4	21.3	20.1	21.1	21.0
ε⊥	4.7	5.0	4.3	4.7	4.4	5.1
Δε	16.2	14.4	17.0	15.4	16.7	15.9
K11 [pN]	19.7	19.1	28.3	22.5	28.9	24.3
K33 [pN]	21.1	22.4	29.0	24.5	29.5	27.4
∆n						
@633nm	0.378	0.257	0.425	0.332	0.425	0.338
γ1/K11						
[ms/µm²]	12.3	30.2	11.7	26.3	9.7	25.9
FoM						
[µm²/s]	11.6	2.2	15.4	4.2	18.6	4.4

designated as HB-02 and HB-04. The amount we doped was 30 wt% in all cases. The measured physical properties of these mixtures are shown in Table IV.

Based on the mixing patterns several comparisons are possible. The most visible dopant effect is seen from the birefringence increase, as shown in Fig. 2(a). Regardless which host mixture was used, doping 30% of such a phenyltolane increases the birefringence of the host mixtures by 12% and 29%, respectively, for UCF-1 and BL038. The larger increase in BL038 is because of its initial lower birefringence.

In Fig. 2(b), the visco-elastic coefficient of HB-1 and HB-2 does not differ much from their host mixtures UCF-1 and BL038. In both mixtures, a small decrease was observed only near the room temperature. Our UCF-1 host mixture contains about 26% of cyclohexyl-tolane isothiocyanates [11] which are the most viscous components therein. By doping biphenyltolanes we effectively reduce the percentage of the cyclohexyl containing components. As a result, the mixture's visco-elastic coefficient drops slightly. Decreased visco-elastic coefficient of HB-2 is believed to originate from breaking symmetry of the components of BL038 which is mainly comprised of cyano compounds. The cyano biphenyls and terphenyls can pair with the neighboring compounds and form dimmers leading to a high viscosity. We also observed a significant increase of splay elastic constant K₁₁ in the HB mixtures. Noticeable difference of the visco-elastic coefficient is observed if HB-1 and HB-2 are compared with HB-3 and HB-4, respectively, as shown in Fig. 2(b). The reason is that compound 5 has a much lower visco-elastic coefficient than compound 4. Finally, the FoM value of both doped mixtures is higher than that of the hosts. At room temperature, the gain is \sim 32% and 35%, respectively, for HB-1 and HB-2. Higher gain is observed for UCF-1 doped by EU-2. Further increase of FoM is possible if the same mixture is used at elevated temperatures, as depicted in Fig. 2(c). The improvement basically originates from the rotational viscosity decrease as the temperature increases. Moreover, due to the



Fig. 2. Temperature dependent (a) birefringence, (b) visco-elastic coefficient, and (c) FoM of investigated LC mixtures. Dots are experimental results and lines are fittings.

clearing temperature increase, the optimal operating temperature of the FoM of doped mixtures (the temperature at which FoM reaches its highest value) also increases. Thus, if we compare the FoM values at the optimal temperature (100 °C for HB-1, 105 °C for HB-2 and HB-3, and 112 °C for HB-4), the improvement is respectively 50%, 180%, 90%, and 240%.

VI. CONCLUSION

We have developed a series of laterally fluorinated NCS-phenyltolane compounds and mixtures and evaluated

their physical properties. The fluoro substitutions lower the melting temperatures, but also increase the rotational viscosity. These compounds can be used as dopants for enhancing the birefringence of the host LC mixtures for color-sequential LCDs or as a eutectic mixture for laser beam steering.

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