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Liquid Crystals

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Online publication date: 06 August 2010

To cite this Article Iwanaga, Hiroki , Naito, Katsuyuki and Effenberger, Franz(2000) 'Oligothiophene dyes for guest-host liquid crystal displays', Liquid Crystals, 27: 1, 115 — 123

To link to this Article: DOI: 10.1080/026782900203281

URL: http://dx.doi.org/10.1080/026782900203281

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LIQUID CRYSTALS, 2000, VOL. 27, NO. 1, 115–123

Oligothiophene dyes for guest–host liquid crystal displays

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(*Received 26 April 1999; accepted 11 August 1999*)

Three types of donor–acceptor-substituted bithiophene and terthiophene compounds were studied as dichroic dyes, which were dissolved in liquid crystals. These compounds were found to have large absorption coefficients, high dichroic ratios, excellent hues, and to be very attractive for guest–host-type liquid crystal displays. They also showed good solvatochromic behaviour in liquid crystal and cyclohexane solutions. These properties were found to be closely related to the solubilities of these compounds in liquid crystals. One donor–acceptor-substituted bithiophene was found to behave as a polarity indicator in various kinds of liquid crystal.

1. Introduction

Guest–host liquid crystal displays (GH-LCDs) have recently attracted much attention because of their wide viewing angle, excellent hues and high brightness levels [1–5]. Among these LCDs, the reflective types are suitable for portable information systems because of their remarkably low power consumption. In particular, three-layered reflective GH-LCDs, consisting of yellow, magenta and cyan guest–host layers, form a most desirable type due to their excellent bright and pure hues.

To realize bright and pure colours in GH-LCDs, the dye must have a high dichroic ratio, large absorption coefficient, high solubility in LCs, and suitable spectral characteristics. Anthraquinone dyes [6–11] have been the subject of intensive studies due to their excellent photostability and good hues.

We have already reported that specific structures of anthraquinone dyes with trifluoromethyl groups are highly soluble in fluorinated LCs, even at low temperatures [12, 13]. The substitution of a sulphur atom for an oxygen atom in a coumarin dye has also been studied [14]. The substitution of a hetero-atom has been found to be very effective in increasing the solubility of dichroic dyes. Furthermore, mixtures of anthraquinone and coumarin dyes have been found to have high solubility and absorption coefficients, and to be very useful for GH-LCDs [14]. These results were applied to our three-layered reflective-type GH-LCDs, and bright, pure colours were obtained.

However, further efforts are required to achieve reflective-type GH-LCDs of ever higher quality. To realize this objective, the search for new molecular structures as dichroic dyes is considered to be one of the most effective methods. We have focused on donor–acceptor-substituted oligothiophene compounds because of their large absorption coefficients and high dichroic ratios, as predicted from their molecular structures.

Additionally, one donor–acceptor-substituted bithiophene dye was found to be a suitable solvent polarity indicator dye because of its pronounced solvatochromic behaviour [15]. There is a possibility that this dye may behave as an indicator in various kinds of liquid crystals.

2. Experimental

2.1. *Measurement of solubilities in fluorinated liquid crystals*

The solubilities of the dyes in fluorinated liquid crystals (LIXON 5052XX, produced by Chisso Petrochemical Corp.) were measured spectroscopically. A saturated solution was obtained by adding an excess of dye to the LC host, shaking the mixture thoroughly, allowing it to stand for a month at 24°C or − 5°C, and then passing it through a 0.2 μm filter. A known quantity of the solution was dissolved in a standard volume of cyclohexane, and its absorption was measured and compared with that of a standard solution of the dye.

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Liquid Crystals ISSN 0267-8292 print/ISSN 1366-5855 online © 2000 Taylor & Francis Ltd
http://www.tandf.co.uk/journals/tf/02678292.html

2.2. *Structure of the liquid crystal cell*

The cell used in the experiment consisted of two glass plates with an inner area of 1 cm × 1 cm, each with a transparent electrode of indium tin oxide. The oxide films were coated with a polymer layer (alignment layer) which induced a homogeneous structure in the LCs. The alignment layers were rubbed in opposite directions relative to each other, producing parallel-alignment liquid crystal cells. To these cells, LCs in which dichroic dyes were dissolved were injected; the thickness of the cells was 20 µm.

2.3. *Measurement of dichroic ratios*

Absorption spectra were measured with a Shimadzu UV-260 spectrophotometer. The maximum absorption of light horizontally and vertically polarized in the rubbing direction was measured and the ratios were calculated.

2.4. *Measurement of cell contrast*

Absorption spectra were measured with the Shimadzu UV-260 spectrophotometer. In parallel-alignment liquid crystal cells, light polarized parallel to the direction of rubbing was introduced and the maximum absorption was measured. A voltage (30 V: saturated voltage) was then applied to the cell and the maximum absorption was again measured. The ratio between maximum absorption in the on and off states was calculated.

2.5. *Measurement of fluorescence spectra*

Fluorescence spectra were measured with a Hitachi F3000 spectrofluorescence photometer at room temperature. First, about 0.2 wt % of dichroic dye was dissolved in the liquid crystal; then 20–30 mg of this solution was mixed with 5 ml of toluene. The excitation light wavelengths for oligothiophene **1**, **2** and **3** (see figure 1) were 555, 419 and 525 nm, respectively.

2.6. *Calculation of hydrophobic parameter (log P)*

Chemical calculations were carried out using a HyperChem V4.5 program (Hypercube Inc.) with a Toshiba PV-3000 personal computer. An optimum molecule conformation was calculated with a molecular mechanics program (MM+). The hydrophobic parameter log P was calculated for the optimized molecular conformation by using an empirical QSAR program (Quantitative Structure–Activity Relationships).

3. Results and discussion

The bithiophene and terthiophene compounds investigated in this study are shown in figure 1. These compounds have electron-withdrawing and -donating substituents on each side. In order to investigate the application of these compounds as dichroic dyes, their dichroic ratios, electro-optical effects, solubilities in fluorinated LCs and absorption spectra were measured. Solvatochromic behaviour and fluorescence spectra were also investigated.

3.1. *Dichroic ratios of oligothiophene compounds*

Dichroic ratios were measured in parallel alignment liquid crystal cells; the results are shown in table 1. Order parameters were calculated from the absorption of light polarized vertically and parallel to the LC alignment; the maximum absorption was used for this determination.

Table 1. Dichroic ratios and order parameters of oligothiophene dyes.

Dye	Concentration/ wt %[a]	A (parallel)[b]	A (vertical)[c]	D[d]	S[e]
1	0.220	0.62	0.075	8.29	0.71
2	0.189	0.59	0.100	5.78	0.61
3	0.114	0.79	0.122	6.48	0.65

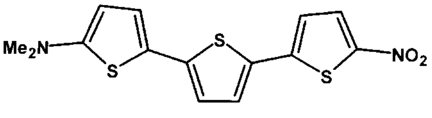
[a] Dye concentration in fluorinated LC.

[b] Absorption of polarized light parallel to the LC alignment direction.

[c] Absorption of polarized light vertical to the alignment direction.

[d] Dichroic ratio, A (parallel)/A (vertical).

[e] Order parameter calculated from [A (parallel) − A (vertical)]/[A (parallel) + 2A (vertical)].



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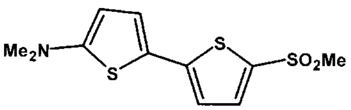

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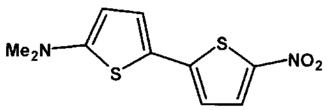

3

Figure 1. Molecular structures of bithiophene and terthiophene compounds.

The oligothiophene compounds were found to have suitable dichroic ratios and order parameters for dyes to be used in GH-LCDs. In particular, oligothiophene **1** has a higher dichroic ratio than those of oligothiophenes **2** and **3**. This is attributed to the length of the molecule; only oligothiophene **1** has three thiophene rings (the others each have two), and its molecule is thus longer than those of **2** and **3**. This result, confirmed that the number of thiophene rings is very important in obtaining a high dichroic ratio in oligothiophene dyes.

3.2. *Electro-optical effects in oligothiophene dyes*

Electro-optical effects were measured using the above-mentioned parallel alignment liquid crystal cells. A voltage was applied to the cells and their absorption was measured in both the on and off states (figure 2). The contrast ratio (*CR*) is defined as shown in equation (1).

$$CR = A(\text{off})/A(\text{on}) \tag{1}$$

where A(off) is the absorption at the maximum absorption wavelength in the off state and A(on) is that in the on state. The results are shown in table 2. The voltage applied to these cells was 30 V; at this voltage the voltage–absorption curve was saturated and a higher voltage was not required.

Theoretically speaking, the dichroic ratio (D) and the contrast ratio (CR) must be equal. However, the CR values were smaller than the dichroic ratios in all of the oligothiophene dyes; the ratio CR/D is shown in table 2. The definition of the dichroic ratio is given as:

$$D = A(\text{parallel})/A(\text{vertical}). \tag{2}$$

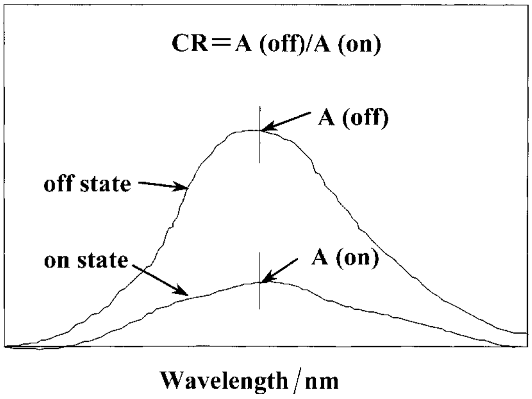

Figure 2. Model of electro-optical effects.

Table 2. Contrast ratio (*CR*) of GH-LCs.

Dye	*CR*	*CR/D*
1	6.93	0.84
2	4.97	0.86
3	4.99	0.77

From this definition, it follows that

$$A(\text{parallel}) = A(\text{off}). \tag{3}$$

The differences between dichroic ratio and contrast ratio are attributed to the differences between A(vertical) and A(on). From equations (1), (2) and (3), the ratio of CR to D shown in table 2 is described by equation (4).

$$CR/D = A(\text{vertical})/A(\text{on}). \tag{4}$$

From table 2, the parameter CR/D is less than unity in each case. This means that even at high voltages, stationary dye is always present. Two possibilities may explain the presence of stationary dye: (a) a small part of the dye is absorbed on the surface of the polymer layer aligned in the rubbing direction and (b) there is a strong anchoring LC layer near the surface of the polymer. The parameter CR/D for the three dyes is approximately 0.8, which means that 20% of the dye is stationary.

3.3. *Solubilities of oligothiophene dyes*

To realize bright, pure colours in GH-LCDs, the dyes must have high solubility in LCs and/or a large absorption coefficient. When the solubility of the dye is sufficiently high, its concentration in the LC can be increased, and the contrast and colour reproduction area of the GH-LCDs is drastically improved. The solubility of the dye at low temperatures is also very important. Dichroic dyes dissolved in LCs may crystallize when GH-LCDs are placed in a cold environment.

There is another serious problem. The LCs generally employed today are fluorinated types, because they exhibit high resistance and are very suitable for the TFT (thin film transistor) driving method. However, the solubilities of dyes in fluorinated LCs are much lower than in the cyanobiphenyl type LCs previously employed [12].

The solubilities of oligothiophene dyes in fluorinated liquid crystals are shown in table 3. The solubilities of these oligothiophene dyes are not very high. However, these dyes have large absorption coefficients, permitting high contrast to be achieved. The temperature dependence of solubility in the range from room temperature to − 5 degrees was found to be small. This is attributed to the

Table 3. Solubility of oligothiophene dyes.

	Solubility/wt %	
Dye	r.t.	− 5°C
1	0.079	0.024
2	0.063	0.021
3	0.16	0.02

molecular structure containing only rigid substituents. When dyes include flexible substituents in their molecular structure, their solubilities at room temperature are very high, but are markedly decreased at low temperatures [12]. The solubilities of the three oligothiophene dyes at low temperatures are nearly equal, but those at room temperature are quite different. The solubility of oligothiophene dye **3** is high, compared with the others, which was found to be closely related to the solvatochromic behaviour as discussed below.

3.4. *Absorption spectra and solvatochromic behaviour*

The absorption spectra are very important with regard to the colours of GH-LCDs. This is especially obvious for three-layered GH-LCDs, in which the dyes themselves are responsible for the colour image, unlike colour filter-type GH-LCDs, in which colour images are realized by filters.

The width of the absorption spectrum is one of the most important properties in relation to hue quality. As the width of the absorption spectrum becomes narrower, the hues become better. The maximum absorption wavelength of the absorption spectrum is also very important. This determines the colour hue, and each type of LCD must have suitable hues. In three-layered GH-LCDs, the yellow layer must have a maximum absorption wavelength from 440 to 460 nm, the magenta layer must have a maximum from 540 to 560 nm, and the cyan layer must have a maximum from 640 to 660 nm.

The solvatochromic behaviour of dyes is the shift of absorption wavelength due to the presence of solvents; this is thought to be closely related to the mutual interactions between dye molecules and LC molecules. The maximum absorption wavelengths of oligothiophene dyes **1**, **2** and **3** in LCs and their solvatochromic behaviour between LCs and cyclohexane are shown in table 4. The spectra of the dyes in liquid crystals and cyclohexane are shown in figures 3, 4 and 5.

The absorption spectra of oligothiophene dyes in LCs have not been reported, and this is the first report of solvatochromic behaviour involving LCs and solvent.

Oligothiophene dye **1** is a magenta dye with a relatively wide absorption spectrum (figure 3, in LCs). The colour purity of this dye is not good due to its wide spectrum, and this dye is therefore considered to be unsuitable for GH-LCDs such as the three-layered types, which require pure colours in each layer. On the other hand, GH-LCDs of the colour filter type require black dyes (a mixture of

Table 4. Maximum absorption wavelengths of oligothiophene dyes in liquid crystal and cyclohexane.

Dye	λ_{max}/nm Cyclohexane	λ_{max}/nm Liquid crystal	Solvatochromic behaviour
1	492	528	36
2	391	408	17
3	472	520	48

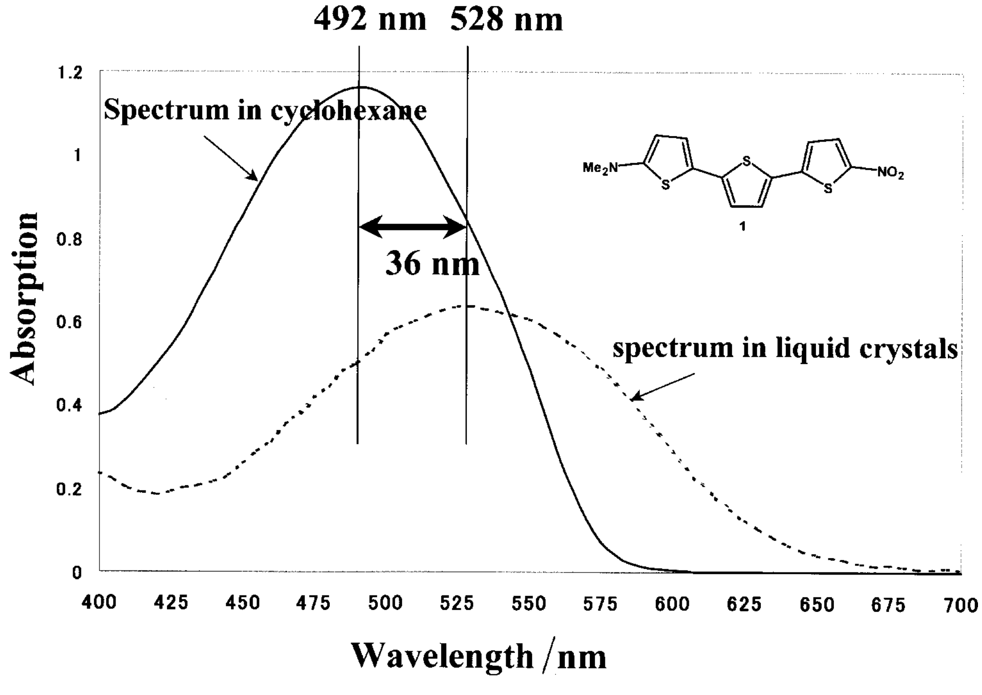

Figure 3. Absorption spectra of oligothiophene dye **1**.

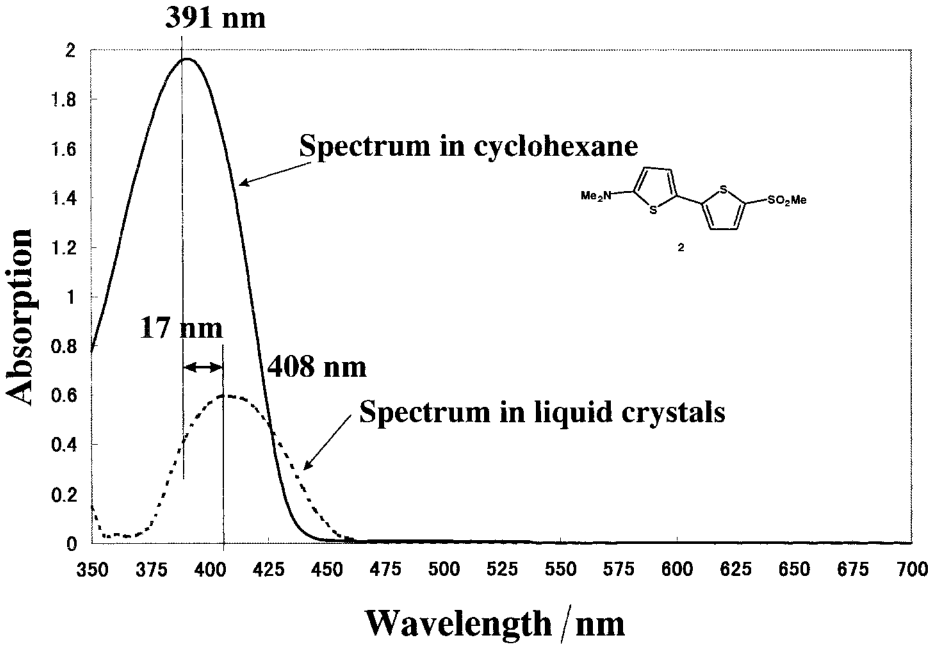

Figure 4. Absorption spectra of oligothiophene dye 2.

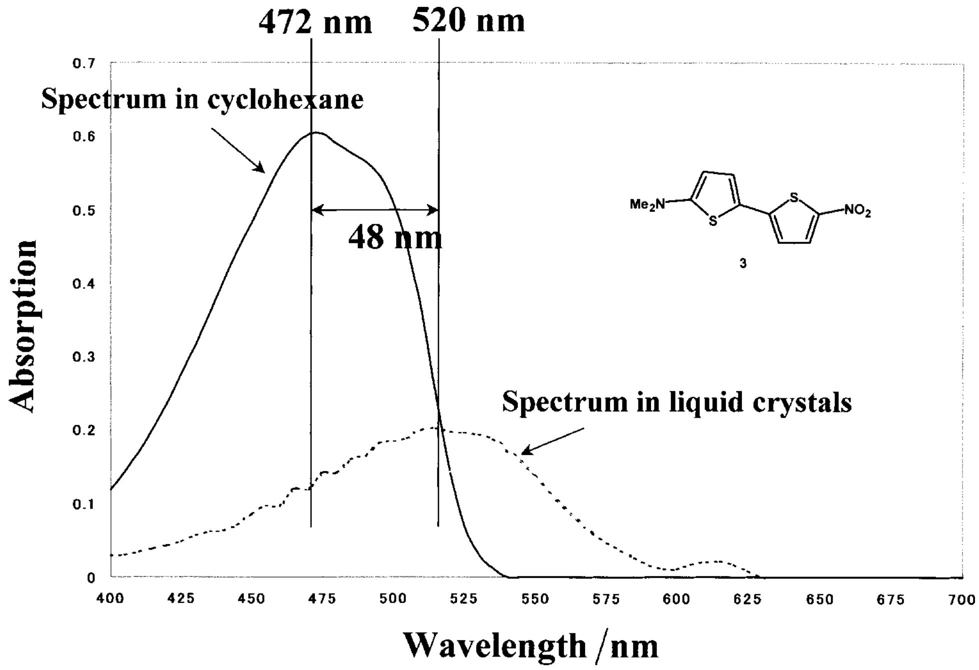

Figure 5. Absorption spectra of oligothiophene dye 3.

yellow, magenta and cyan dyes) which absorb all visible light with no gaps between the spectra. This is a very important factor in LCD contrast. Consequently, oligothiophene dye 1, which has a wide absorption spectrum, is suitable for GH-LCDs of the colour filter type because it avoids absorption gaps in the visible range. The wide absorption spectrum is thought to be closely related to the three continuous thiophene rings.

Oligothiophene dye 2 is a yellow dye with a relatively narrow absorption spectrum (figure 4, in LCs). The colour purity of this dye is very good due to its narrow spectrum, and it is considered to be suitable for GH-LCDs such as the three-layered type. This dye is useful as one element of the yellow dye mixture.

Oligothiophene dye 3 is a magenta dye with a relatively narrow absorption spectrum (figure 5, in LCs).

The colour purity of this dye is very good due to the narrow spectrum, and it is considered to be suitable for GH-LCDs such as the three-layered type. When this dye is used in a three-layered GH-LCD as a magenta dye, the hue of the magenta layer tends toward red. Hence red colours (obtained by the subtractive process of magenta and yellow) are very good and blue colours (obtained by the subtractive process of magenta and cyan) are adversely affected. Using a mixture including magenta dyes tending toward blue can help to balance the colours of the LCDs.

The absorption spectra of oligothiophene dyes **1**, **2** and **3** in cyclohexane are also shown in figures 3, 4 and 5. The maximum absorption wavelengths in cyclohexane are shorter than the corresponding wavelengths in LCs. The solvatochromic behaviour differences between liquid crystals and cyclohexane are summarized in table 4.

The solvatochromic behaviour, dielectric constants of the LCs and the solubilities of the dichroic dyes are thought to be correlated.

Solvents with large dielectric constants behave as good ionic mediums. These solvents are considered effectively to stabilize the dyes in excited states, which have more polar structures than dyes in ground states. The stabilization of dyes in excited states induces the longer wavelength shifts of the maximum absorption wavelengths of dyes; this is the origin of positive solvatochromic behaviour. On the other hand, solvents with large dielectric constants are considered to dissolve polar compounds like dyes, more effectively than the solvents with small dielectric constants; thus solvatochromic behaviour can be correlated with the solubilities of the dyes.

Figure 6 shows the relation between ε (dielectric constant parallel to the molecular axis) of the LCs and maximum absorption wavelengths of oligothiophene **3**. As ε becomes larger, the maximum absorption shifts to longer wavelengths (for LIXON 5052XX, LIXON 4033XX, MLC-6041-000 and ZLI-4900-000). The maximum absorption wavelength of oligothiophene **3** in cyclohexane is 472 nm (21 186 cm^{-1}), and the degrees of shift to longer wavelengths reflect the value of the solvatochromic behaviour (cyclohexane–liquid crystals). We chose cyclohexane as a base for solvatochromic behaviour measurement because the π^* values of cyclohexane equal 0. The π^* values were determined by Kamlet *et al.* [16] and correlated with the solvatochromic behaviour values.

The relationship between dipole moments and solubilities of the dyes in liquid crystals is explained as follows. Since a dipole moment shows the value of the polarity of one molecule, a chemical compound such as a dye with a large polarity can be more easily dissolved as the dipole moment of a solvent becomes larger. Also, larger solvent dipole moments will stabilize dyes in their excited state more effectively. The stabilization of dyes in excited states induces the solvatochromic behaviour. Thus, a solvent with a larger dipole moment will dissolve a larger amount of dye, and it will increase the value of the solvatochromic behaviour.

Further to figure 6, the maximum absorption wavelength of dye **3** in liquid crystal E7 is larger than expected from the value of the dielectric constant. Thus, in the case of cyanobiphenyl-type LCs such as E7, the solvatochromic behaviour (cyclohexane–liquid crystals) is greater than in fluorinated-type liquid crystals such as LIXON 5052XX, LIXON4033XX, etc. Cyanobiphenyl-type LCs have a relatively larger dipole moment than fluorinated-type LCs. Moreover, the solubilities of dyes in fluorinated-type LCs were found to be much smaller than those in cyanobiphenyl-type LCs [12]. From these observations, the following points are clear: the dielectric constant and dipole moment of a solvent can be correlated with the solubility of a dye and with the solvatochromic behaviour. Above all, the solubilities of dyes can be predicted from solvatochromic behaviour.

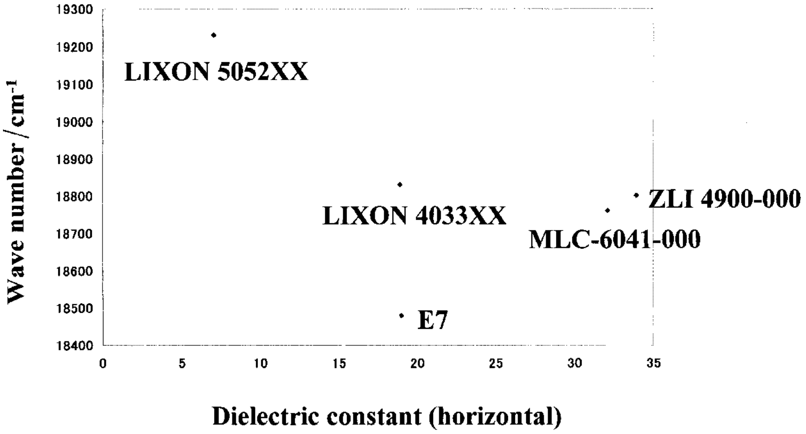

Figure 6. Relation between dielectric constant and maximum wave number for oligothiophene dye 3.

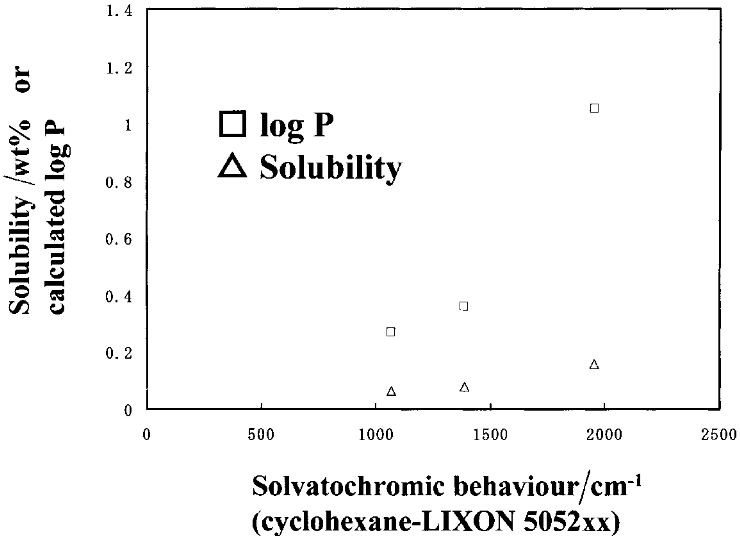

Figure 7. Relation between solvatochromic behaviour and dye solubility.

Oligothiophene dye **3** with a marked solvatochromic behaviour will act as an indicator of the solubilities of dyes in LCs. The LCs currently used are mixtures of many different molecules, and to predict dye solubility is difficult. Our method of predicting solubilities is considered to be very conventional. From figure 6, the lowest maximum absorption wavelength of oligothiophene **3** occurs with LIXON 5052XX, (19 230 cm^{-1}, solvatochromic behaviour: 1956 cm^{-1}); the largest is with E7, (18 480 cm^{-1} solvatochromic behaviour: 2706 cm^{-1}). The solubilities of anthraquinone dye with thiophenyl groups were 2.0 wt% in cyanobiphenyl-type LCs and below 0.01 wt% in LIXON 5052XX [12].

We have also considered the relation between solvatochromic behaviour values the solubilities of oligothiophene dyes **1**, **2** and **3**. LIXON 5052XX (fluorinated-type LC) was used in this investigation, see figure 7. The log *P* parameter was calculated for each oligothiophene dye; this is also shown in figure 7. Log *P* is a hydrophobic parameter, where *P* indicates the distribution ratio of dye between 1-octanol and aqueous solution. From figure 7, the correlation between solubility and log *P* may be observed. The log *P* of the main constituent of LIXON 5052XX is calculated to be 6.91, which is far larger than the log *P* parameter of oligothiophene dyes. The log *P* parameter can be used as one more indicator of dye solubility.

3.5. *The nature of solvation for dichroic dyes in liquid crystals*

One more specific property was observed for LC solutions of oligothiophene dye **3**. The LC solution of oligothiophene dye **3** was heated and maximum absorption wavelengths were observed continuously. Below the temperature of the clearing point, the maximum absorption wavelengths hardly shifted. However, at temperatures above the clearing point, it shifted sharply to a shorter wavelength. This phenomenon was observed in various liquid crystal materials and is summerized in table 5. The wavelength shift above and below the clearing point was 16–17 nm. This can be explained from the nature of the solvation in the liquid crystal phase (figure 8). In the liquid crystal phase, the dye aligns in the direction of the LC long axis. LC molecules and dye molecules are parallel with each other, and a strong dipole–dipole interaction is realized. However, in the isotropic phase (LC solutions heated above their clearing point), the solvation resemble that in a normal solvent. Dipole–dipole interaction between LC and dye molecules is weaker than in the LC phase.

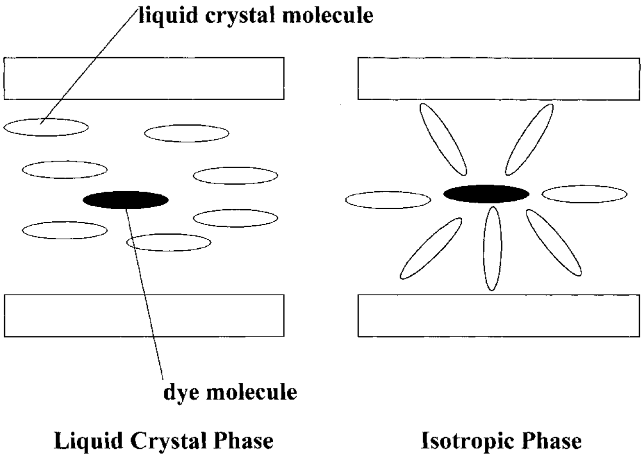

Figure 8. Nature of solvation in liquid crystal and isotropic phases.

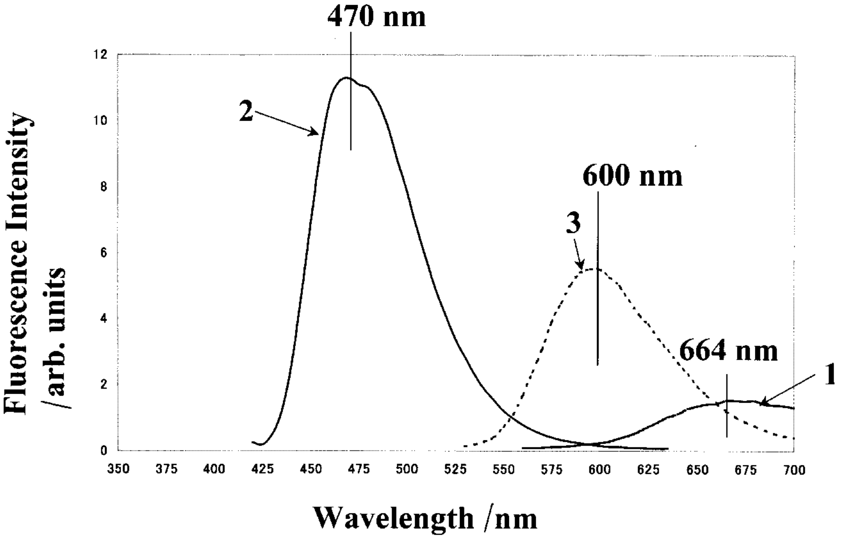

Figure 9. Fluorescence spectra of oligothiophene dyes **1**, **2** and **3** (in toluene).

Table 5. Temperature dependence of solvatochromic behaviour in liquid crystals.

Dye	Liquid crystal	$\Delta\lambda_{max}$/nm[a]
3	LIXON 4033	16[c]
3	MLC6041-000	17[c]
3	LIXON 5052	17[c]
G-176[b]	LIXON 5052	5[c]

[a] The difference between maximum absorption wavelength at above and below clearing point.
[b] Purchased from Nippon Kanko Sikiso Co., Ltd.
[c] Shifted to shorter wavelength.

The maximum absorption wavelength in the isotropic phase is apparently shifted to a shorter wavelength than in the LC phase, in several kinds of fluorinated type liquid crystals; thus the intensity of solvation is stronger in the LC phase than in the isotropic phase.

In the case of G176, which is a widely used dichroic dye, the shift is very small. Oligothiophene dye **3** was found to behave as an excellent indicator of interaction between molecules.

3.6. *Fluorescence spectra of oligothiophene dyes*

The three oligothiophene dyes in this study exhibited fluorescence when they were dissolved in solvent or LCs (figure 9). When dyes in LCs are used in reflective LCDs, the light efficiency is increased and the images displayed on the LCDs become bright. However, it is possible that the hue of the images will be affected by fluorescence. The intensity and maximum wavelength of the fluorescence spectra are affected by the solvent, dye concentration and temperature, and it is considered very difficult to control these factors. If the hue of the images is important, an effective quenching agent is required [14].

4. Conclusion

Donor–acceptor-substituted bithiophene and terthiophene compounds were investigated as dichroic dyes dissolved in liquid crystals. They were found to have adequate dichroic ratios, large absorption coefficients and suitable spectra. Bithiophene dyes have sharp absorption spectra, giving excellent hues. They are suitable for multilayered GH-LCDs such as three-layered reflective GH-LCDs. On the other hand, terthiophene dyes have a broad absorption spectra and absorb a wide range of visible light. These are very suitable for colour filter-type GH-LCDs, which require a pure black LC layer.

Solvatochromic behaviour was found to be closely related to the solubilities of the dichroic dyes in LCs. Oligothiophene compounds can be used as indicators for quickly predicting the properties of liquid crystals and the solubility of dichroic dyes.

The authors would like to thank M. Okajima for his encouragement throughout this work, and K. Sunohara and M. Oguchi for helpful comments.

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